Description of 2019 NXSchool Experiments

Below are descriptions on the experiments offered for the practical tutorials during the 2019 NXSchool. We ask you to submit your preferences for experiments that you wish to participate, though since the spirit of the school is to provide a **broad** exposure to different methods, we ask you to **select experiments from all the different types of techniques** (e.g. not only small angle scattering or only powder diffraction experiments). While we do our best to accommodate your preference, it is impossible to accommodate all your preferences due limits on capacity and scheduling, and we do ensure each participant has an overall broad exposure.

Neutron Experiments:

N1: Triple-Axis Spectrometers
N2: Powder Diffractometer
N3: WAND² powder/single-crystal diffractometer
N4: Engineering Materials Diffractometer
N5: Four-Circle Diffractometer
N6: IMAGINE
N7: Small Angle Neutron Scattering
N8: Small Angle Neutron Scattering (Bio)
N9: BASIS Backscattering Spectrometer
N10: NOMAD Nanoscale-Ordered Materials Diffractometer
N11: TOF Imaging
N12: Magnetism Reflectometer
N13: Liquids Reflectometer
N14: CORELLI Elastic Diffuse Scattering Spectrometer
N15: POWGEN Powder Diffractometer
N16: TOPAZ Single-crystal Diffractometer
N17: NSE Neutron Spin Echo Spectrometer
N18: VISION Inelastic Neutron Spectroscopy
N19: SEQUOIA Fine-Resolution Fermi Chopper Spectrometer
N20: High Pressure, Shull Wollan Center
N21: Low temperature, HFIR Sample Environment
N22: High Temperature, SNS Sample Environment

X-ray Experiments:

X1: High Energy X-ray Diffraction Microscopy and Strain Measurement
X2: X-ray Tomography
X3: Fluorescence and X-ray Ptychography Imaging
X4: X-ray Magnetic Circular Dichroism
X5: X-ray absorption spectroscopy measurements

X6: Energy Dispersive X-ray Diffraction
X7: Radiography
X8: Time-Resolved X-ray Diffraction
X9: Grazing Incidence Small-Angle X-ray Scattering
X10: X-ray photon correlation spectroscopy
X11: X-ray Absorption Near Edge Spectroscopy
X12: Ultra-Small Angle X-ray Scattering
X13: Synchrotron Powder Diffraction
X14: Pair Distribution Function
X15: Small Angle X-ray Scattering
X16: Crystal Truncation Rod Scattering
X17: X-ray Fluorescence Microtomography
X18: X-ray liquid surface scattering
X19: High-Pressure Powder Diffraction
X20: Fundamentals of beamline operation
X21: High-Resolution Inelastic X-ray Scattering Measurements
X22: Reciprocal Space Diffraction
X23: Coherent X-ray Diffraction Imaging
X24: X-ray Micro-Laue Diffraction
X25: Coherent Bragg Rod Analysis

2019 Neutron Experiment descriptions

N1a and N1b: 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 first demonstrate how magnetic excitations are measured on triple-axis spectrometer. Then, we will analyze the actual magnetic excitation data previously measured using a $BiFeO_3$ single crystal on CTAX and HB-1. Combining the low and high energy excitation data observed on CTAX and HB-1, respectively, 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 determination of NiO

Neutron diffraction measurements will be utilized to investigate the onset of long-range magnetic order and determine the magnetic structure in NiO. Data collected at various temperatures, ranging from 600K to 288K, using the HB-2A Neutron Powder Diffractometer at the HFIR will be examined. 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: WAND^2 powder/single-crystal diffractometer, HFIR HB-2C

Crystallographic superstructures Ho_2PdSi_3 and Pr_2PdSi_3

The intermetallic compound series R_2PdSi_3 (R = rare earth metal) exhibits some interesting magnetic properties as giant magneto-resistance effect, strong anisotropy in the electronic properties and a generic field induced phase. The magnetic structures are quite complex with large magnetic unit cells due to the delicate interplay between competing crystal electric field effect and magnetic exchange interaction and the addition of geometric frustration. The hexagonal crystallographic structure is formed from the sequence of triangular rare earth layers and Pd/Si layers stacked along the c-axis. The c/a ration is close to one and gives raise to the expectation of a strong anisotropy between hexagonal axis and plane expressed in large values of the second order of Stevens's operator B20. However, the Pd/Si layers obey site occupation rules of its ions and the stacking of the layers yields a crystallographic superstructure. In the R_2PdSi_3 with heavy rare earth ions (R = Gd, Tb, Dy, Ho, Er, Tm) the connection between the crystallographic superstructure and the magnetic structure has been studied extensively, but it is unknown if a superstructure for compounds with light rare earths (for instance Pr) exists, due to the lanthanide contraction.

We hereby propose to investigate the structure of Pr_2PdSi_3 using WAND^2. We will orient the crystal in HK0 scattering plane and collect a full reciprocal map. With the out-of plane coverage we should be able to cover up to HK0.5 reciprocal space and be able to search for additional reflections.

N4: NRSF2 Engineering Materials Diffractometer, HFIR HB-2B

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 which 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 (HIDRA) 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 data previously measured at NRSF2, 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 (d0) 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. A faux-experiment will be performed at HIDRA, demonstrating how data such as the data being processed is taken. The upgrade of NRSF2 to HIDRA will be discussed in detail to explain the new capabilities and strengths of the upgraded instrument.

N5: 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.

N6: IMAGINE, HFIR CG-4D

Laue white beam diffraction – examples from single crystals in a diamond pressure cell and under dynamic polarization

The IMAGINE CG4-D beamline is designed for rapid survey of the reciprocal space. The beamline optics select a quasi-Laue bandpass, stimulating multiple reflections in a single exposure. The instrument is equipped with a large 2D detector that simultaneously record stimulated Bragg spots. The experiment consists of two parts, an introduction to dynamically polarizing samples - a technique where the signal-to-noise ratio is significantly improved by utilizing the strong spin dependent scattering cross section of hydrogen - and secondly the use of a diamond anvil cell for high pressure single crystal neutron diffraction.

At IMAGINE, neutron single crystal diffraction can be performed using a special neutron diamond anvil cell. The students themselves will load small crystals into the diamond cell together with an appropriate pressure transmitting medium. The will then compare the diffraction from such a small crystal inside the diamond cell against a larger crystal measured for reference without a pressure cell. Both these diffraction patterns will be indexed and analyzed.

N7: Small Angle Neutron Scattering, HFIR CG-2 General Purpose SANS

A Contrast Matching Study of Porous Silica using Small-angle neutron scattering

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 (i.e. varying the volume fraction of H_2O in the mixture of H2O and D_2O), one can change the average scattering length density of the solvent and hence vary the contrast between the scattering objects and surrounding medium. In this experiment, six porasil samples (porous silica) with different H_2O volume fractions (0%, 20%, 40%, 60%, 80% and empty porasil sample) will be investigated. Model-independent data analysis will provide information on porosity, specific surface area as well as contrast matching point to get average scattering length density of the material. Model-dependent data analysis will provide information on the pore diameter and pore-pore distance.

N8a and N8b: Small Angle Neutron Scattering, HFIR CG-3 Bio-SANS

SNS, EQ-SANS, BL-6

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.

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N9: 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 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 synthesized ionic liquid $[H_2NC(dma)_2][BETI]$.

N10: 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.

N11: TOF Imaging, SNS BL3

Characterizing Additive Manufacturing Inconel 718 using Neutron wavelength-dependent Bragg edge Imaging

For decades, neutron imaging has flourished at steady-state facilities (i.e. reactors) that offer intense beams of polychromatic neutrons. At these facilities, scientific applications spawn from energy materials, additive manufacturing, engineering, to plant physiology, biology, archeology, etc. Recently, advanced techniques such as polarized neutron imaging and grating interferometry have been implemented at these neutron user facilities. More recently, and with the development of time-of-flight (TOF) imaging detectors, neutron wavelength-dependent imaging techniques (Bragg-edge and resonance imaging) have thrived at spallation neutron sources and have yielded the construction of state-of-the-art neutron imaging beamlines such as RADEN at J-PARC, Japan, or IMAT in the UK. Spalled neutrons offer novel imaging contrast mechanisms not easily attainable at reactors. Here, the students will apply Bragg edge imaging to additively manufactured metal alloys under room temperature and heating conditions to study the evolution of the alloy's microstructure.

N12: 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 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 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.

N13: Liquids Reflectometer, SNS BL-4B

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.

N14: CORELLI Elastic Diffuse Scattering Spectrometer, SNS BL-9

Introduction to diffuse scattering analysis based on single crystal measurement

CORELLI is a statistical chopper spectrometer with energy discrimination located at beam-line 9 at the SNS. CORELLI is designed and optimized to probe short-range correlation of crystalline materials through single-crystal diffraction and elastic diffuse scattering. CORELLI combines the high efficiency of white-beam Laue diffraction with energy discrimination by modulating the beam with a unique statistical chopper. We will practice the experimental setup, data collection, data reduction on the single crystal $Zr_{0.85}Ca_{0.15}O_{1.85}$ on Corelli. Data collection strategy will be optimized based on initial sample orientation determination. Data reduction and visualization (including the comparison of total and elastic-only spectrum) will be performed using Mantid. The normalized data will be used to perform three dimensional (3D)-PDF using the punch-fill method to reveal the short-range correlation in the system.

N15: 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.

N16: TOPAZ Single-crystal Diffractometer, SNS BL-12

High-resolution single crystal structure analysis using wavelength-resolved Laue diffraction

TOPAZ is a high-resolution single crystal diffractometer for the study of nuclear and magnetic structures of materials at sub-atomic resolution. It uses a large array of neutron time-of-flight detectors for data collection in wavelength-resolved Laue mode to cover a large 3D volume of reciprocal space, or Q-space (after unit conversion from neutron events recorded in detector x, y and a band of neutron wavelengths). We will practice the experimental setup, data collection, data reduction and perform a structure refinement of a single crystal dataset of scolecite ($CaAl_2Si_3O_{10}\cdot 3H_2O$) measured on TOPAZ to locate the missing hydrogen atoms on the water molecules. Scolecite is the calcium member of the natrolite family within the zeolite group. The cation and hydrogen bonding interaction of the water molecules with the framework 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 CrystalPlan program; peak integration will be performed in 3D Q-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 and JANA2006 will also be explored.

N17: NSE Neutron Spin Echo Spectrometer, SNS BL-15

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.

N18: Inelastic Neutron Spectroscopy - INS (VISION), SNS BL-16B

Proton dynamics in phosphoric acid

Phosphoric acid, H_3PO_4, is a tribasic acid commercially available an 85% aqueous solution. The annual production of phosphoric acid is in the tens of megatons range. It is used mainly in the production of fertilizers, but also in the food and cleaning agents industry. In its anhydrous form it crystallizes as a monoclinic solid with a complex network of hydrogen bonds. We will use VISION to examine proton dynamics phosphoric acid. Use will be made of the diffraction detector on the beam line, which permits the simultaneous collection of diffraction and inelastic data. VISION has a dedicated computer cluster for data analysis. In parallel with the experiment, we will calculate the vibrational spectrum of phosphoric acid with Density Functional Theory (DFT) to show how these calculations support spectral interpretation. Use of the new software, O'climax to convert computed vibrational modes to a density of vibrational states directly comparable with VISION data will be demonstrated.

N19: Fine-Resolution Fermi Chopper Spectrometer (SEQUOIA), 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,ω) 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.

N20: High Pressure, Shull Wollan Center

Pressure-induced phase transitions of water ice

A large variety of high pressure experiments are performed across SNS and HFIR (often in conjunction with cooling or magnetic fields). Students will be shown the dedicated ultra-high pressure beamline, SNAP, and the Sample Environment facilities. Several other offline capabilities and opportunities exist such as a high pressure, high temperature laboratory in the Shull Wollan Center (SWC) next to SNS, where students will perform high pressure experiments.

Firstly, they will conduct an experiment on H_2O- ice using the SNAP diamond anvil cell with its ultra-large diamond anvils (5 carat and more). H_2O has one of the most diverse phase diagrams of any substance known. We all know ice I, the hexagonal form that freezes at 0°C and cools our drinks. However, there are at least 17 other known crystallographic structural modifications at varying pressure and temperature conditions. Some of these transitions are optically observable as will be illustrated in the large diamond cell. Secondly, students will observe a laser-heating experiment in the diamond cell where materials such as ice melt at very high pressure and temperature (several thousand Kelvin). Such melting is easily visible with the so-called laser speckle-method. Finally, students will also see the SNS high pressure facilities with Sample Environment and SNAP.

N21: Low temperature, HFIR Sample Environment

Operation of Liquid Helium Cryostats and Closed Cycle Refrigerators

The Sample Environment Group maintains and operates several liquid helium cryostats and closed cycle refrigerators that are used in neutron scattering experiments. These types of sample environments are used on many of the diffraction and spectroscopy beam lines at HFIR and SNS. The proper use of this equipment allows the experimenter to control sample temperatures down to 1.5 K. For this experiment, students will learn the basic theory and operation of a liquid helium cryostat and a closed cycle refrigerator. The students will learn how to properly mount research samples in the apparatus, align samples to the neutron beam, perform sample changes and refill with liquid cryogens.

N22: High Temperature, SNS Sample Environment

Sample preparation, planning, and operation of high temperature vacuum furnaces and high temperature Closed Cycle Refrigerators (CCR)

The Sample Environment (SE) group in neutron sciences at ORNL operates several different high-temperature devices, ranging from room temperature up to 1600^{o} C, in support of neutron scattering experiments. The sample preparation for these experiments is complex, but with the proper preparation and planning the success rate for experiments can increase. For this experiment, students will learn how the different furnaces operate and gain knowledge on the different internal components of the furnaces. Students will learn what proper materials are used for sample holders at high temperature. The students will be presented with the different types of heating that the equipment provides, such as radiative vs. conductive, and what the effects are on the sample in terms of temperature gradients and homogeneity. The students will learn how to properly mount samples onto the sample sticks and high temperature CCRs, using good mechanical and vacuum practices, and how to determine the sample position inside of the equipment to ensure alignment of the sample in the neutron beam.

2019 X-ray Experimental Descriptions:

X1: High-Energy X-ray Diffraction Microscopy and Strain Measurement, 1-ID-E

Characterizing polycrystalline materials using in-situ high energy diffraction microscopy and powder diffraction techniques

Jun-Sang Park and Jonathan Almer

Polycrystalline materials encompass large groups of materials such as metals, ceramics, and minerals. They 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 probe to investigate this relationship in a bulk polycrystalline material. In this experiment, we will conduct high-energy diffraction microscopy and powder diffraction experiments to obtain information about a polycrystalline sample at different length scales.

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 weakly absorbing objects. This is the case for most biological samples, such as soft tissues, but it is also true for wood, polymers etc. In this experiment, we will evaluate the differences between using an absorption vs a propagation phase contrast protocol in various materials

(wood and polymers). 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: Fluorescence and X-ray Ptychography Imaging, 2-ID-D/E

Fluorescence and High-resolution X-ray Ptychographic Imaging of Integrated Circuits
Junjing Deng, Jeffrey Klug, and Olga Antipova

Modern integrated circuits (ICs) employ a myriad of materials organized at nanoscale dimensions, and certain critical tolerances must be met for them to function. To understand departures from intended functionality, it is essential to examine ICs as manufactured ideally in a nondestructive way, and with sufficient spatial resolution to resolve the smallest circuit feature. Ptychography is a scanning coherent lensless imaging technique that allows the imaging of extended samples with spatial resolution not limited by the focusing optics. Using multi-keV coherent X-rays from modern synchrotron, X-ray ptychography is a suitable technique to nondestructively image circuit details with sub-20-nm resolution. In this experiment, we will measure coherent diffraction patterns during ptychographic scans of an IC sample with feature size ranging from hundreds of nm down to 20 nm. We will then reconstruct a real space image of the IC structure from the measured diffraction patterns by performing a phase retrieval computation.

X4: X-ray Magnetic Circular Dichroism, 4-ID-D

Magnetic proximity effect studied using XMCD
Yong Choi

X-ray magnetic circular dichroism (XMCD) measures the difference in absorption of circularly polarized x-rays by a magnetic material. This technique can provide element and orbital specific magnetic information. In this experiment, element-specificity of XMCD will be used to investigate the magnetic proximity effect in a Pt/Fe film. Whereas Pt metal is nominally paramagnetic, the Pt atoms in contact with the Fe layer can have induced magnetic moment. The induced magnetism in the Pt layer will be probed using x-ray magnetic circular dichroism spectroscopy at the Pt $L_{2,3}$ edges.

X5: X-ray absorption spectroscopy measurements, 5-BM-D

X-ray absorption spectroscopy measurements
Qing Ma and Denis Keane

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.

X6: Energy Dispersive X-ray Diffraction, 6-BM-A

Energy Dispersive X-ray Diffraction

Andrew Chuang and John Okasinski

The energy-dispersive x-ray diffraction (ED-XRD) 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.

X7: Radiography, 7-BM

Time resolved radiography of liquid sprays

Alan Kastergren and Brandon Sforzo

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, liquid rockets, jet engines, paint application, and medical inhalers. 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

Characterization of Helical Superconducting Undulator

Don Walko and Haidan Wen

A recently completed project at the Advanced Photon Source has resulted in the design, construction, and use of a helical superconducting undulator (HSCU) at beamline 7-ID. This one-of-a-kind radiation source has several novel characteristics compared to planar

undulators, such as power density and polarization. In this lab, we will compare several properties of the HSCU radiation to that of the APS-standard Undulator A. We will measure the harmonic content of the undulators' outputs and derive their power densities. Then we will use a polarimeter to measure the degree of circular polarization of the HSCU and the degree of linear polarization of Undulator A, and compare with theory.

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

GISAXS from organic photovoltaic thin films

Zhang Jiang, Qingteng Zhang, Joseph Strzalka, and Wei Chen

Since their introduction in the mid-90's, organic photovoltaics (OPV) based on the bulk heterojunction (BHJ) formed between a blend of electron donor and acceptor materials have become a fast-growing area of research, resulting in steady improvement in solar cell efficiencies from approximately 1% to over 10% today. 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 the complex interrelationship between structure, processing and performance. 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 materials 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

Suresh Narayanan, Eric Dufresne, and Alec Sandy

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 and George Sterbinsky

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, Matt Firth, and 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-BM & 17-BM

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

Wenqian Xu, Andrey Yakovenko, & Saul Lapidus

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 analyses of high-energy X-ray data

Olaf Borkiewicz, Kamila Wiaderek, Kevin Beyer, and Leighanne Gallington

Pair distribution function (PDF) depicts local atomic structure as a histogram 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, and 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 and 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: Crystal Truncation Rod Scattering, 13-ID-C

Metal oxide interfacial structures

Joanna Stubbs and Peter Eng

Metal oxide surfaces mediate reactions that control the chemistry of natural waters, battery technologies, catalysis, nuclear fuels, and numerous other natural and engineered systems. Molecular-level measurements of interfacial structures are essential to developing accurate models of natural phenomena and optimizing technologies. However, many available techniques rely on ultra-high vacuum environments, precluding measurements under realistic conditions. In contrast, crystal truncation rod (CTR) diffraction can be conducted in complex environments including gases, liquids, and hazardous material containments. The technique reveals atomic-scale interfacial structures, relaxations, and adsorbate positions on single-crystal surfaces and at buried interfaces. During this experiment we will mount, align, and measure the surface of a metal-oxide crystal then process CTR data and quantitatively fit models to it to discover atomic relaxations and structural perturbations driven by interfacial chemistry.

X17: 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.

X18: X-ray liquid surface scattering, 15-ID-C

Biomolecules at air-water interface

Binhua Lin 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.

X19: 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.

X20: Fundamentals of beamline operation, 20-BM

Fundamentals of beamline operation and Cu XAFS

Steve Heald, Mali Balasubramanian, and Chengjun 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.

X21: High-Resolution Inelastic X-ray Scattering Measurements, 30-ID-C

Phonons in Single Crystals

Ahmet Alatas and Ayman Said

Typically, scattering experiments with x-rays or neutrons are done without energy analysis after the scattering event. Therefore, an integration of all scattered energies is done experimentally in the detector. The information extracted from these experiments is related to information on the structure in the studied system, or, more precisely, to correlation functions of the structure. If the energy of the scattered intensity is analyzed, it is called an inelastic scattering experiment and- in addition to the structural information- dynamical properties of the system can be studied, i.e., information on correlations in time is obtained, Moreover, inelastic x-ray scattering (IXS) provides access to very rich excitation spectra; phonons, magnons, electronic excitations, plasmon and Compton scattering depending on the transferred energy (meV to several hundreds of eV). The Advanced Photon Source has two beamlines (Sector 3 and 30), with very high-energy resolution (1.5-2 meV), specialized for studying collective excitations (phonons) where their energies lie in the order of milli-electronvolts (meV). IXS is very important technique in applications ranging from condensed matter physics to life science and mineral physics to geophysics.

During the NX-school, inelastic x-ray scattering experiments on single crystal aluminum will be demonstrated using HERIX 30 instrument located at sector 30 beamline. We will determine sound velocity and elastic constant along [00L] direction from measure dispersion curve and compare the results with the values found in the literature. Experiment will involve aligning and orienting single crystal before collecting energy spectrum.

X22: 3-D Reciprocal Space 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.

X23: Coherent X-ray Diffraction Imaging, 34-ID-C

Coherent X-ray Diffraction Imaging of Nanocrystals

Wonsuk Cha and 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 300 nm) gold crystal. We will then computationally invert the measured 3D diffraction pattern to a 3D image of the crystal.

X24: X-ray Micro-Laue Diffraction, 34-ID-E

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

Ruqing Xu, Wenjun Liu, and 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 spatial resolution of less than 300 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.

X25: Coherent Bragg Rod Analysis Practical, 12-ID-D

Atomic Imaging Heterostructures and Interfaces by Phasing Coherent Bragg Rods
Hua Zhou

Ubiquitous in a wide range of nature processes and technologies, a subtle modification (e.g. structurally, chemically, or electronically) near an interface can have a decisive effect on properties of the collective as well as each individual. A compelling case manifesting such subtlety is oxide heterostructures and heterointerfaces exhibiting fascinating emergent behaviors due to numerous combinative contributions of atomic structures and chemistries, which can be effectively harnessed for the design of advanced materials for information and energy applications and accelerating materials integration into advanced devices. Surface/interface X-ray scattering from modern synchrotron sources integrated with phase retrieval direct methods provides a very powerful toolkit to decipher the interfacial subtlety. This is essential to our ability to provide a quantitative and realistic description of the interfacial boundaries by which to engineer properties of functional interfaces using atomic structure-driven design principles in a reliable and controlled manner.

In this X-ray school afternoon practical session, we will firstly go through a brief introduction of how to obtain atomic mapping of heterostructure and heterointerfaces with sub-Ångstrom resolution by phase retrieving coherent Bragg rods (COBRA), wherein complete atomically structural information hidden, in particular on the COBRA method in combination with the difference map algorithm achieving unprecedented speed of convergence and precision. In the following, we will mount, align, and measure a high quality perovskite oxide epitaxial thin film (e.g. 5-10 unit cell thick LaNiO3 on SrTiO3 substrate) grown by molecule beam epitaxy, and then process COBRA data and quantitatively carry out the phase retrieval reconstruction to obtain the sub-Å resolution electron density profile of the oxide heterostructure, and to discern the atomic structural perturbations driven by epitaxial strain and interfacial coupling.