Description of the 2021 NXSchool Remote Experiments

Below are descriptions on the experiments offered for the remote tutorials during the 2021 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 (Magnetic Powder Diffraction)
N3: WAND powder/single-crystal diffractometer
N4: HIDRA Engineering Materials Diffractometer
N5: DEMAND Single-Crystal Diffractometer
N6: Small Angle Neutron Scattering (General Purpose SANS)
N7: Neutron Imaging Station
N8: Small Angle Neutron Scattering (BIO-SANS, HFIR)
N9: Small Angle Neutron Scattering (EQ-SANS, SNS)
N10: NOMAD Nanoscale-Ordered Materials Diffractometer
N11: BASIS Backscattering Spectrometer
N12: SNAP Spallation Neutrons at Pressure
N13: Magnetism Reflectometer
N14: CORELLI Elastic Diffuse Scattering Spectrometer
N15: POWGEN Powder Diffractometer
N16: TOPAZ Single-Crystal Diffractometer
N17: HYSPEC Hybrid Spectrometer
N18: NSE Neutron Spin Echo Spectrometer
N19: ARCS Wide-Angular Range Chopper Spectrometer
N20: High Pressure Science, Sample Environment Section

X-ray Experiments:
X1: High Energy X-ray Diffraction Microscopy
X2: X-ray Tomography
X3: Fluorescence and X-ray Ptychography Imaging
X4: X-ray Fluorescence Microscopy
X5: X-ray Magnetic Circular Dichroism
X6: X-ray Absorption Spectroscopy
X7: Extended X-ray Absorption Fine Structure
X8: Energy Dispersive X-ray Diffraction
X9: Magnetic X-ray Scattering
X10: Grazing Incidence Small-Angle X-ray Scattering
X11: X-ray Photon Correlation Spectroscopy
X12: Ultra-Small Angle X-ray Scattering
X13: Synchrotron Powder Diffraction

X14: Pair Distribution Function
X15: Grazing-Incidence Pair Distribution Function
X16: Small Angle X-ray Scattering
X17: Coherent Bragg Rod Analysis
X18: Crystal Truncation Rod Scattering
X19: X-ray Fluorescence Microtomography
X20: Anomalous Small-Angle X-ray Scattering
X21: High-Pressure Powder Diffraction
X22: Fundamentals of Beamline Operation and XAFS
X23: Angle-Resolved Photoemission Spectroscopy
X24: Resonant Soft X-ray Scattering
X25: Reciprocal Space Diffraction
X26: Coherent X-ray Diffraction Imaging

2021 Neutron Experiment descriptions

N1: Triple-Axis Spectrometers, HFIR HB-1, HB-1A, HB3 & CTAX

Spin wave and phonon dispersion in Fe-Ga solid solutions

The triple axis spectrometry (TAS) is a versatile technique in the measurement of the scattering function in energy and momentum space. The difficulty in using a TAS instrument lies in the constant need to translate reciprocal space parameters to real space motor positions and vice versa. The TAS team will demonstrate how to carry out a TAS experiment using vTAS (virtual Triple Axis Spectrometer), which is an interactive software that shows both the TAS geometry and the momentum space. The demonstration includes how to align a single crystal sample and set up scans on SPICE (Spectrometer Instrument Control Environment) and will use Fa-Ga alloys as an example. These materials exhibit giant magnetostriction in a polycrystalline and ductile form and are of tremendous scientific and technological interest for use in devices such as actuators, transducers and sensors.

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: WAND^2 Powder/Single-Crystal Diffractometer, HFIR HB-2C

Crystallographic superstructures in 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 Pd/Si layers obey site occupation rules of its ions and the stacking of the layers yields a crystallographic superstructure.

WAND² has a 2D-position sensitive detector covering 120° in-plane and 15° out-of plane. By rotating the sample, a huge area of reciprocal space is mapped. The high efficiency and low background of the instrument allows the detection of very weak reflections. Using the remote control, the sample will be aligned and a scan for a full reciprocal map setup. The data will be reduced and analyzed using MantidWorkbench and FullProf.

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

Non-destructive residual stress/strain measurement of machined aluminum bar

"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 a precision machined aluminum bar as an example. Understanding machining effects on samples and the evolution of residual stress is very important. A token sample has been prepared whereby a measurement of residual stress via neutron diffraction can be compared to other destructive techniques such as the Montour method. This part will be measured and the results compared to that of a FE model as well as results obtained via contour method.

N5: DEMAND Single-Crystal 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. Unrelated, this solid solution also exhibits antiferromagnetism at low temperatures. Using single crystal neutron diffraction, one can refine the nuclear structure (atom positions, anisotropic atomic displacement parameters, Fe/Mn ratio, Li content) and the magnetic structure (Fe/Mn moment magnitudes). Data sets, exercises and user guides will be provided to refine both nuclear and magnetic structures using Fullprof.

N6: 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 measured. 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.

N7: Neutron Imaging Station, HFIR CG-1D

Liquid transport in biocatalytic yarns

Investigation of water (or aqueous solution) transportation affected by biocatalytic coating requires carefully designed experiments to understand the influence of coating parameters and the textile structure. Among all the methods that have been used in characterizing the wicking profiles of water inside textiles, neutron radiography is the most straightforward one by directly imaging the water flow without introducing any dye molecules. By participating this experiment, you will investigate the real-time water uptake process by continuously acquiring neutron radiographs at a high frame rate. After the process reaches equilibrium, a computed tomography measurement is followed to help revealing the spatial distribution of water inside each sample.

N8 and N9: 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.

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.

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.

N11: 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 $[H_2NC(dma)_2][BETI]$.

N12: SNAP Spallation Neutrons at Pressure, SNS BL-3

Pressure-induced phase transitions of water and ice at room temperature

The SNAP instrument is ORNL's dedicated high pressure neutron diffractometer. It is a highly versatile instrument optimized for the small sample sizes compatible with the existing suite of neutron compatible pressure cells. The beamline has available standard gas pressure and clamp cells as well as the entire range of Paris-Edinburgh presses. These can reach pressures of up to 20 GPa. More recently, SNAP and the High Pressure sample environment group have been leading the development of record breaking neutron diamond cells which have achieved the highest pressure recorded with neutron scattering as well as the largest volume of any sample held at static pressures above 1 MBar (100 GPa).

During this experiment, students will be introduced to the various pressure cells and the opportunities and challenges available at SNAP. Students will participate in a virtual tour of the instrument and become familiarized with its components and how they can be configured for specific experiments. Students will then be invited to a DAS remote session to setup an experiment and control the instrument. Data collected at the beamline will be analyzed in real-time in the analysis cluster.

The science case that will be the theme of the data collection and analysis portion is that of water ice. This substance has one of the most diverse phase diagrams known. We all know ice I, the hexagonal form that freezes at 0 ^{o}C and cools our drinks. However, there are at least other 17 known crystallographic structural modifications at varying pressure and temperature conditions, all of which are more dense than the liquid form under equilibrium conditions. At room temperature, upon compression, water 'freezes' at about 13 kbar into ice VI, further transforming at 30 kbar to ice VII, both with inter-penetrating hydrogen bond networks. Overall, the tetrahedral and highly directional nature of the hydrogen bond leads to a fascinatingly diverse P-T structural phase diagram. Once analyzed, the data will reveal the characteristic features of an amorphous (liquid) substance, as well as the subsequent solid-solid transitions under pressure.

N13: 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. Polarized Neutron Reflectometry (PNR) is a powerful 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. The sample will be mounted in the closed cycle refrigerator and students 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 learn how to process the data using the data reduction program 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.

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 beamline 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 describe 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 (NAC, Na2Ca3Al2F14) 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 SHELX. The option to refine the neutron structure in JANA2006 and GSAS will also be demonstrated.

N17: Hybrid Spectrometer (HYSPEC), SNS BL-14B

Separating nuclear and magnetic scattering 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. Polarized neutron measurements allow to

unambiguously distinguish between the structural and magnetic scattering features and determining the direction of magnetic moments and their fluctuations. There are currently two distinct modes of running polarized experiments at HYSPEC: a "half-polarized" mode where successive measurements with polarized incident beam oriented parallel and anti-parallel to the external magnetic field are performed without polarization analysis of the scattered beam, or a "XYZ-polarization analysis where neutron spin flip effects are measured by analyzing of the final polarization with respect to the incoming polarization. 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 exercise we will discuss the experimental procedures and will demonstrate, using previously collected data, the use of "half-polarized" or "XYZ-polarization analysis to measure weak ferromagnetic scattering, or to separate the nuclear coherent, nuclear spin-incoherent, and magnetic scattering for a series of standard materials (Fe, TiZr, V-rods, and MnO powder sample). The exercise will enable students to gain a better understanding of the polarized neutrons scattering technique, as well as some experience with data processing and visualization using MSlice -Mantid package.

N18: 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.

N19: Wide-Angular Range Chopper Spectrometer (ARCS), SNS BL-18

Dynamics of metal hydride systems: Harmonic oscillators and beyond

The hydrogen in zirconium hydride (ZrH_2) sits at the interstitial positions between the zirconium. In the simplest description, the energy levels can be considered to be the same as a particle in a potential well. The aim of this experiment is to measure the vibrational spectrum of ZrH_2 as a function of energy and wavevector transfer, and determine how well it conforms to the predictions of the scattering from a harmonic oscillator. Practical applications of data collection and analysis will be given to generate the scattering function $S(Q,\omega)$ from the data. This will be compared to theoretical predictions based on the harmonic oscillator description, with a discussion of what may cause any discrepancies found. As time permits, other samples or experimental conditions (temperature, incident energy) will be measured to highlight differences in the energy spectra. As this is a remote school, the experiment teams will learn to use the remote tools to collect the data.

N20: High Pressure Science, Sample Environment Section

High pressure synthesis in a neutron diamond anvil cell

A large variety of high pressure experiments are performed across the suite of SNS and HFIR using the full range of scattering techniques, e.g. powder and single crystal diffraction, inelastic or small

angle neutron scattering. Unlike the diamond anvil cells used for the vast majority of X-ray pressure experiments, high pressure neutron scattering uses a range of cells optimized for maximum pressures, sample volumes and combination with other extremes such as high field or (ultra-)low temperature. During this experiment, the students will participate in an interactive live demonstration of the assembly of a neutron diamond anvil cell followed by an in situ high pressure synthesis of a metastable silicon phase. Therefore, a cell will be pressurized live to ~15 GPa and corresponding ruby pressure measurements will be conducted. At this pressure, the normal semiconducting silicon becomes metallic which is directly seen through optical observation. Recovery from this metallic phase then yields a novel metastable Si structure. Finally, the experiment also includes a live tour of SNS and HFIR pressure facilities that showcase additional pressure capabilities (e.g. gas pressure cells, clamp cells, McHugh pressure cells for SANS) as well as high pressure laboratories at SNS (offline Raman laboratory, laser-heating set-ups etc.).

2021 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 Hemant Sharma

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

X-ray 3D imaging using fly scan and streaming data analysis

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

Fluorescence and High-resolution X-ray Ptychographic Imaging of Integrated Circuits

Jeffrey Klug and Junjing Deng

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 Fluorescence Microscopy, 2-ID-E

X-Ray Fluorescence Imaging of PV samples and battery particles

Olga Anitpova and Lu Xi Li

X-ray Fluorescence Microscopy (XFM) is a powerful tool for elemental characterization of variety of biological, environmental, and material science samples. Zone-plate focused beam allows simultaneous mapping of multiple elements within sample with 200-600 nm resolution. While XFM works best for 2D imaging of thin samples, 3D imaging allows more comprehensive understanding of internal composition and elemental co-localization of more complex samples. X-ray fluorescence tomography involves collection of multiple projections of sample, which requires substantial time and may induce radiation damage and sample degradation. During this experiment we will examine the sample stability during repetitive data collection using MAPS and aspects of 3D image reconstruction using XRFtomo.

X5: 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, we will measure an induced ferromagnetic moment from a Pt/Co multilayer film. Whereas Pt metal is nominally paramagnetic, the Pt atoms in contact with the Co layer can have induced magnetic moment. An XMCD measurement at the Pt L_3 edge will be conducted in fluorescence mode to probe the net magnetic moment in the Pt layer in contact with the ferromagnetic Co layer.

X6: X-ray Absorption Spectroscopy, 5-BM-D

X-ray absorption spectroscopy measurements

Qing Ma, Mike Guise, 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.

X7: Extended X-ray Absorption Fine Structure (EXAFS), 10-BM-B and 10-ID-B

EXAFS setup and measurements

Carlo Segre

Students will see how the beamlines are set up for EXAFS and then measure a standard foil and some reference compounds. Both transmission and fluorescence modes will be shown with the use of a fluorescence detector and an ion chamber. Methods to reject higher harmonics will be demonstrated on the bending magnet by detuning of the monochromator and on the ID beamline by use of an optical mirror. Also, the difference between continuous and step scanning will be shown.

X8: 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.

X9: Magnetic X-ray Scattering, 6-ID-B

X-ray resonant magnetic scattering (XMRS)

Jong-Woo Kim and Philip Ryan

X-ray resonant magnetic scattering (XRMS) measures a microscopic magnetic structure of materials with polarized x-rays. This experiment will go over the basics of aligning a single crystal in a diffractometer and measuring a magnetic Bragg diffraction peaks from a rare-earth compound. The magnetic Bragg peak intensity as a function of incident x-ray energy will be taken to observe the resonance enhancement at the element absorption edge and 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.

X10: Grazing Incidence Small-Angle X-ray Scattering (GISAXS), 8-ID-E

GISAXS from organic photovoltaic thin films

Joseph Strzalka and Zhang Jiang,

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.

X11: X-ray Photon Correlation Spectroscopy, 8-ID-I

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

Qingteng Zhang, Eric Dufresne, 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 over a range of time scales in the range of 20 microseconds – 1000 seconds. 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.

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

USAXS/SAXS/WAXS studies of structure of common materials

Jan Ilavsky 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

Andrey Yakovenko, Wenqian Xu, and 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. This practical session will cover basics of synchrotron 1D and 2D powder diffraction, sample preparation, data collection and preliminary

data analysis. Attendees will watch a live in situ experiment remotely performed at 17-BM and discuss with the beamline staff.

X14: Pair Distribution Function, 11-ID-B

Pair distribution function measurements with High-Energy X-rays

Tiffany Kinnibrugh, Kamila Wiaderek, and Leighanne Gallington

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

X15: Grazing-incidence Pair Distribution Function, 11-ID-C

Grazing-incidence Pair Distribution Function (GI-PDF) measurements

Olaf Borkiewicz and Yang Ren

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 any 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 (see also previous experiment X14).

The atomic structure of amorphous and nanostructured thin films on flat substrates can be measured using photon energies > 80 keV in grazing-incidence geometry. This approach significantly enhances the signal of the thin film and enables investigations unachievable through traditional experiments in transmission geometry. We will compare transmission geometry, flat incidence and grazing incidence geometry to determine the structure of as-grown and annealed 100-nm thin films.

X16: 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.

X17: Coherent Bragg Rod Analysis (COBRA), 12-ID-D

Atomic Imaging of Heterostructures and Interfaces by Retrieving 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 year X-ray summer school practical session (via remote access virtual platform), 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 $LaNiO_3$ on $SrTiO_3$ 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.

X18: Crystal Truncation Rod Scattering, 13-ID-C

Crystal Truncation Rod Diffraction for Atomic-Scale Surface Measurement

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 virtual "hands-on" experiment, we will demonstrate all aspects of CTR data collection and analysis. Participants will have the opportunity to "walk" into the experimental station with us and observe firsthand how we mount, align, and measure the surface of a metal-oxide crystal. As the data rolls in we will perform real-time data reduction followed by quantitative fitting of the collected CTR data leading to a model that reveals the interface's atomic relaxations and structural perturbations driven by interfacial chemistry.

X19: 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. The penetrating power of X-rays into low-Z material such as seeds means that a micro-XRF spectrum will average over 100s of microns, blurring the spatial resolution. In this experiment, we will combine X-ray fluorescence spectroscopy with computed microtomography, using both the imaging and spectroscopic properties of X-rays to study the elemental distribution within a seed of arabidopsis thaliana. By rotating and translating a seed through a micro-focussed X-ray beam, a virtual slice will be made to image the elemental distribution of different metal elements within the seed. The virtual demonstration experiment will include mounting and centering the sample, collecting the rotational and positional data, processing the X-ray fluorescence spectra, and performing tomographic reconstruction to visualize the distinct distributions of metals with the seed.

X20: Anomalous Small Angle X-ray Scattering, 15-ID-C

Anomalous Small Angle X-ray Scattering (ASAXS) studies of nanomaterials

Mrinal Bera, Wei Bu, Binhua Lin, and Natalie Chen

In this virtual experimental session, we will demonstrate some of the basic concepts of Anomalous Small Angle X-ray Scattering (ASAXS) in determining distribution of element of interest within and around nanomaterials. In particular, emphasis will be put on to the methods of collecting good quality ASAXS data followed by systematic data reduction and analyses recently developed at NSF's ChemMatCARS (Sector-15, Advanced Photon Source) through a virtual experiment on core-shell nanoparticles.

X21: High-Pressure Powder Diffraction, 16-BM-D

Pressure-induced phase transition in ZnO

Ross Hrubiak

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.

X22: Fundamentals of Beamline Operation and XAFS, 20-BM

Fundamentals of beamline operation and XAFS examples

Steve Heald 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/.

X23: Angle-Resolved Photoemission Spectroscopy, 29-ID

Angle-resolved photoemission spectroscopy (ARPES) of a topological insulator

Jessica McChesney and Fanny Rodolakis

During the practical students will be able to collect angle resolved photoemission spectroscopy (ARPES) and core-level x-ray photoemission spectroscopy data (XPS) data on the quintessential topological insulator Bi_2Se_3.

X24: Resonant Soft X-ray Scattering, 29-ID

Measurement of charge order in complex oxides using resonant soft X-ray scattering

Jessica McChesney and Fanny Rodolakis

During the practical students will be introduced to resonant soft X-ray scattering (RXS) and how it can determine magnetic, orbital, or charge ordering in complex oxides.

X25: 3-D Reciprocal Space Diffraction, 33-BM-C

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.

X26: 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.