Description of the 2022 NXSchool Experiments

Below are descriptions off the experiments offered for the practical tutorials during the 2022 NXSchool. We ask you to submit your preferences for six out of eight neutron and x-ray experiments (three each) that you wish to participate. Since the spirit of the school is to provide a **broad** exposure to different methods, we ask you to **select experiments from three different groups of techniques**. Please also check that your selections are not in conflict with each other with regards to the days they are offered (if listed), e.g., if all three of your selections are only available Tuesday and Friday, then for sure you won't be able to get one of them. 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

Group 1: Inelastic Neutron Scattering (N1-N5)

N1: Triple-Axis Spectrometers, HFIR HB-1A & HB3
N2: BASIS Backscattering, SNS BL-2
N3: HYSPEC Hybrid Spectrometer, SNS BL-14B
N4: VISION Neutron Spectroscopy, SNS BL-16B
N5: SEQUOIA Fine-Resolution Fermi Chopper Spectrometer, SNS BL17

Group 2: Neutron Diffraction (N6-N13)

N6: Powder Diffractometer, HFIR HB-2A
N7: WAND^2 powder/single-crystal diffractometer, HFIR HB-2C
N8: DEMAND Single-crystal Diffractometer, HFIR HB-3A
N9: NOMAD Nanoscale-Ordered Materials Diffractometer, SNS BL-1B
N10: VULCAN Engineering Materials Diffractometer, SNS BL-7
N11: CORELLI Elastic Diffuse Scattering Spectrometer, SNS BL-9
N12: POWGEN Powder Diffractometer, SNS BL-11A
N13: TOPAZ Single-crystal Diffractometer, SNS BL-12

Group 3: Neutron Imaging (N14)

N14: TOF Imaging, SNS BL3

Group 4: Large-Scale Structure (N15-N20)

N15: GP-SANS General Purpose Small-Angle Neutron Scattering, HFIR CG-2
N16: Bio-SANS Biological Small-Angle Neutron Scattering, HFIR CG-3
N17: EQ-SANS Extended Q-Range Small-Angle Neutron Scattering, SNS BL-6
N18: MAGREF Magnetism Reflectometer, SNS BL-4A
N19: LIQREF Liquids Reflectometer, SNS BL-4B
N20: NSE Neutron Spin Echo Spectrometer, SNS BL-15

Group 5: Complex Sample Environments (N21-N25)

N21: High Pressure Science, Shull Wollan Center & SNS BL-3
N22: High Temperature, SNS Sample Environment
N23: Low temperature, SNS Sample Environment
N24: Soft Matter, Shull Wollan Center

X-ray Experiments

Group 1 (X1-X3): Imaging Experiments

X1: High-Energy X-ray Diffraction Microscopy and Strain Measurement, 1-ID-E (Wed/Fri)
X2: X-ray Tomography, 2-BM (Tues/Thurs/Fri)
X3: X-Ray Fluorescence Microscopy, 2-ID-E (Tues/Wed)

Group 2 (X4-X8): X-ray Spectroscopy Techniques

X4: High-resolution X-ray Fluorescence, 1-BM-C (Wed/Thurs)
X5: X-ray Absorption Spectroscopy, 5-BM-D (Tues/Thurs)
X6: Extended X-ray Absorption Fine Structure (EXAFS), 10-BM-B and 10-ID-B (Fri/Fri)
X7: Fundamentals of Beamline Operation and XAFS, 20-BM (Tues/Wed/Thurs)
X8: Angle-Resolved Photoemission Spectroscopy, 29-ID (Wed/Fri)

Group 3 (X9-X13): Resonant/Magnetic Scattering/Spectroscopy Techniques

X9: X-ray Magnetic Spectroscopy, 4-ID-D (Thurs/Fri)
X10: X-ray Magnetic Scattering, Helicity Dependence, 4-ID-D (Tues/Wed)
X11: X-ray Resonant Magnetic Scattering, 6-ID-B (Tues/Wed/Thurs/Fri)
X12: Resonant Soft X-ray Scattering, 29-ID (Tues/Thurs)
X13: Resonant Inelastic X-ray Scattering, 27-ID-B (Tues/Wed/Thurs/Fri)

Group 4 (X14-X17): Coherence Based Techniques

X14: X-ray Ptychography Imaging, 2-ID-D (Tues/Wed)
X15: X-ray Photon Correlation Spectroscopy, 8-ID-I (Tues/Wed)
X16: Coherent Bragg Rod Analysis (COBRA), 12-ID-D (Thurs/Fri)
X17: Coherent X-ray Diffraction Imaging, 34-ID-C (Tues/Thurs)

Group 5 (X18-X20): Small-Angle X-ray Scattering

X18: Grazing Incidence Small-Angle X-ray Scattering (GISAXS), 8-ID-E (Thurs/Fri)
X19: Small-Angle X-ray Scattering, 12-ID-B (Tues/Wed)
X20: Anomalous Small Angle X-ray Scattering, 15-ID-C (Thurs)

Group 6 (X21-X24): Diffraction-1

X21: Energy Dispersive X-ray Diffraction, 6-BM-A (Tues/Thurs)
X22: Synchrotron Powder Diffraction, 11-BM & 17-BM (Tues/Thurs)
X23: Pair Distribution Function, 11-ID-B (Tues/Wed)
X24: Grazing-Incidence Pair Distribution Function, 11-ID-C (Thurs/Fri)

Group 7 (X25-X28): Diffraction-2

X25: Crystal Truncation Rod Scattering, 13-ID-C (Fri)
X26: High-Pressure Powder Diffraction, 16-BM-D (Wed/Fri)
X27: X-ray Diffraction Study of Proteins, 23-ID (Wed/Fri)
X28: 3-D Reciprocal Space Diffraction, 33-BM-C (Tues/Wed/Thurs/Fri)

Neutron Experiment Descriptions

Group 1: Inelastic Neutron Scattering (N1-N5)

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

Spin wave and phonon dispersion in Fe-Ga solid solutions

Triple axis spectroscopy (TAS) is a versatile technique for measuring the scattering function in energy and momentum space. These measurements allow one to probe the static and dynamic properties of a wide range of materials. The TAS team will demonstrate how to carry out a TAS experiment. This tutorial will cover single crystal sample alignment and how to set up typical elastic and inelastic scans in SPICE (Spectrometer Instrument Control Environment), using Fa-Ga alloy single crystal samples as an example. These materials exhibit giant magnetostriction and are of tremendous scientific and technological interest for use in devices such as actuators, transducers, and sensors.

N2: 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 $[H_2NC(dma)_2][BETI]$.

N3: HYSPEC Hybrid Spectrometer, 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 demonstrate using previously collected data the use of XYZ-polarization analysis 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.

N4: VISION Neutron Spectroscopy, 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.

N5: SEQUOIA Fine-Resolution Fermi Chopper Spectrometer, SNS BL17

Dynamics of metal hydride systems: Harmonic oscillators and beyond

The hydrogen in zirconium hydride (ZrH2) sits at the interstitial positions between the zirconium. At the simplest description, the energy levels are the same as a particle in a potential well. The aim of this experiment is to measure the vibrational spectrum of ZrH_2 as a function of energy and wavevector transfer, and determine how well it conforms to the predictions of the scattering from a harmonic oscillator. Practical applications of sample preparation, data collection and analysis will be given to generate the scattering function $S(Q,\omega)$ from the data. This will be compared to theoretical predictions based on the harmonic oscillator description, with a discussion of what may cause any discrepancies. As time permits, other metal hydrides will be measured to highlight differences in their energy spectra.

Group 2: Neutron Diffraction (N6-N13)

N6: Powder Diffractometer, HFIR HB-2A

Solving the magnetic structure of a chiral symmetry spin Ising system

The POWDER neutron diffractometer is an instrument optimized for studying complex magnetic systems under a wide variety of sample environment conditions (e.g. temperatures from 0.03 to 1800 K, magnetic fields up to 8 T and pressures up to 2 GPa). In this demonstration we will start with a tour of the beamline, describing its components, demonstrating its operation, and discussing the available sample environments. We will then look at data collected on the chiral pyrogermanate

$Er_2Ge_2O_7$ at ultra-low temperatures and under an applied magnetic field. A low temperature antiferromagnetic transition will be identified, and Rietveld refinements will be performed to model both the nuclear and magnetic scattering. To solve the magnetic structure, students will be shown how to determine a magnetic ordering vector in a powder sample and then introduced to the tools of representational analysis to generate possible magnetic structures and come to a best fit model. The solution will be discussed within the context of the physics of the system and the implications of the solved structure for the magnetic spin-Hamiltonian will be considered.

N7: 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. WAND^2 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.

N8: 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.

N9: 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.

N10: VULCAN Engineering Materials Diffractometer, SNS BL-7

In-situ neutron diffraction measurement of intergranular strain evolution in 316 stainless steel under uniaxial loading

Anisotropic materials such as stainless steels will develop strong intergranular strains in the regime of plastic deformation. Neutron diffraction allows strain/stress measurement at depth by its high penetration through most engineering materials. The lattice strains of different lattice plane can be calculated by Bragg peak shift with respect to zero strain/stress a reference. At the Spallation Neutron Source, VULCAN can probe changes of lattice strain of all possible *hkl* directions under in-situ loading. The neutron data will be separated and reduced based on the load intervals. Single peak refinement will be used for analyzing the intergranular strains of (111), (200), (220) and (311) lattice planes in the material under uniaxial loading. Through this practice, students will learn in-situ loading neutron diffraction measurement set-up at VULCAN; lattice strain data calculation from diffraction pattern using VDRIVE software and understand the nature of intergranular strain evolution of material under loading.

N11: 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 a single crystal of Bixybite $(Mn_{1-x}Fe_x)_2O_3$. 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.

N12: 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 learn how 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 on Powgen time-of-flight (TOF) neutron diffraction data using GSAS-II. Exercises will include

- Sample 1: A simple structure (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: Refine magnetic structure of Quasi-One-Dimensional van der Waals CrSbSe3 Sample 4: Finally, those who get through the first three examples will be able to learn to refine a more complex structure. We will look at two models to determine the true crystal structure of Ba_2CuWO_6, which shows a Jahn-Teller distortion.

N13: 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 TOPAZ ReductionGUI. The structure will be refined using JANA2020. The option to refine the neutron structure in SHELX-2019 and GSAS will also be explored.

Group 3: Neutron Imaging (N14)

N14: TOF Imaging, SNS BL3

First wavelength-dependent neutron grating interferometry at the Spallation Neutron Source

For this experiment, the students will participate in a collaborative research project on time-of-flight imaging at SNS's SNAP beamline. The project focuses on the demonstration of neutron grating interferometry (nGI) at the SNS using a symmetric Talbot-Lau grating system provided by the Paul Scherrer Institute (PSI) neutron imaging team. This research is an international collaborative effort between three neutron scattering facilities located at ORNL, the Paul Scherrer Institute (PSI), and the Forschungsneutronenquelle Heinz Maier-Leibnitz (FRM-II).

As demonstrated at several facilities, nGI systems can provide unique information based on the phase and amplitude shift of the neutron wave that is being propagated through a sample. These systems are made of three gratings: the source grating, or G_0, provides the coherence of the neutron source; the phase grating, G_1, induces a phase shift; and the absorption grating, G_2, is necessary since the detector resolution is not sufficient to observe the fringe pattern. Hence, G_1 is often stepped perpendicularly to the beam so that the neutron wave modulation can be measured on the detector pixel.

One of the figures of merit of an nGI setup is its autocorrelation length or dark field length, x, which determines the feature size sensitivity the setup can achieve. A broader dark field length means a broader achievable length scale with the nGI. The dark field length, x, is given by $x_{GI} = lL_s/p_1$, where l is the neutron wavelength, L_s is the sample-to-detector distance and p_1 is the G_1 grating period. Since p_1 is fixed, for a fixed wavelength, L_s needs to be increased to cover a significant autocorrelation length range, leading to a decrease in image resolution. Since the SNS uses a broad range of neutron wavelengths, the range of dark field length, x, can be much broader than at reactor facilities without having to move the sample away from the detector, thus without compromising spatial resolution. The symmetrical nGI system can accept a broad range of neutrons, thus making it ideal for the SNS VENUS beamline, currently under construction. This instrument capability commissioning is also applicable to CUPI^{2}D, the future neutron imaging beamline at the SNS Second Target Station (STS). Based on analytical calculations, we expect this nGI experiment to provide the broadest dark field length, x, achieved at a neutron source, between ~ 100 nm and 5 mm.

Group 4: Large-Scale Structures (N15-N20)

N15: GP-SANS General Purpose Small-Angle Neutron Scattering, HFIR CG-2

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.

N16: Bio-SANS Biological Small-Angle Neutron Scattering, HFIR CG-3

Methods for interpreting biological small-angle neutron scattering data

Contrast-matching SANS is widely used to characterize a wide variety of soft and biological matter systems. The large difference in coherent scattering lengths of hydrogen and deuterium nuclei enables unique measurements that are not possible by other techniques. By varying the composition between fully protonated and fully deuterated solvents (such as H_2O and D_2O), one can vary the average scattering length density of the solvent and hence alter the contrast between the individual components in a complex system and surrounding medium. To demonstrate the strength of contrast variation, in this experiment, we will measure human serum albumin protein (HSA) dissolved in a series of H_2O/D_2O solvent ratios (0 – 100%) to demonstrate how changing the contrast between HSA and the solvent impacts the scattering signal. We will demonstrate different analysis approaches that can be applied to the SANS data. We will apply simple methods like Guinier Analysis for determining radius of gyration, Kratky plots for protein flexibility. Using contrast variation data of HSA, we will determine the contrast matching solvent condition to completely mask protein signal. For advanced analysis, we will apply *ab initio* methods using the ATSAS package to obtain solution structures of the protein like the dummy atom model or ensemble of models and investigate how its solution structure compares to its crystal structure.

N17: EQ-SANS Extended Q-Range Small-Angle Neutron Scattering, SNS 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.

N18: MAGREF 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.

N19: LIQREF 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.

N20: 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.

Group 5: Complex Sample Environments (N21-N25)

N21: High Pressure Science, Shull Wollan Center & SNS BL-3

Pressure-induced phase transitions of water ice

We will make use of various high pressure capabilities of SNS and HFIR through exploring the phase diagram of water ice. Firstly, we 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^{o}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 we will illustrate in the large diamond cell. This experiment will take place at SNAP, the SNS's dedicated high pressure diffractometer. Secondly, we 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. This will be performed in the high pressure, high temperature laboratory in the Shull Wollan Center (SWC) next to SNS. Finally, a large variety of high pressure science is performed across SNS and HFIR often even in conjunction with cooling for magnetic fields. We will also show you the high pressure facilities with Sample Environment and the various capabilities used across the instrument suite.

N22: High Temperature, SNS Sample Environment

Sample preparation, planning, and operation of high temperature vacuum furnaces

The Sample Environment (SE) group in neutron sciences at ORNL operates several different high-temperature devices, ranging from room temperature up to over 2000^{o} C, in support of neutron scattering experiments. For this experiment, students will learn how the different furnaces operate and gain knowledge on the different internal components of the furnaces. Students will have the opportunity to see a conventional furnace that is disassembled, and to assemble some main components of the systems in a hands-on exercise. 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, 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. Additionally, students will learn how different levitation techniques are possible, and which ones are currently implemented at the SNS. Students will be able to experience the sample preparation process, the mechanical mechanisms, and operation of an aerodynamic levitator. If time allows, they will then be able to attempt levitation melting in an electrostatic levitator. Experiments at high temperature require special considerations for sample preparation, operation, and safety; the discussion of how to coordinate with sample environment and beamline staff to prepare for these experiments from the proposal stage through beamtime will follow.

N23: Low temperature, SNS Sample Environment

In situ Resonant Ultrasound Spectroscopy probe for Neutron Scattering

As an example of new "multimodal" experimental capabilities, this practice involves room temperature measurement and data analysis with the new *in situ* Resonant Ultrasound Spectroscopy (RUS) probe.

RUS is being integrated with low and high temperature sample environment to expand the options for in-situ characterization of materials during neutron scattering experiments. The RUS technique provides high-precision, non-destructive, neutron-scattering-compatible measurements of the elastic constants and sound-attenuation in materials. In addition, the ultrasonic response in the vicinity of phase transitions enables a high-precision tracking of the sample state in-situ, more precisely than through temperature or magnetic field sensors.

The students will learn how to perform measurements of elastic moduli exploring the sensitivity of the probe with small changes of temperature and to calculate elastic moduli from measured resonance modes.

N24: Soft Matter, Shull Wollan Center

Preparation and Characterization of Lipid Model Membranes

Biological membranes are dynamic, supramolecular assemblies that are primarily composed of lipids, proteins, and carbohydrates. They exhibit dynamics ranging from individual molecular motions to collective membrane fluctuations. Because of their complexity, the studies of biological membranes using neutron and X-ray scattering rely on the use of model membrane systems made up of a few different types of biomolecules. Typical model membrane systems used in scattering experiments consist of multilamellar and monodisperse unilamellar vesicles (MLVs and ULVs, respectively). In this workshop, we will prepare MLVs and ULVs. ULVs are prepared via extrusion of MLVs through polycarbonate membrane filters with defined pore sizes. We will characterize the MLVs and ULVs using dynamic light scattering (DLS) to determine their diameter and polydispersity and differential scanning calorimetry (DSC) to determine their phase transitions. These sample preparation and characterization techniques are crucial for the success of neutron and X-ray scattering experiments. We will then take a representative SANS and SAXS data sets and demonstrate how to analyze them.

X-ray Experiment Descriptions

Group 1 (X1-X3): Imaging Experiments

X1: High-Energy X-ray Diffraction Microscopy and Strain Measurement, 1-ID-E (Wed/Fri)

Characterizing polycrystalline materials using in-situ high energy diffraction microscopy and powder diffraction techniques (high-energy x-ray diffraction, high-energy diffraction microscopy, HEDM, in situ loading, multi-modal imaging)

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 (Tues/Thurs/Fri)

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

Francesco De Carlo and Pavel Shevchenko

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: X-Ray Fluorescence Microscopy, 2-ID-E (Tues/Wed)

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.

Group 2 (X4-X8): X-ray Spectroscopy Techniques

X4: High-resolution X-ray Fluorescence, 1-BM-C (Wed/Thurs)

High-resolution X-ray fluorescence with Superconducting Quantum Sensors

Orlando Quaranta, Tejas Guruswamy, Nino Miceli

Superconducting quantum sensors represent the cutting edge of the high-resolution, high-sensitivity photon detection technology. A particular type, the Transition Edge Sensors (TESs), are now being used at beamlines for various types of X-ray fluorescence (XRF) experiments. This technique measures the energy of the photon reemitted by a sample when excited with an X-ray beam of suitable energy, allowing the identification of the chemical composition of complex samples, with possibility to extract information of the relative quantities of the components. The ability to precisely measure the fluorescence photon energies is consequently crucial, TESs represent the best detector technology available for this task. XRF measurements of complex samples will be performed with both a TES instrument and a commercial detector to understand the limits and point of strength of the two technologies.

X5: X-ray Absorption Spectroscopy, 5-BM-D (Tues/Thurs)

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.

X6: Extended X-ray Absorption Fine Structure (EXAFS), 10-BM-B and 10-ID-B (Fri/Fri)

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.

X7: Fundamentals of Beamline Operation and XAFS, 20-BM (Tues/Wed/Thurs)

Fundamentals of beamline operation and XAFS examples

Shelley Kelly, 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/.

X8: Angle-Resolved Photoemission Spectroscopy, 29-ID (Wed/Fri)

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

Jessica McChesney

Angle-resolved photoemission spectroscopy (ARPES) is one of the quintessential tools for investigating the electronic structure of crystalline materials and has been used to characterize the collective behavior of the electrons in a wide variety of materials exhibiting the superconductivity, topological quasiparticles and density waves. Spectra are obtained by measuring the kinetic energy and emission angle of photoemitted electrons; via conservation laws we are able to map this back to the energy dispersion (energy-momentum distribution) and Fermi surface of the electrons within the solid. During the practical students will be able to collect ARPES and core level x-ray photoemission spectroscopy (XPS) data on the topological insulator Bi_2Se_3. These measurements will demonstrate how photoemission can be used to determine not only the oxidation state of the constituent elements, but also how the many-body effects are manifest as changes to low-laying electronic states.

Group 3 (X9-X13): Resonant/Magnetic Scattering/Spectroscopy Techniques

X9: X-ray Magnetic Spectroscopy, 4-ID-D (Thurs/Fri)

Magnetic proximity effect studied using XMCD

Yong Choi, Joerg Strempfer

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.

X10: X-ray Magnetic Scattering, Helicity Dependence, 4-ID-D (Tues/Wed)

Diffraction with circularly polarized x rays to probe chiral spin structure.

Joerg Strempfer, Yong Choi

Chiral structures are prevalent in biological and condensed matter physics materials. Taking advantage of the interaction between the chiral structures and the helicity of circularly polarized incident x-rays, diffraction with circularly polarized x-rays allows probing chiral or cycloidal structures in crystalline samples. In this tutorial, we will investigate the cycloidal spin structure present in a Dy single crystal. We will measure the satellite peaks arising from the periodic spin winding and verify the magnetic origin of the observed peaks by monitoring the polarization dependence.

X11: X-ray Resonant Magnetic Scattering, 6-ID-B (Tues/Wed/Thurs/Fri)

X-ray resonant magnetic scattering (XRMS)

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.

X12: Resonant Soft X-ray Scattering, 29-ID (Tues/Thurs)

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

Fanny Rodolakis

Nanomaterials with quantum properties are at the forefront of physics research because these are promising to revolutionize electronic devices based on electronic conduction at low power. While the silicon is the workhorse in most electronics that we use today (portable electronics, sensors and detectors), its heat generation is limiting the growth of this industry in future when it is pursued at small nanoscale dimensions. Interestingly, in an alternative strategy, it is possible to consider other materials with different ways to control the conductivity in future electronic devices. One class of such materials are thin film oxide materials that have different bonds than silicon between atoms, which allow interesting patterns for the charge and electronic orbital around atoms them that are responsible for conduction. These ordered states inside the material are the key for control of conductivity and cannot be obtained in traditional materials like silicon.

Soft X-ray scattering, which is the combination of X-ray scattering and X-ray absorption spectroscopy, is uniquely positioned to reveal these charge and orbital orderings in oxide materials, which is not possible with other techniques. It offers a unique element, site and valence specific probe to study spatial modulations of charge, spin and orbital degrees of freedom in solids on the nanoscopic length scale. In this experiment we tune the X-rays to the orbital state of the Mn atom inside the $La_{0.67}Sr_{0.33}MnO_3$ thin film to perform a visualization of the orbital ordering formation.

X13: Resonant Inelastic X-ray Scattering, 27-ID-B (Tues/Wed/Thurs/Fri)

Elementary excitations in iridium compounds by RIXS

Mary Upton, Jung Ho Kim, Diego Casa

Resonant inelastic x-ray scattering (RIXS) exploits a resonant enhancement of low energy scattering cross-section near the element absorption edge. Elementary excitations of spin, orbital and charge degrees of freedom in transition metal compounds are probed and those dynamics are studied in the RIXS measurements, which provides a microscopic understanding of the system in study. In this experiment, we will understand the mechanism of the RIXS spectrometer, showcase how RIXS spectrum of an iridium compound near the Ir L3 absorption edge varies depending on the incident x-ray energy and the momentum transfer.

Group 4 (X14-X17): Coherence Based Techniques

X14: X-ray Ptychography Imaging, 2-ID-D (Tues/Wed)

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.

X15: X-ray Photon Correlation Spectroscopy, 8-ID-I (Tues/Wed)

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.

X16: Coherent Bragg Rod Analysis (COBRA), 12-ID-D (Thurs/Fri)

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.

X17: Coherent X-ray Diffraction Imaging, 34-ID-C (Tues/Thurs)

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.

Group 5 (X18-X20): Small-Angle X-ray Scattering

X18: Grazing Incidence Small-Angle X-ray Scattering (GISAXS), 8-ID-E (Thurs/Fri)

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.

X19: Small-Angle X-ray Scattering, 12-ID-B (Tues/Wed)

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.

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

Quantitative Determination of Spatial Elemental distribution in nanomaterials using Anomalous Small Angle X-ray Scattering (ASAXS)

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

In this experimental session, we will demonstrate some of the basic concepts of Anomalous Small Angle X-ray Scattering (ASAXS) in determining the distribution of an element of interest within and around nanomaterials. Emphasis will be put on 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 an ASAXS experiment on core-shell nanoparticles.

Group 6 (X21-X24): Diffraction-1

X21: Energy Dispersive X-ray Diffraction, 6-BM-A (Tues/Thurs)

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.

X22: Synchrotron Powder Diffraction, 11-BM & 17-BM (Tues/Thurs)

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 participate in an in-situ experiment at 17-BM and discuss with the beamline staff.

X23: Pair Distribution Function, 11-ID-B (Tues/Wed)

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.

X24: Grazing-Incidence Pair Distribution Function, 11-ID-C (Thurs/Fri)

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

Olaf Borkiewicz

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.

Group 7 (X25-X28): Diffraction-2

X25: Crystal Truncation Rod Scattering, 13-ID-C (Fri)

Crystal Truncation Rod Diffraction for Atomic-Scale Surface Measurement

Joanne 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 demonstrate all aspects of CTR data collection and analysis. Participants will 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.

X26: High-Pressure Powder Diffraction, 16-BM-D (Wed/Fri)

Pressure-induced phase transition in ZnO

Ross Hrubiak and Dimitry 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/m2), 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.

X27: X-ray Diffraction Study of Proteins, 23-ID (Wed/Fri)

X-Ray Crystallography of Proteins

Michael Becker

X-ray crystallography is an extremely valuable method for determining 3-dimensional structures of proteins and other bio-macromolecules at or near atomic resolution. Since pioneering work by

Kendrew and Perutz in the 1950s, over 145,000 X-ray-derived protein structures have been deposited in the Protein Data Bank (https://www.rcsb.org/), including recent critical structures related to COVID-19, for example. Here, we will collect X-ray diffraction data from various protein crystals using advanced software tools at GM/CA@APS (https://www.gmca.aps.anl.gov/), as well as demonstrate post-collection data-analysis steps, including data processing, calculation of electron-density maps, and building of molecular models. Discussions may include a review of the method, as well as considerations on its relationship to other structure-determination methods, and on the direction of advances, such as serial crystallography

X28: 3-D Reciprocal Space Diffraction, 33-BM-C (Tues/Wed/Thurs/Fri)

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.