Neutron Tutorials:

Group 1 (N1-N3): Inelastic Neutron Scattering

N1: CTAX and HB-1 Triple-Axis Spectrometers, HFIR (Mon, Tue, Fri)
N2: HYSPEC Hybrid Spectrometer, SNS (Thu, Fri)
N3: ARCS Wide-Angular Range Chopper Spectrometer, SNS (Tue, Thu, Fri)

Group 2 (N4-N6): Chemical Spectroscopy

N4: BASIS Backscattering Spectrometer, SNS (Mon, Tue, Fri)
N5: VISION Vibrational Spectrometer, SNS (Mon, Tue, Thu)
N6: NSE Neutron Spin Echo Spectrometer, SNS (Thu, Fri)

Group 3 (N7-N11): Neutron Powder Diffraction

N7: HB-2A Powder Diffractometer, HFIR (Mon, Tue, Thu)
N8: HIDRA Engineering Materials Diffractometer, HFIR (Tue, Thu, Fri)
N9: NOMAD Nanoscale-Ordered Materials Diffractometer, SNS (Mon, Tue, Thu)
N10: VULCAN Engineering Materials Diffractometer, SNS (Mon, Thu)
N11: POWGEN Powder Diffractometer, SNS (Mon, Thu, Fri)

Group 4 (N12-N15): Neutron Single Crystal Diffraction

N12: WAND^2 Wide-angle Neutron Diffractometer, HFIR (Mon, Tue, Fri)
N13: DEMAND Single Crystal Diffractometer, HFIR (Mon, Thu, Fri)
N14: CORELLI Elastic Diffuse Scattering Spectrometer, SNS (Mon, Tue, Thu)
N15: TOPAZ Single Crystal Diffractometer, SNS (Tue, Thu)

Group 5 (N16): Neutron Imaging

N16: MARS Multimodal Advanced Radiography Station, HFIR (Thu, Fri)

Group 6 (N17-N19): Small-Angle Neutron Scattering

N17: GP-SANS General Purpose Small Angle Neutron Scattering, HFIR (Mon, Tue, Thu)
N18: Bio-SANS Biological Small-Angle Neutron Scattering, HFIR (Mon, Tue)
N19: EQ-SANS Extended Q-Range Small-Angle Neutron Scattering, SNS (Mon, Tue, Fri)

Group 7 (N20-N21): Neutron Reflectometry

N20: MAGREF Magnetism Reflectometer, SNS (Mon)
N21: LIQREF Liquids Reflectometer, SNS (Mon, Tue)

Group 8 (N22-N24): Complex Sample Environments

N22: SNAP Spallation Neutrons at Pressure, SNS (Mon, Tue, Thu)
N23: High Temperature, SNS (Tue, Thu, Fri)
N24: Resonant Ultrasound Spectroscopy, SNS (Thu, Fri)

Group 9 (N25): Neutron Data Science

N25: Data Science for Neutron Scattering (Fri)

X-ray Tutorials:

Group 1 (X1-X4): Imaging

X1: High Energy X-ray Diffraction Microscopy and Strain Measurement (Wed, Fri)
X2: X-ray Tomography
X3: X-ray Fluorescence Microscopy (Fri)
X4: X-ray Reflection Interface Microscopy (Tue, Thu)

Group 2 (X5-X7): X-ray Spectroscopy

X5: Extended X-ray Absorption Fine Structure (EXAFS) (Wed, Fri)
X6: Fundamentals of Beamline Operations and X-ray Absorption Spectroscopy
X7: Angle-Resolved Photoemission Spectroscopy (Tue, Wed, Thu)

Group 3 (X8-X10): Inelastic X-ray Scattering

X8: Nuclear Resonant Inelastic X-ray Scattering and Nuclear Forward Scattering (Fri)
X9: High-Resolution Inelastic X-ray Scattering (Tue, Thu)
X10: Resonant Inelastic X-ray Scattering

Group 4 (X11-X12): Resonant/Magnetic Scattering/Spectroscopy

X11: X-ray Magnetic Spectroscopy (Tue, Wed)
X12: X-ray Magnetic Scattering (Thu, Fri)

Group 5 (X13-X16): Coherence Based Techniques

X13: X-ray Ptychography Imaging (Tue, Wed)
X14: X-ray Photon Correlation Spectroscopy (Tue, Wed)
X15: Coherent Bragg Rod Analysis (COBRA) (Tue, Thu)
X16: Coherent X-ray Diffraction Imaging (Tue, Thu)

Group 6 (X17-X20): Small-Angle X-ray Scattering

X17: Grazing Incidence X-ray Scattering (Wed, Thu)
X18: Ultra-Small-Angle Scattering (Tue, Wed, Thu)
X19: Anomalous Small-Angle X-ray Scattering (Tue, Thu)
X20: Studying hierarchical materials with SAXS, MAXS and WAXS (Tue, Thu)

Group 7 (21-X24): Diffraction-I

X21: Energy Dispersive X-ray Diffraction (Tue, Thu)
X22: Synchrotron Powder Diffraction (Tue, Thu)
X23A: High-Pressure Powder Diffraction, Paris-Edinburgh cell
X23B: High-Pressure Powder Diffraction, Diamond Anvil cell
X24: Grazing-Incidence Pair Distribution Function

Group 8 (X25-X28): Diffraction-II

X25: Macromolecular Crystallography at Synchrotron Light Sources (Fri)
X26: 3D Reciprocal Space Diffraction
X27: Time-Resolved X-ray Diffraction
X28: Investigation of Quantum Solids with Dark Field Microscopy (Wed, Fri)

Group 9 (X25-X28): Detectors, Instrumentation, and Controls

X29: High-Resolution X-ray Fluorescence (Tue, Thu)
X30: Beamline Control and Data Acquisition with BlueSky (Wed, Fri)

2023 Neutron Tutorials Descriptions

Group 1 (N1-N3): Inelastic Neutron Scattering

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

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: HYSPEC Hybrid Spectrometer, SNS

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.

N3: ARCS Wide-Angular Range Chopper Spectrometer, SNS

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.

Group 2 (N4-N6): Chemical Spectroscopy

N4: BASIS Backscattering Spectrometer, SNS

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

N5: VISION Vibrational Spectrometer, SNS

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.

N6: NSE Neutron Spin Echo Spectrometer, SNS

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 3 (N7-N11): Neutron Powder Diffraction

N7: HB-2A Powder Diffractometer, HFIR

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.

N8: HIDRA Engineering Materials Diffractometer, HFIR

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.

N9: NOMAD Nanoscale-Ordered Materials Diffractometer, SNS

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

Lattice strain determination of intergranular strain and phase transformation evolution in 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, and the strain induced phase transformation facilitates both work hardening and ductility of the material. At the Spallation Neutron Source, VULCAN can probe changes of lattice strain of all possible *hkl* directions and monitor the phase transformation under in-situ loading. This practical will focus heavily on the complex data analysis, specifically on lattice strain data calculation from diffraction pattern using VDRIVE software. 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 and Rietveld refinement will be used for phase analysis. The students will also have the opportunity to see the VULCAN instrument and its unique set-ups for strain measurements including dedicated load frames. Through this the students will understand the nature of intergranular strain evolution and strain induced phase transformation of material under loading.

N11: POWGEN Powder Diffractometer, SNS

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.

Group 4 (N12-N15): Neutron Single Crystal Diffraction

N12: WAND² Wide-angle Neutron Diffractometer, HFIR

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.

N13: DEMAND Single Crystal Diffractometer, HFIR

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.

N14: CORELLI Elastic Diffuse Scattering Spectrometer, SNS

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.

N15: TOPAZ Single Crystal Diffractometer, SNS

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 Olex2 will also be explored.

Group 5 (N16): Neutron Imaging

N16: MARS Multimodal Advanced Radiography Station, HFIR, and VENUS, SNS

Seeing the inside

Given the unique interactions of neutrons with materials, neutron imaging is a complementary technique to X-ray imaging that can spatially resolve internal features/distributions non-destructively in bulk systems. Neutrons' high sensitivity to light elements such as H and Li enables the ability to visualize the water transport in plant roots or the Li transport in an operating battery. By participating in this experiment, you will gain the experience of running a neutron imaging experiment, including 2D radiographs and 3D tomography, and performing data analysis on a material science sample. Additionally, you will be able to tour the SNS's upcoming Versatile Neutron Imaging Instrument (VENUS) that focuses on time-of-flight imaging to make use of neutron scattering Bragg features for improved contrast and phase identification and is posed to join the user program in 2024.

Group 6 (N17-N19): Small-Angle Neutron Scattering

N17: GP-SANS General Purpose Small Angle Neutron Scattering, HFIR

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.

N18: Bio-SANS Biological Small-Angle Neutron Scattering, HFIR

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 maltose-binding protein (MBP) dissolved in a series of H_2O/D_2O solvent ratios (0 – 100%) to illustrate how changing the contrast between MBP and the solvent impacts the scattering signal. We will employ different analysis approaches that can be applied to the SANS data, including simple methods like Guinier Analysis

for determining radius of gyration and Kratky plots for protein flexibility. Using contrast variation data of MBP, we will identify the solvent condition that achieves contrast matching, completely masking the protein signal. For more advanced analysis, we will employ ab initio methods using the ATSAS package to obtain solution structures of the protein, such as the dummy atom model or ensemble of models, and investigate how its solution structure compares to its crystal structure.

N19: EQ-SANS Extended Q-Range Small-Angle Neutron Scattering, SNS

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.

Group 7 (N20-N21): Neutron Reflectometry

N20: MAGREF Magnetism Reflectometer, SNS

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.

N21: LIQREF Liquids Reflectometer, SNS

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 [1]. 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.

1. A. Karim, et al., *Short-time relaxation and polymeric interfaces.* Physical Review B **42,** 6846 (1990).

Group 8 (N22-N24): Complex Sample Environments

N22: SNAP Spallation Neutrons at Pressure, SNS

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 tour of the instrument and become familiarized with its components and how they can be configured for specific experiments. Students will run a DAS 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.

N23: High Temperature Group, 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.

N24: Resonant Ultrasound Spectroscopy, SNS Sample Environment

In situ Resonant Ultrasound Spectroscopy probe for Neutron Scattering

As an example of new "multimodal" experimental capabilities, this practice involves 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.

Group 9 (N25): Neutron Data Science

N25: Data Science for Neutron Scattering, SNS

Data science permeates the field of neutron scattering at all levels, from controlling instrumentation on the beamline, through data reduction and into data analysis. This tutorial targets beginner-to-intermediate level and aims to provide an *interactive* introduction to some of the cornerstones of data science including:

- creating and managing repositories using git and accessing code and collaborating with GitHub,
- What is conda (and why is it important)?
- An overview of the vast landscape of useful tools (and how to import these into your own code)
- An introduction to python scripting for neutron science

After this introduction, we will offer a series of parallel interactive tutorials to cover a range of abilities. These tutorials will focus on some specific applications in the field of neutron scattering (such as the mantid framework), as well as some more general themes (for example, fitting data).

2023 X-ray Tutorials Descriptions

Group 1 (X1-X4): Imaging

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 particular, the high-energy diffraction microscopy (HEDM) program at the Advanced Photon Source (APS) has made significant progress, providing users with in situ, non-destructive and multi-modal 3D microstructure mapping capabilities that can extract multiple attributes like strain, crystallographic orientation, and shape of individual grains or aggregate of grains.

In this tutorial, we will

- Introduce the HEDM program and planned capabilities post APS-U.
- Discuss different flavors of HEDM with application and data examples.
- Discuss the types of information that can be extracted from such data set.

X2: X-ray Tomography, 2-BM

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 tutorial, we will evaluate the differences between using an absorption vs a propagation phase contrast protocol using previously collected datasets of wood and polymer samples. We will also show how X-ray tomography is performed including basic tomography principles, sample mounting and alignment, data collection, data analysis and 3D rendering. Finally, we present our most recent developments in streaming data collection and real-time tomography reconstruction.

X3: X-ray Fluorescence Microscopy, 2-ID-E

X-ray fluorescence imaging of photo-voltaic samples and battery particles

Olga Anitpova and Lu Xi Li

Ptychography, a scanning coherent lens-less imaging technique, has revolutionized the study of extended samples by offering spatial resolution unrestricted by the illumination beam size. X-ray ptychography, in particular, has emerged as a powerful tool across various disciplines, from biology to energy materials and microelectronics. In this enlightening tutorial session on X-ray ptychography, we will provide an overview of this technique and take you on a virtual tour of the ptychographic instruments within the Microscopy group at the APS.

X4: Reflection Interface Microscopy, 33-ID

X-ray reflection interface microscopy (XRIM)

Zhan Zhang

X-ray Reflection Interface Microscopy (XRIM) is an X-ray imaging tool on the diffraction condition, sometimes also referred to as full-field diffraction imaging and it is very similar to Dark Field X-ray Microscopy (DFXM).

As a 'full field' imaging method, XRIM directly shows the structure variation of the sample within the field of view, without rastering the sample or the beam. The images are easy to interpret, therefore real-time, in-situ measurements are quite straightforward to do.

As a 'dark field' method, XRIM utilizes accessible diffraction conditions to offer contrast in the image. We can either go through multiple scattering conditions to make different structure 'visible', or we can stay at a given scattering condition to monitor the change of domains.

As a 'interface' method, XRIM is primarily focused on the surface/interface region, which includes mostly the thin film domain structure evolution and their response to various external stimuli, such as temperature, electrical or magnetic field.

We will discuss the basic ideas of surface diffraction and X-ray full-field imaging, as well as the actual beamline setup. A few past experiments as examples will be discussed, including the very first XRIM measurement of imaging single unit cell height steps on the surface of orthoclase; the domain structure of PbTiO3 thin film on SrTiO3 substrate and its evolution as a function of temperature; the monoclinic domain distribution of SrRuO3 thin film on SrTiO3 substrate and its response to the applied electrical field; etc.

Group 2 (X5-X7): X-ray Spectroscopy

X5: 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.

X6: Fundamentals of Beamline Operation and XAFS, 20-BM

X-ray Absorption Fundamentals: Beamline Setup, Data Collection and Analysis

Chengjun Sun, Mikhail A. Solovyev, Shelly D. Kelly, Yanna Chen, and Debora Motta Meira

Most XAS beamlines are optimized by beamline scientists. This tutorial gives students the opportunity to experience the importance of the setup parameters firsthand by comparing the effects on the measured spectra, learning how to spot these problems in their spectra. 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 through a pre-recorded video, including following tasks: Task 1 – characterize the harmonic content of the beam; Task 2 – characterize the energy resolution; Task 3 – insert harmonic rejection mirror; Task

4 – Cu EXAFS and XANES; and Task 5 – polarization dependence of High Tc superconductors. After finishing these tasks, we will demonstrate the linear combination fitting of both the EXAFS and XANES by fitting the data for an arbitrarily oriented sample; We will also model Cu foil EXAFS with FEFF theory to demonstrate theoretical fitting. Analysis will be done using the Demeter software that can be downloaded from http://bruceravel.github.io/demeter/. Prior experience in synchrotron experimentation is desirable.

X7: Angle-Resolved Photoemission Spectroscopy, 29-ID

Angle-resolved photoemission spectroscopy (ARPES)

Jessica McChesney

Angle-resolved photoemission spectroscopy (ARPES) is one of the quintessential tools for investigated the electronic structure of crystalline materials and has been used to investigate and characterize the collective behavior of the electrons in a wide variety of materials including 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 this tutorial, students will be given a tour of the beamline including how to manipulate a sample in ultra-high vacuum. We then go through an example experiment showing how to collect angle-resolved photoemission spectroscopy (ARPES) and core level X-ray photoemission spectroscopy (XPS) data on the topological insulator Bi2Se3. We will then discuss how these measurements 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 (X8-X10): Inelastic X-ray Scattering

X8: Nuclear Resonant Inelastic X-ray Scattering and Nuclear Forward Scattering, 3-ID

Nuclear resonant inelastic X-ray scattering and nuclear forward scattering

Michel Hu

NRIXS: Nuclear resonant inelastic X-ray scattering (NRIXS) is a spectroscopy method to study atomic vibrations and dynamics, currently done with synchrotron radiation at a few high-energy third generation facilities. It finds a wide range of applications in condensed matter physics, materials science, chemistry, biophysics, geosciences, and high-pressure researches. In an NRIXS experiment, one measures the number of nuclear resonant absorption events as a function of energy transfer from an incident X-ray beam to the sample under study. Besides the resonant enhancement so that minute sample can be studied, a unique aspect of using resonant isotopes is its isotope/atom selectivity. This means that vibrations can be probed locally in systems that have resonant isotopes in specific places, e.g., bio-molecules, catalysts, thin films, and materials under extremely high pressure. Many atomic dynamics and lattice thermodynamics information can be derived from NRIXS measurements. Phonon Density of States (DOS) characterizes lattice dynamics of a material and can be derived under the quasi-harmonic approximation. Combined with modeling and simulations, results from NRIXS can provide unique and clarifying insights into many fields of research.

NFS: Nuclear forward scattering (NFS) provides information on atomic environments by measuring the interaction between resonant nucleus and the local electric and magnetic fields. The information that can be obtained includes valence, spin state, and magnetic ordering in the material

under investigation. The method employs particular resonant nuclei (Mossbauer isotopes) that can be excited at modern synchrotron radiation facilities. It is nuclear resonant scattering measured in time domain, in contrast to conventional Mossbauer spectroscopy, which has been used to study material properties mentioned above.

X9: High-Resolution Inelastic X-ray Scattering, 30-ID

Studying phonons in single crystals using high-resolution inelastic X-ray scattering.

Ahmet Alatas

Typically, scattering experiments with X-rays or neutrons are done without energy analysis after the scattering event. Therefore, an integration of all scattered energies is done experimentally in the detector. The information extracted from these experiments is related to information on the structure in the studied system, or, more precisely, to correlation functions of the structure.

If the energy of the scattered intensity is analyzed, it is called an inelastic scattering experiment and- in addition to the structural information- dynamical properties of the system can be studied, i.e., information on correlations in time is obtained, Moreover, inelastic X-ray scattering (IXS) provides access to very rich excitation spectra; phonons, magnons, electronic excitations, plasmon and Compton scattering depending on the transferred energy (meV to several hundreds of eV).

Advanced Photon Source has Sector 30 beamline (30ID), with very high-energy resolution (1.3-1.5 meV) spectrometer, specialized for studying collective excitations (phonons) where their energies lie in the order of milli-electronvolts (meV). IXS is very important technique in applications ranging from condensed matter physics to life science and mineral physics to geophysics.

During the NX-school, momentum-resolved inelastic X-ray scattering experiments on single crystal aluminum will be demonstrated using HERIX 30 instrument located at sector 30 beamline. We will determine sound velocity and elastic constant along [00L] direction from measured dispersion curve and compare the results with the values found in the literature. Previously collected data will be used during the tutorial as in the real time experiment. It includes aligning and orienting single crystal before collecting energy spectrum.

X10: Resonant Inelastic X-ray Scattering, 27-ID-B

Resonant inelastic X-ray scattering (RIXS)

Mary Upton

Resonant inelastic X-ray scattering (RIXS) measures the energy and momentum dispersion of electronic excitations such as magnons, dd transitions and charge transfer excitations. At the 2023 tutorial sector 27 staff will give a few examples of measurements which have been performed and about the practical considerations that go into RIXS experiments. We will try to give a sense of when RIXS is the right technique to use and when other technique is more promising (for example, inelastic neutrons measurements). We would also really like to talk to NX school participants about how RIXS might contribute to their own research.

Group 4 (X11-X13): Resonant/Magnetic Scattering/Spectroscopy

X11: X-ray Magnetic Spectroscopy, 4-ID-D

X-ray dichroic spectroscopies

Daniel Haskel

In this tutorial we will revisit fundamentals and applications of X-ray dichroic spectroscopies as probes of structural, orbital, and magnetic anisotropies in condensed matter. X-ray dichroism, loosely defined as the difference in X-ray absorption between orthogonal polarization states (linear horizontal and vertical; or circular left and right), can be observed in non-magnetic (e.g. chiral, ferroelectric) and magnetic (ferro-, ferri-, anti-ferromangetic) materials. We will discuss practical aspects of generating tunable polarization states at X-ray light sources, the microscopic origin of the various flavors of dichroic effects (XLD, XMLD, XNCD, XMCD), experimental methods for measuring dichroism with high precision (polarization modulation and phase lock-in methods), and illustrate with a broad range of examples from experiments carried out at APS and elsewhere.

X12: X-ray Magnetic Scattering 4-ID-D

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

Joerg Strempfer

Resonant X-ray scattering allows probing the electronic and magnetic structure element selectively by tuning the X-ray energy to the respective absorption edges of the elements in the compound. In addition, the polarization dependent magnetic scattering cross-section offers the possibility to gain information on magnetic order by exploring the polarization properties of the X-rays available at the synchrotron radiation source. In this tutorial, we will introduce the magnetic scattering cross section and look at possibilities of how to manipulate and analyze the polarization of the x-ray beam. Current examples on how to determine moment directions in crystalline samples using variable linear polarization will be given. Finally, we will show an example on how to take advantage of the interaction between the chiral structures and the helicity of circularly polarized incident X-rays and how diffraction with circularly polarized X-rays allows probing chiral or cycloidal structures.

Group 5 (X13-X16): Coherence Based Techniques

X13: X-ray Ptychography Imaging, 2-ID-D

High-resolution X-ray Ptychographic Imaging of Microelectronics

Junjing Deng and Yi Jiang

Ptychography, a scanning coherent lensless imaging technique, has revolutionized the study of extended samples by offering spatial resolution unrestricted by the illumination beam size. X-ray ptychography, in particular, has emerged as a powerful tool across various disciplines, from biology to energy materials and microelectronics. In this enlightening tutorial session on X-ray ptychography, we will provide an overview of this technique and take you on a virtual tour of the ptychographic instruments within the Microscopy group at the APS.

To showcase the capabilities of X-ray ptychography, we will focus on microelectronics that incorporate diverse materials and features at multiple length scales. Through the utilization of a pre-recorded ptychographic dataset obtained from an integrated circuit, we will guide you through a simulated ptychography scan. Subsequently, we will perform a phase retrieval computation to

reconstruct a real-space image of circuitry structures, thereby highlighting the nanoscale resolution achievable through nondestructive imaging.

X14: X-ray Photon Correlation Spectroscopy, 8-ID-I

X-ray photon correlation spectroscopy study of dynamics in Soft and Hard matter

Qingteng Zhang, Eric Dufresne, and Suresh Narayanan

X-ray photon correlation spectroscopy (XPCS) is a well-established technique to study equilibrium and non-equilibrium fluctuations in soft and hard matter systems. XPCS has been successfully applied to study dynamics in colloidal suspensions, nanoparticle dispersion in polymers, metallic glasses, ferroelectric and superconducting materials, to name a few. 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 pixel array detector, the dynamics over a range of length scales in the range of 1000 nm – 0.1 nm can be probed simultaneously over a range of time scales in the range of 100 microseconds – 1000 seconds.

APS-U will result in a 100-fold increase in the coherence and as a result, XPCS technique will be able to study a wide range of soft and hard materials over a wide range of length and time scales, with a very high sensitivity and extending into domains such as biology and high pressure that could be envisioned till date. The new APSU XPCS feature beamline will be ready for the user community when APS-U turns on in mid-2024.

During the tutorial, we will present the fundamentals of the XPCS technique, beamline instrumentation citing scientific examples to highlight the physics that can be discerned from such experiments.

X15: 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.

X16: 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 S-ray imaging techniques. Coherent X-ray diffraction 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 tutorial we will provide an overview of Bragg coherent X-ray diffraction imaging (BCDI) and describe how to design and plan a BCDI experiment. We will then work with previously acquired data and computationally invert 3D diffraction patterns to a 3D image of a crystal. This data analysis will be done with 3D diffraction patterns from small crystals (about 300 nm) with/without defects.

Group 6 (X17-X20): Small-Angle X-ray Scattering

X17: Grazing Incidence X-ray Scattering, 9-ID-D

Investigating organic electronic materials with grazing-incidence X-ray scattering

Joseph Strzalka and Zhang Jiang

Grazing incidence X-ray scattering (GIXS) in both the small- and wide-angle regimes (GISAXS/GWAXS) has become an indispensable tool for studying the structure of thin film materials. GIXS 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. An area of particular interest and growth for our user community is the characterization of organic electronic materials, which generally exhibit a complex interrelationship between structure, processing and performance. Using previously obtained GIWAXS data from organic photovoltaic (OPV) materials as a case study, we will familiarize participants with strategies and software for the analysis of the material structure that apply to other systems, such as stretchable electronics or mixed ionic electronic conducting materials. The program will also afford an opportunity to discuss opportunities to apply GISAXS/GIWAXS for in situ or operando studies, as well as studying dynamics via grazing-incidence X-ray photon correlation spectroscopy.

Program:

1. Introduce the method and the instrument
2. Work with data from previous years
    a. Take linecuts
    b. Fit linecuts to extract peak positions and widths
    c. Evidence for paracrystallinity – peak widths increase with order
    d. Demo pole figure
3. Discuss student research plans
    a. Introduction of the new 9-ID beamline
    b. In situ/operando possibilities at beamline
    c. GI-XPCS

X18: Ultra-Small-Angle Scattering, 9-ID

Understanding microstructures in a wide variety of materials

Jan Ilavsky

Ultra-Small-Angle Scattering technique extends the Small-angle scattering range up to 5 micrometers (20 micrometers APS-U), which is critical for studies of materials where important microstructure features extend over such wide range. APS is host to world-unique USAXS-SAXS-WAXS instrument, which provides measurements over 5 decades in sizes (5micron to <1Angstrom) within about 3 minutes (<1 minute APS-U). This instrument is used by a wide range of scientific fields including: polymers, metallurgy, geology, and most materials related fields, but also food science, building materials, chemistry, on solid/liquid side, and fuel spray research on "gas" side. This instrument is one of the most versatile devices on the APS floor with extensive user community.

This hands-on tutorial will present overview of the technique, sample preparation, and show sample environments we offer to users. Most of the time will be demonstration of prior USAXS results and their analysis using Irena SAS data analysis package, authored by beamline staff.

X19: Anomalous Small Angle X-ray Scattering, 15-ID

Anomalous X-ray scattering to determine the elemental distribution within nanomaterials

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

In this experimental tutorial, 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 developed at NSF's ChemMatCARS (Sector-15, Advanced Photon Source) through a virtual experiment on core-shell type of nanoparticles.

X20 Studying hierarchical materials with SAXS, MAXS and WAXS, 5-ID-D

Studying hierarchical materials at DND station

Qing Ma, Mike Guise, and Denis Keane

DND-CAT's 5IDD station at APS specializes in Small-, Medium- and Wide-angle scattering to study materials at multiple length scales. We study a range of materials from polymers and nanomaterials to molecules in solution. We will present a tour of the 5ID beamline including details which will be improved after the APS Upgrade (virtual or in-person TBD). We will then go in-depth into the three-detector SAXS/MAXS/WAXS system and the many sample environments we have developed including an in-vacuum solution scattering system, an Instron servo-hydraulic compression/tension system, and many modalities of sample environmental control including temperature and humidity variation. We may conclude with an overview of a typical data analysis pipeline with possible hands-on analysis of synthetic data.

Group 7 (X21-X24): Diffraction-1

X21: 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) technique allows for selective measurement of material information from a discrete 3D volume within a larger bulk sample and its surrounding environment. This is achieved by using a polychromatic incident beam and measuring at a fixed scattering angle with an energy-dispersive detector. The resulting gauge volume provides an opportunity to map both phases and strain in complex samples. During the tutorial session, participants will be introduced to the fundamentals of the ED-XRD technique, given a short tour of the end-station, and shown several examples that illustrate the usefulness of the technique. These examples will 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, and examining samples confined inside a complex environment, such as a furnace or large volume, high-pressure cell. At the end of the session, participants will have the opportunity to discuss potential ED-XRD experiments that may be of interest to their research.

X22: Synchrotron Powder Diffraction, 11-BM & 17-BM

High resolution and in-situ powder diffraction data processing & 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, various in situ sample environments, data collection and preliminary data analysis. Attendees will analyze data collected before the APS Upgrade shutdown and discuss with the beamline staff.

X23-A: High-Pressure Powder Diffraction, 16-BM-D

White beam Laue diffraction from amorphous samples at high pressures in a Paris-Edinburgh cell

Tyler Eastmond

In this hands-on tutorial students will be introduced high pressure diffraction experiments including sample/cell preparation and analysis of PDF data. The nuances of constructing a PEC sample assembly will be introduced and the students will learn how to prepare a PEC experiment. Students will then use data previously obtained on an amorphous material at various pressures and will then be led as to how to unravel the PDF as a function of pressure and temperature.

To overcome the issues of restricted geometric access due to limited openings on high pressure cells (and to a lesser extent, leverage all the flux that becomes available in a bending magnet source), white beam diffraction is used. The Paris-Edinburg cell (PEC) and the diamond anvil cell (DAC) are two high pressure devices that offer complementary capabilities in terms of sample volumes, access, and pressure-temperature distributions. HPCAT facilitates both kinds of experiments on the bending magnet beamline 16BM. This tutorial will focus on PEC which is a very versatile assembly that allows one to work with mm sized samples and conduct a whole suite of high P-T experiments that include powder diffraction, radiography, bulk sound velocity measurements that allow us to determine phase boundaries, melting, viscosity, and liquid and amorphous structure measurements, amongst others, in the nominal pressure range of 0.01 – 20 GPa (1 GPa ~ 10 kbars) and ambient to 2000 K.

References:

Probing High-Pressure Structural Evolution in Polyurea with In Situ Energy-Dispersive X-ray Diffraction and Molecular Dynamics Simulations, Tyler Eastmond et al., Macromolecules 2021 54 (2), 597-608.

X23-B: High-Pressure Powder Diffraction, 16-BM-D

White beam Laue diffraction from crystalline samples at high pressures in a diamond anvil cells

Dmitry Popov

In this hands-on tutorial students will be introduced to high pressure diffraction experiments including sample/cell preparation and analysis of PDF data. Students will be introduced to the basics of sample loading in a DAC using the various capabilities HPCAT offers. They will then be lead through an exercise utilizing previously obtained high pressure Laue diffraction data to explain how this experiment can answer many questions regarding phase transitions at high pressure.

To overcome the issues of restricted geometric access due to limited openings on high pressure cells (and to a lesser extent, leverage all the flux that becomes available in a bending magnet source), white beam diffraction is used. The Paris-Edinburg cell (PEC) and the diamond anvil cell (DAC) are two high pressure devices that offer complementary capabilities in terms of sample volumes, access, and pressure-temperature distributions. HPCAT facilitates both kinds of experiments on the bending magnet beamline 16BM. This tutorial will focus on DAC which allows one to attain extremely high pressures (100 GPa or 1 Mbar) on □m sized samples and at elevated temperatures (up to 4000 K, using laser heated DACs). At HPCAT, we have a very vibrant research program that utilizes white beam Laue diffraction to understand complex issues like phase transition mechanisms and defect structure evolution under pressure. This single crystal (or multi crystal) diffraction technique overcomes the limitation imposed by a small geometric opening traditionally associated with DACs.

References:

White Laue and powder diffraction studies to reveal mechanisms of HCP-to-BCC phase transformation in single crystals of Mg under high pressure, Evgenii Vasilev et al., Sci. Reports, (2023), 13, 2173.

X24: Grazing-Incidence Pair Distribution Function, 11-ID-B

Pair distribution function analyses of high-energy X-ray data

Olaf Borkiewicz

Beamline 11-ID-B at the Advanced Photon Source has been dedicated to the collection of high-quality total X-ray scattering data suitable for pair distribution function (PDF) analyses for nearly two decades. The PDF is a histogram of all atom-to-atom correlations on a length-scale of up to several nanometers. As such it depicts the local arrangement of atoms within the sample, independent of periodicity and translational symmetry and thus can be applied to study disordered, crystalline, amorphous, nanoscale, homogeneous and heterogeneous materials alike. During the experiment, data collected using high energy X-rays (58 and 87 keV) on a series of standard materials and various samples will be used to discuss data reduction procedures, methods of PDF extractions and modelling of PDF data em24ploying various programs. In addition to traditional transmission-geometry data, we will work with data collected on amorphous and nanostructured thin films measured under grazing-incidence conditions. The latter approach significantly enhances the signal of the thin film and enables investigations unachievable through a traditional, transmission-geometry experiments. We will compare transmission geometry, flat incidence and grazing incidence data. The experiment will include lectures and hands-on exercises to cover all topics.

Group 8 (X25-X28): Diffraction-2

X25: Macromolecule Crystallography at Synchrotron Light Source, 19-ID

Macromolecule crystallography at synchrotron light source

Tutorial instructor: Dr. Youngchang Kim
Facility tour: Dr. Joseph Brunzelle

X-ray diffractions from crystals of macromolecules have been providing us with invaluable structural details in three dimensions. These macromolecules can be proteins, protein-nucleic -acid

complexes, protein assemblies, protein-small molecule drug complexes which make up all living system. This detailed structural information allows us understanding how the molecules function in living cell, how they interact, catalyze, and regulate activities of other molecules and help us to design better functioning macromolecules or small molecule drugs.

Synchrotron light source provides X-ray much brilliant than home X-ray source, so we can get reasonable diffraction signal even from very small crystals. Now at APS with state of art synchrotron source along with much advanced detectors, robots and crystallographic software, we can collect data from a small crystal, very quickly, in a matter of a few minutes, (hours to days before), process data and determine the structure.

This tutorial is to introduce macromolecule crystallography at Synchrotron light source, testing (how crystals are made), how diffraction data are collected at the synchrotron beamlines, how structures are determined from the diffraction data, how the crystal structures are analyzed in function and how the structural data are used.

X26: 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.
The topics include:

- Overview of technique, beamline tour if possible
- Designing/planning an experiment
- Sample mounting and environments
- Data reduction/analysis
- Tour

X27: Time-Resolved X-ray Diffraction, 7-ID

Time-resolved X-ray diffraction from ultrafast laser-pumped materials

Don Walko

Time-resolved X-ray diffraction is a powerful method of determining the responses of crystalline systems to ultrafast external stimuli. Laser-pump X-ray-probe experiments can be used to study acoustic waves, thermal transport, and photoinduced phase transitions. These techniques have applications in fields such as microelectronics and solar cell systems.

In this tutorial, students will be given a tour of the beamline; they will be shown the instrumentation required to direct *two* distinct beams at the sample and achieve overlap in space and in time. They will then analyze a thermal transport experiment, wherein a laser is used to heat a thin metal film grown on a transparent substrate. The time-dependent shift of the film’s Bragg peak acts as a thermometer for the film, allowing the film temperature to be measured on a sub-nanosecond timescale. From these data the conductance of the film/substrate interface will be calculated.

X28: Investigation of Quantum Solids with Dark Field Microscopy, 6-ID-C

Quantum solids under dark-field X-ray microscope

Zahir Islam

This tutorial will provide a basic introduction to dark-field X-ray microscopy (DFXM) with an emphasis on studies of quantum solids and devices. In contrast to precision X-ray diffraction from single-crystal or polycrystalline samples which provides an 'average' view of ordered materials, DFXM affords researchers real-space images of 'mesoscale' structures that are deviations from an average order. In DFXM, a Bragg diffracted beam is passed through an X-ray objective lens to form a magnified image on an area detector. By decoding spatial information on a crystal Bragg peak, a super-lattice (*e.g.*, due to a charge, magnetic, or orbital order) peak, or an epitaxial-film peak, in the form of intensity contrasts, DFXM provides a complementary and an incisive picture over many orders-of-magnitude in length scales. DFXM carried out concurrently with *in situ* multi-modal measurements such as specific heat and electrical transport measurements may allow one to directly correlate materials properties to mesoscale features. This tutorial will introduce state-of-the-art DFXM capabilities and techniques that have been developed in recent years followed by hands-on DFXM image analyses to extract location-selective information. To get the most out of this tutorial those interested should read the reference articles beforehand.

Reference:

Zhi Qiao et al., "*A large field-of-view high-resolution hard x-ray microscope using polymer optics*", Rev. Sci. Instrum. **91**, 113703 (2020); https://doi.org/10.1063/5.0011961

Jayden Plumb et al., "*Dark Field X-ray Microscopy Below Liquid-Helium Temperature: The Case of $NaMnO_2$*," arXiv:2211.09247 cond-mat.mtrl-sci (2022)

Omar Abulshohoud et al., "*A general method for multiresolutional analysis of mesoscale features in dark-field x-ray microscopy images*," arXiv:2210.15757 cond-mat.mtrl-sci (2022)

Júlia Garriga Ferrer et al., "*darfix – data analysis for dark-field X-ray microscopy*," J. Synchrotron Rad. **30**, 527(2023); https://doi.org/10.1107/S1600577523001674

Elliot Kisiel et al., "*Full-Field Nanoscale X-ray Diffraction-Contrast Imaging using Direct Detection*," arXiv:2212.07303 physics.ins-det (2022).

Group 9 (X29-X30): Detectors, Instrumentation and Controls

X29: High-resolution X-ray Fluorescence, 1-BM-C

Introduction to X-ray detector technologies and to high-resolution X-ray fluorescence with superconducting quantum sensors

Orlando Quaranta and Tejas Guruswamy

X-ray detectors perform a fundamental role in any synchrotron experiment. They collect the photons emitted by the sample under illumination and convert their properties into useful information on its atomic structure. Several types of detectors are routinely used at beamlines depending on the needs of the experiment: area detectors, fast counting, energy dispersive, etc. Typically X-ray detectors are based on semiconductors, mostly Si for lower energies and higher Z materials like Ge or CdTe for hard X-rays. Detectors of these types and more are available at beamlines and as part of the Detector Pool; we will discuss some of the available options and demonstrate typical configuration and operation using EPICS and areaDetector.

In recent years, for X-ray spectroscopy, an alternative type of detectors has made its appearance in the synchrotron environment: Superconducting Quantum Sensors. These 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 the possibility to extract the relative quantities of the components. The ability to precisely measure the fluorescence photon energies is consequently crucial, and TESs represent the best detector technology available for this task.

An introduction to the various detector technologies will be provided and representative XRF measurements of complex samples will be measured by a TES detector in a lab environment. Results will be provided to the students to be analyzed.

X30 Beamline Control and Data Acquisition with BlueSky

APS Bluesky 101

Pete Jemian

Bluesky is a python-based framework for communicating with instrumentation and data acquisition, analysis, and visualization. It was initially developed at NSLS-II and is being adopted by several other light sources including the APS. APS Bluesky 101 guides users through the first measurements at the APS using the Bluesky framework, then visualize and analyze data after the measurement. After an introduction to the simulated beamline (provisioned with components typical of many APS instruments), users will have hands-on control using Bluesky. Data acquisition will cover step scans (scaler v. motor), alignment, and area detector imaging. Data analysis will cover retrieving data from the databroker (database of recorded experiment data) for plotting and processing.

The course will use Jupyter notebooks and the Python computer language for the data acquisition and analysis steps. While not required, it is recommended that participants have at least an introductory knowledge of Python.