2024 X-ray Experiment Descriptions

Group 1 (X1-X4): X-ray Imaging

X1: High Energy X-ray Diffraction Microscopy and Strain Measurement, 1-ID-E & 20-ID

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. 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 Fluorescence Microscopy, 2-ID-E

X-ray fluorescence microscopy

Olga Anitpova and Lu Xi Li

X-ray fluorescence microscopy at 2-ID-E offers unparalleled sensitivity for measuring the distribution of trace elements in many-micrometer-thick specimens with sub-micron spatial resolution, part-per-million detection sensitivity, and the ability to acquire tomographic datasets. Samples (biological tissue sections, cells, crystals, etc.) are raster-scanned with x-ray beam and x-ray fluorescence signal is recorded for each pixel. Using those signals from elements such as Zn, Cu, Ca, K, Ti, Cr, Co, and others, we can determine their distribution within the sample. Combined with other techniques it helps understanding a variety of biological processes and pathologies, unveil aspects of battery functions, and evaluate solar cell materials. We will discuss critical aspects of sample preparation, data collection and analysis, including possible artifacts, as well as demonstrate software for XRF tomography reconstruction

Group 2 (X3-X5): X-ray Spectroscopy

X3: Extended X-ray Absorption Fine Structure (EXAFS), 10-ID-B

Setting up EXAFS Experiments at an Insertion Device beamline

Carlo Segre, Joshua Wright, and Yujia Ding

We will demonstrate how to set up and acquire EXAFS data at an insertion device beamline, 10-ID-B. This involves setting up the undulator, calibrating the energy and setting up for both transmission and fluorescence EXAFS.

X4: Beamline Operation and X-ray Absorption Spectroscopy, 20-BM-B

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

Chengjun Sun, Mikhail Solovyev, and Shelly Kelly

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/.

X5: Angle-Resolved Photoemission Spectroscopy, 29-ID

Angle-Resolved Photoemission Spectroscopy: Probing Electronic Structure Beyond Oxidation States

Martins Barbosa and Jessica McChesney

Angle-resolved photoemission spectroscopy (ARPES) is an essential technique for investigating the electronic structure of crystalline materials. It is widely used to study the collective behavior of electrons in various materials, including those exhibiting superconductivity, topological quasiparticles, and density waves. By measuring the kinetic energy and emission angle of photoemitted electrons, ARPES allows us to map the energy dispersion and Fermi surface of electrons within a solid using conservation laws. During the visit, students will receive a comprehensive introduction to ARPES and core-level x-ray photoemission spectroscopy (XPS) experiments. The course will include a detailed, step-by-step guide to conducting these experiments. Participants will work with real, previously acquired data and will learn advanced data analysis techniques. This hands-on approach will demonstrate how photoemission spectroscopy extends beyond probing the oxidation state of constituent elements, offering deeper insights into the electronic properties of materials.

Group 3 (X6-X8): Inelastic X-ray Scattering

X6: Nuclear Resonant Scattering, 3-ID

To study atomic dynamics and electronic properties

Michel Hu and Esen Alp

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 research. 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, NRIXS can provide unique and clarifying insights into many fields of research.

SMS: Synchrotron Mossbauer spectroscopies (SMS) in both time and energy domain 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.

X7: High-Resolution Inelastic X-ray Scattering, 30-ID

High-Resolution Inelastic X-ray Scattering Measurements at Sector 30: Phonons in Single Crystals

Ahmet Alatas and Ayman Said

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

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

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

In this tutorial, 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.

X8: Resonant Inelastic X-ray Scattering, 27-ID-B

Measuring Electronic Excitations with Resonant Inelastic X-ray Scattering

Mary Upton, Jung Ho Kim, and Diego Casa

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 (X9-X13): Resonant/Magnetic Scattering/Spectroscopy

X9: X-ray Magnetic Scattering, 4-ID

Diffraction with circularly polarized x rays to probe chiral spin structure

Jörg 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.

X10: X-ray Resonant Magnetic Scattering 6-ID-B

X-ray resonant magnetic scattering (XRMS)

Philip Ryan and Jong Woo Kim

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 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.

X11: Resonant Soft X-ray Scattering, 29-ID

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

Hao Zheng

Resonant Soft X-ray Scattering (RSXS), a combination of X-ray scattering and X-ray absorption spectroscopy, is uniquely positioned to reveal electronic orderings in quantum materials. It offers a unique element 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 tutorial, we take $La_{0.67}Sr_{0.33}MnO_3$ thin film as an example and demonstrate how RSXS measures its charge order in an elemental-selective manner. Participants will get hands-on experience by tuning the X-rays to the orbital state of the Mn atom to perform a visualization of the orbital ordering formation.

X12: X-Ray Magnetic Spectroscopy, 4-ID

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-ferromagnetic) 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.

X13: Soft X-ray Spectroscopy, 29-ID

Spectroscopy with Polarized Soft X-rays

John Freeland

Spectroscopy is a powerful X-ray tool to gain element resolved information from materials of interest in areas ranging from chemistry to physics. Adding the ability to control the polarization of the X-rays allows one to gain deeper insight into electronic and magnetic properties of materials. The soft X-ray regime is ideal for many materials of interest since the transition metals with partially filled d bands have very strong absorption resonances. In this tutorial, we will cover the basics of spectroscopy including how to harness the polarization of the light to gain insight into charge distributions around atoms (i.e. orbitals) and to also probe magnetic order. We will also cover the basics of spectroscopy measurements and instrumentation involved on the beamline at 29-ID.

Group 5 (X14-X17): Coherence Based Techniques

X14: X-ray Ptychography, 2-ID-D

X-ray Ptychography Imaging

Junjing Deng and Steven Henke

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 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. By leveraging a pre-recorded ptychographic dataset obtained from an integrated circuit, we will guide you through the phase-retrieval computation process using Ptychodus (https://github.com/AdvancedPhotonSource/ptychodus). Ptychodus provides a GUI interface to a collection of different reconstruction algorithms developed at the APS. Through this hands-on exercise, you will be able to obtain a real-space image of the circuit structures, showcasing the nanoscale resolution achievable through this nondestructive imaging technique.

X15: X-ray Photon Correlation Spectroscopy, 8-ID-E & 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.

X16: Coherent Bragg Rod Analysis (COBRA), 28-ID

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 tutorial session, due to ongoing APS-U project, we may combine APS on site beamline visit and/or virtual demonstration platform using computer resources provided. We will firstly present 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 mock on the virtual platform the experimental procedure of mounting, aligning, and measuring a high-quality perovskite oxide epitaxial thin film (e.g. 5-10 unit cell thick LaNiO3 on SrTiO3 substrate) grown by molecule beam epitaxy at the APS. Then, we will flow through the detailed COBRA data processing/reduction steps. Furthermore, we will demonstrate how to 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-F

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 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/operando 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 (X18-X22): Small Angle X-ray Scattering

X18: 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.

X19: Small and Ultra-Small Angle Scattering from Complex Materials, 12-ID-E

USAXS/SAXS/WAXS analysis of complex hierarchical materials - from Angstrom to microns

Jan Ilavsky

Complex hierarchical structures are common in our everyday life since most of the important natural (wood, soil, …) as well as man-made engineering (alloys, polymers, gels, ...) materials do have them. Such complex structures require appropriately complex analysis approaches. Ultra-Small and Small angle scattering techniques powerful tools which, when combined with other techniques, can provide unique inside into complex structures. APS USAXS/SAXS/WAXS instrument is commonly used to study wide range of materials - from chemistry, polymers, physics, materials engineering, and other technologically important fields to more nature-oriented fields, such as geology. Applications in food sciences and other non traditional fields have been growing recently also. The tutorial will walk students through sample preparation, data collection and reduction to data analysis of interesting case examples from recent USAXS history.

X20: Small Angle X-ray Scattering of biological and nanoscale systems, 12-ID-B

Small Angle X-ray Scattering of biological and nanoscale systems

Xiaobing Zuo, Ivan Kuzmenko, 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 tutorial, the beamline 12-ID-B and its capabilities will be introduced. If the beamline is back to operation, a few measurements will be demonstrated on a variety of different samples, i.e., proteins, polymers or nano-particles, and the data will be analyzed and interpreted. If the beamline is not ready then, example researche carried out at this beamline will be discussed.

X21: Studying hierarchical materials with SAXS, MAXS, and WAXS, 5-ID-D

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

Denis Keane, Steven Weigand, and Mike Guise Jr.

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 nano-materials 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.

X22: Nanomaterial Characterization using Anomalous SAXS, 15-ID

Elemental distribution within/around nanomaterials using Anomalous Small Angle X-ray Scattering

Mrinal Kanti Bera 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.

Group 7 (X23-X25): Diffraction-I

X23: Energy Dispersive X-ray Diffraction, 6-BM

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.

X24: Synchrotron Powder Diffraction, 11-ID-B & 11-BM

High Resolution and In-Situ Powder Diffraction Data Processing & Analysis

Wenqian Xu, Andrey Yakovenko, 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 be able to watch a live experiment at 11-ID-B/C beamline, analyze data, and discuss with the beamline staff. Students are expected to bring their own laptops for the data analysis part of the tutorial.

X25: Pair Distribution Function Analyses, 11-ID-B

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

Olaf Borkiewicz, Kamila Magdalena Wiaderek, and Justin Michael Hoffman

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 employing 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 (X26-X28): Diffraction-II

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 tutorial will include the following topics:

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

X27: Single Crystal Diffuse Scattering, 6-ID-D

Single Crystal Diffuse Scattering and 3D- ΔPDF analysis

Matthew Krogstad

We will outline the procedure for measuring diffuse scattering from a single crystal. Without x-rays, previously collected sample data will be investigated and reduced from a stack of detector images to oriented reciprocal space. We will also discuss the use of 3D-ΔPDF to interpret single cystal diffuse scattering.

X28: Time-Resolved X-ray Diffraction, 7-ID

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

Don Walko and Burak Guzelturk

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 crystalline 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.

Group 9 (X29-X32): Instrumentation and Complex Environments

X29: High Pressure Charge Transport, 16-BM-B

Charge transport measurements in a Paris-Edinburgh large volume press at high pressures and temperatures

Tyler Eastmond and Innocent Chinwe Ezenwa

HPCAT (sector 16) hosts a robust Paris-Edinburgh (PE) press program that uses a suite of characterization tools to probe the influence of high pressures (up to 70 kbars) and temperatures (up to 2500 K) on material structure and properties. These techniques include energy dispersive X-ray diffraction, which allows for crystal structure determination or liquid/amorphous structure analysis using pair distribution functions, as well as measurements of macroscopic properties such as sound speed, fluid viscosity, density, liquid (im)miscibility, and electrical resistance or impedance. This tutorial will provide an introduction to the techniques and applications of high-pressure and temperature (HP-HT) synchrotron research, followed by hands on experience in preparing and conducting electrical transport measurements in the PE press.

Although the benefits of large sample volume in the PE cell cannot be over emphasized, its cell preparation is detailed and complex, and differs when compared with other high-pressure techniques such as the diamond anvil cell. Students will be given the opportunity to assembly PE cells prior to running high-pressure experiments. Following cell preparation, students will perform a guided experiment in which charge transport measurements are conducted in the PE press. The measurement of charge transport in a material can characterize solid-liquid or solid-solid phase transformation at HP-HT conditions and will help students see first-hand how pressure influences material properties. During the experiments, students will be introduced to electrical resistance and electrical impedance spectroscopy measurements, the former being useful for electronic charge transport in metals and the latter for more complex charge transfer in ceramics. An emphasis will be made on choosing the appropriate characterization tool for a given experiment. After acquiring data, students will be shown how to process and analyze data using in-house and commercial software.

X30 High pressure tools for X-ray diffraction, 13-ID

A day in the life of an XRD experiment at extreme conditions

Stella Chariton and Dongzhou Zhang

In this tutorial we will demonstrate the many features of the "Dioptas" software (http://www.clemensprescher.com/programs/dioptas) using real-world data collected at GSECARS. We will explain the many different characteristics of high-pressure X-ray diffraction (HP XRD) patterns - how different are they from ambient conditions data, how different are the data collection procedures, how do patterns change with pressure, temperature, strain, composition etc? We will also learn how a calibration file is created. We will offer a mini (virtual/video) tour in the user experience: planning, conducting and analyzing data collected using the DAC, PE cell and LVP. We will learn how to identify and address common problems in the HP XRD data. Finally, we will briefly visit other useful tools for XRD at high pressure, such as how to determine pressure and how to control temperature (laser and resistive-heating or cryogenic) in the DAC and other high-pressure devices. The tutorial will conclude with mini fun self-evaluation quizzes.

X31: 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.

X32: X-ray 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.