**2018 X-ray Experiment descriptions:**

## X1: High Energy X-ray Scattering for Strain Measurement, 1-ID

### “Texture and strain measurement in polycrystalline materials using high energy x-rays”

#### Jun-Sang Park and Jonathan Almer

Polycrystalline materials encompass large groups of materials such as metals, ceramics, and minerals are employed 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 polycrystalline materials. In this experiment, we will use high energy x-rays to measure the lattice strains and texture in a polycrystalline sample under in-situ thermo-mechanical loading.

## X2: X-ray Ptychography Imaging, 2-ID-D/E

“*High-resolution X-ray Ptychographic Imaging of Integrated Circuits*”

Junjing Deng, Jeffrey Klug, Olga Antipova

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

## X3: Nuclear Resonance Scattering, 3-ID

*“An introduction to NRIXS”*

#### Michael Hu and Ercan Alp

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. Vastly disparate energy scales involved in nuclear excitations (many keV) and atomic lattice excitations (tens of meV) implicate the decoupling of these two processes. NRIXS can be described as nuclear resonant excitation plus phonon annihilation or creation. As a result, on the scale of the energies of phonons, the energy of nuclear resonant absorption is modified only through atomic motions in a sample. The unique aspect of using resonant isotopes to measure phonon energies is mainly the selectivity. This means that vibrations can be probed locally in systems that have resonant isotopes in specific places, e.g., bio-molecules like myoglobin, 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.

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

*“Magnetic proximity effect studied using XMCD”*

#### Yong Choi

X-ray magnetic circular dichroism (XMCD) measures the difference in absorption of circularly polarized x-rays by a magnetic material. This technique can provide element and orbital specific magnetic information. In this experiment, element-specificity of XMCD will be used to investigate the magnetic proximity effect in a Pt/Fe film. Whereas Pt metal is nominally paramagnetic, the Pt atoms in contact with the Fe layer can have induced magnetic moment. The induced magnetism in the Pt layer will be probed using x-ray magnetic circular dichroism spectroscopy at the Pt L2,3 edges.

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

Denis Keane and Qing Ma

X-ray absorption spectroscopy techniques have been widely used in the research activities of multiple disciplines, for example chemistry, chemical engineering and environmental science. These techniques are also very versatile and can be adapted to suit a variety of sample conditions, including bulk, thin film, powder, and liquid. Measurements can be carried out through various channels or modes such as absorption, fluorescence, electron yields, etc., and in various geometries from normal incidence geometry (for transmission or grazing exit fluorescence measurements) to grazing incidence geometry. We will demonstrate elemental selectivity and chemical speciation in several types of samples including bulk mixtures and thin films.

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

### “Energy Dispersive X-ray Diffraction”

#### John Okasinski and Andrew Chuang

The energy-dispersive x-ray diffraction (EDXRD) configuration enables one to selectively measure scattering from a discrete 3D volume within a larger bulk sample and surrounding environment. This is achieved through the use of a polychromatic incident beam and measuring at a fixed scattering angle with an energy-dispersive detector. The gauge volume attained creates the opportunity to map both crystalline phases and strain in complex samples. Three examples that make use of this technique include: mapping the progress and heterogeneity of the electrochemistry within a battery; mapping the strain in a structural component, such as near a weld joint; a sample confined inside of a complex environment, such as a furnace or large volume, high pressure cell.

## X7: Magnetic X-ray Scattering, 6-ID-B/C

### “Resonant and/or non-resonant x-ray scattering from a magnetic material in external magnetic field

#### Zahir Islam and Jong-Woo Kim

Resonant and non-resonant diffraction is a cornerstone for discovering novel quantum states of matter. Exploring extended phase diagram in extreme magnetic fields is crucial to understanding the nature of the quantum phenomena. This experiment will emphasize time-resolved diffraction to collect a magnetic field dependence of magnetic or charge Bragg peaks within a short pulse of high magnetic field. Order parameter, such as a Bragg-peak splitting signaling a structural transition, or a new superlattice peak due to a spin/charge/orbital ordering, will be measured as a function of temperature and fields.

## X8: Radiography, 7-BM

### “Time resolved radiography of liquid fuel sprays”

#### Alan Kastergren and Katie Matusik

Multiphase flows are critical to numerous technologies we depend upon. For example, liquid sprays have a large impact on the performance of internal combustion engines and liquid rockets.  These flows are typically opaque to visible light, which makes measurements of these flows challenging.  Time-resolved x-ray radiography has been developed over the past 15 years at Argonne to quantitatively probe these flows, and now represents one of the best ways to study dense multiphase flows.  Students will use radiography to probe the structure of a commonly used spray flowfield as a function of space and time.

## X9: Time-Resolved X-ray Diffraction, 7-ID

### “Time-resolved x-ray diffraction”

#### Don Walko, Anthony DiChiara, Haidan Wen

Heat transport is becoming an increasingly critical issue in the design of nanoscale electronics; as device components shrink, the interfaces between components play a more prominent role. In this experiment, laser-pump, x-ray diffraction-probe measurements will be used to determine the thermal conductance of a film/substrate interface. Pulses from an ultrafast Ti:sapphire laser will be used to heat a thin metal film grown on a transparent substrate. X-ray Bragg diffraction then probes the response of crystalline matter to the laser, with a time resolution limited by the length of APS x-ray bunches (~100 ps). The laser is synchronized to the APS accelerator, with electronics that can vary the delay time between the arrival of the laser and the x rays at the sample. The angular shift of the film’s Bragg peak is thus mapped out for several laser/x-ray time delays. The position of the Bragg peak acts as a thermometer for the film, from which the conductance of the film/substrate interface will be derived.

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

### “GISAXS from organic photovoltaic thin films”

#### Zhang Jiang, Joseph Strzalka, Wei Chen

Since their introduction in the mid-90’s, organic photovoltaics (OPV) based on the polymer:fullerene bulk heterojunction (BHJ) have become a fast-growing area of research, resulting in steady improvement in solar cell efficiencies from approximately 1%, approaching the 10% efficiency expected to result in their widespread commercialization. This inexpensive and scalable technology promises to play an important role in meeting the world’s energy needs. Understanding and further optimizing OPV technology requires, in part, insights into how the morphology of these thin film devices affects their function, and how different processing conditions influence the morphology and hence the solar cell efficiency. Grazing incidence x-ray scattering (GIXS), which can non-destructively probe statistically meaningful regions and reveal hierarchical structure on lengthscales varying from Ångstroms to hundreds of nanometers on surfaces or buried interfaces, has become an essential tool for this effort. Participants will measure and analyze GIXS from thin film samples of typical OPV material processed under different conditions, thereby gaining direct experience of the kind of information that can be gained from these measurements, as well as familiarity with the hardware and software in use at 8-ID-E.

## X11: X-ray photon correlation spectroscopy, 8-ID-I

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

#### Suresh Narayanan, Eric Dufresne and Alec Sandy

X-ray photon correlation spectroscopy (XPCS) is a well-established technique to study the equilibrium dynamics in soft and hard matter systems. XPCS has been successfully applied to study dynamics in colloidal suspensions, nanoparticle dispersion in polymers, polymer thin films, etc. XPCS uses the partially coherent nature of the synchrotron beam to probe speckles and its fluctuations in time. By using a 2-D detector such as a CCD, the dynamics over a range of length scales in the range of 100 nm - 10 nm can be probed simultaneously.

In this experiment, a colloidal suspension of silica spheres in the size range of 100 nm dispersed in a viscous solvent like glycerol will be studied. By varying the particle concentration, single particle Brownian diffusion and the effect of particle interactions will be studied.

## X12: X-ray Absorption Near Edge Spectroscopy - 9-BM

*“Auto forensics: XANES analysis of catalytic converters”*

#### Tianpin Wu, George Sterbinsky, Steve Heald

All automobiles have catalytic converters, which are important for controlling emissions. All catalytic converters contain a catalyst. When a catalyst fails, it is rarely due to a problem with the converter. It is typically a symptom of something else. This experiment will demonstrate how spectroscopic techniques can be used to determine what materials are in a spent catalyst from a catalytic converter. This information can in turn be used to deduce what may have been wrong with the automobile engine. For a fragment of catalyst obtained from a local muffler shop, an energy-dispersive detector will be used to identify the elemental composition through the use of calibration foils. Then, the XANES of select elements will be obtained, and chemical fingerprinting will be used to identify the compounds. The students will be guided through the process of coming up with a hypothesis as to “what killed the car?”

## X13: Ultra-Small Angle X-ray Scattering, 9-ID

*“USAXS/SAXS/WAXS studies of structure of common materials”*

#### Jan Ilavsky, Matt Firth, Ivan Kuzmenko

This instrument provides a unique facility for ultra-small-angle, small-angle, and wide-angle scattering studies over an unprecedented range of length scales within a single measurement—from less than an Ångstrom to few microns. Engineering materials (e.g. metals, polymers, ceramics, *etc*) often exhibit complex, hierarchical, microstructures spanning this wide range of sizes. Students will become familiar with this unique technique and measure selected examples of materials they may use during their day-to-day life, such as toothpaste, food fats, cheese etc. Analysis of the USAXS data using general purpose Irena software will be showcased as part of the experiment.

## X14: Synchrotron Powder Diffraction, 11/17-BM

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

#### Wenqian Xu & Andrey Yakovenko

X-ray powder diffraction is a versatile technique that reveals detailed information about the chemical composition and crystallographic structure of materials, and affords great flexibility for in-situ studies of samples under non-ambient conditions. In this experiment, students will gain hands-on experience with all aspects of modern synchrotron powder diffraction experimentation, from sample preparation to strategies for data collection, processing, and analysis. Students will become familiar with the world-class suite of dedicated powder diffraction instruments offered at the APS, including both high-resolution and two-dimensional area detector measurements, as well as a wide range of in-situ sample environments. They will learn how to access and use these tools to address scientific challenges related to their own research. The second day of this experiment will include an interactive tutorial on data processing and Rietveld analysis using the software package GSAS-II, including the determination of crystallographic structural details from powder diffraction data measured on the first day of the experiment. While this experiment is intended for those new to synchrotron-based powder diffraction, in depth questions will also be addressed if time permits.

## X15: Pair Distribution Function, 11-ID-B

### "Pair distribution function measurements with high-energy X-rays."

#### Olaf Borkiewicz, Kamila Wiaderek, Kevin Beyer, Karena Chapman

Pair distribution function (PDF) analysis measures local atom structure as the distribution of atom-atom distances from Ångstroms up to several nanometers. A strength of the technique is that it does not assume translational symmetry of the structure, as required for traditional crystallographic approaches, and thus PDF can be applied to study disordered, crystalline, amorphous, nanoscale, homogeneous and heterogeneous materials alike. Experimentally, the PDF is derived from a specialized powder diffraction measurement in transmission geometry: High-energy X-rays are used to measure the structure function to a high value of momentum transfer, *Q*. Further normalization of the structure factor and subsequent direct Fourier transformation will yields the Pair-Distribution-Function (PDF). This experiment will cover strategies for data collection and processing, and simple modeling approaches. We will explore how the experimental variables (beam energy, beam/sample size, detector distance, capillary composition) impact the quality and resolution of the resulting data.

## X16: Small Angle X-ray Scattering, 12-ID-B

### "Small Angle Scattering (SAXS) of biological, organic and inorganic systems."

#### Xiaobing Zuo, Byeongdu Lee

Small angle X-ray scattering (SAXS) and Grazing incidence SAXS (GISAXS) are the scattering techniques to determine nanoscale structures and provided at 12-ID-B stations of APS. Examples of research experiments performed at the beamline include in-situ nanoparticle growth, in-situ monitoring nanoparticle catalyst under reaction, block copolymer morphology, aggregation of charged polymers, self or directed assembly of nanoparticles, structure of gel, conformation of protein and RNA, nano and bio hybrid materials, and so on. In this experiment, the beamline and its capabilities will be introduced, and measurements will be carried out on a variety of different samples, i.e., proteins or polymers or nano-particles or nano-particle assemblies. The data will be analyzed and interpreted.

## X17: High-Pressure Powder Diffraction, 16-BM-D

### "Pressure-induced structural phase transition in ZnO"

#### Changyong Park and Dmitry Popov

Pressure is a powerful tool to investigate materials’ physical properties like hardness, elasticity, and strength. It can be used to adjust the electrical conductance and magnetism, sometimes leading to a discovery of new superconducting materials with help of combined cryogenic cooling. It also can cause reversible or irreversible phase transitions when the range of pressure is extended beyond the stability field, which many times lead to a discovery of new materials. In the solid state, the range of pressure to cause these physical changes typically goes far to GPa level (Giga Pascal, 1 Pa = 1N/m2), for which we need to use a special apparatus, Diamond Anvil Cell (or DAC). In this experiment, students will perform high-pressure powder x-ray diffraction with a pre-loaded DAC sample and learn how it helps to study the materials physical property. The pressure-induced volume contraction and eventually the phase transition in ZnO will be demonstrated and an entry level lattice parameter refinement will be exercised to quantitatively describe the observation.

## X18: Fundamentals of beamline operation, 20-BM

*“Fundamentals of beamline operation and Cu XAFS”*

#### Steve Heald and Chugjun Sun

There are several parameters that need to be optimized for successful experiments. In the case of x-ray spectroscopy, the most important include the energy resolution, harmonic content, and sample quality (thickness and uniformity). We will work through setting up a beamline and run several “hands on” exercises looking at these parameters and how they affect the final data. Once the beamline is characterized and properly set up, it will be used to measure two types of Cu samples. An oriented high Tc superconductor sample will be used to illustrate the utility of using the x-ray polarization to isolate signals from the in-plane and out-of-plane bonds. Linear combination fitting of both the EXAFS and XANES will be demonstrated by fitting the data for an arbitrarily oriented sample. We will also measure the Cu foil EXAFS and fit it with the FEFF theory to demonstrate theoretical fitting. Analysis will be done using the Demeter software that can be downloaded from <http://bruceravel.github.io/demeter/>. Prior experience in synchrotron experimentation is desirable.

## X19: High-Resolution Inelastic X-ray Scattering Measurements, 30-ID

*“Phonons in Single Crystals”*

#### Ahmet Alatas and Ayman Said

Typically, scattering experiments with x-rays or neutrons are done without energy analysis after the scattering event. Therefore, an integration of all scattered energies is done experimentally in the detector. The information extracted from these experiments is related to information on the structure in the studied system, or, more precisely, to correlation functions of the structure. If the energy of the scattered intensity is analyzed, it is called an inelastic scattering experiment and- in addition to the structural information- dynamical properties of the system can be studied, i.e., information on correlations in time is obtained. Moreover, inelastic x-ray scattering (IXS) provides access to very rich excitation spectra; phonons, magnons, electronic excitations, plasmon and Compton scattering depending on the transferred energy (meV to several hundreds of eV).

The Advanced Photon Source has two beamlines (Sector 3 and 30), with very high-energy resolution (1.5-2 meV), specialized for studying collective excitations (phonons) where their energies lie in the order of milli-electronvolts (meV). IXS is a very important technique in applications ranging from condensed matter physics to life science and mineral physics to geophysics.

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

## X20: High-speed X-ray Imaging, 32-ID-B

### “X-ray vision of metal 3D printing process”

#### Tao Sun, Niranjan Parab, and Cang Zhao

Laser powder bed fusion (LPBF), an extensively used metal 3D printing technique, has been witnessing tremendous growth over the past three decades, particularly in the fields of medical, aerospace, automobile, and defense. Although the LPBF process is conceptually simple, there are many highly dynamic and transient physical phenomena involved because of the extremely high heating and cooling rates. Oftentimes, a product made by LPBF contains rough surface, significant porosity and residual stress, and/or unfavorable phase and grain structures. These defects will largely degrade the performance of the manufactured parts. Owing to the high flux and high penetration power of hard x-rays afforded by the synchrotron facility, imaging with x-ray photons provides a unique tool for directly probing the metal 3D printing process with extremely high spatial and temporal resolutions. In the experiments, the students will use high-speed x-ray imaging technique to observe the laser-metal interaction in LPBF process. In particular, the formation processes of structural defects caused by keyholing and lack of fusion will be recorded with a rate up to 50,000 frames per second. The students will also learn basic image processing and analysis approaches to extract quantitative structural information from the x-ray images.

## X21: Grazing Incidence Interface Diffraction, 33-BM

### “Exploring 3-D Reciprocal Space: a Powerful Tool to Answer Basic & Applied Materials Science Questions”

#### Evguenia Karapetrova

The efficient exploration of large volumes of reciprocal space, made possible by the advent of high frame-rate, 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.

## X22: Coherent X-ray Diffraction Imaging, 34-ID-C

### “Coherent X-ray Diffraction Imaging of Nanocrystals”

#### Ross Harder

The high brightness, and resulting high degree of coherence, of modern synchrotron x-ray sources has enabled the development of advanced x-ray imaging techniques. Coherent x-ray diffraction (CXD) imaging exploits the coherence of the synchrotron source to replace the lens of a traditional microscope with computational algorithms to form images. This imaging method allows one to surpass the resolution limits of modern x-ray optics. It also provides for an unencumbered space around the sample for complex in-situ environments. In addition, when the coherent scattering in the vicinity of a Bragg peak of a crystal is measured, a high sensitivity to distortions of the crystal lattice due to strain can be exploited. In this experiment we will measure the coherent scattering in the vicinity of a Bragg peak of a small (typically 300nm) gold crystal. We will then computationally invert the measured 3D diffraction pattern to a 3D image of the crystal.

## X23: X-ray Micro-Laue Diffraction, 34-ID-E

### “Measuring crystal microstructures with x-ray micro-beam Laue diffraction”

#### Ruqing Xu, Wenjun Liu, Jon Tischler

The x-ray micro-beam Laue diffraction at beamline 34-ID provides a unique diffraction probe of material microscructures with highly-focused, polychromatic x-ray beam and 3D spatial resolution. A pair of custom-profiled K-B mirrors provide sub-micron x-ray focal size, the scanning-wire differential aperture provides depth-resolution along x-ray’s penetration, and high-speed area detectors allows 3D mapping over relatively large sample volumes. The technique can reveal detailed local structural information of crystalline materials, such as crystallographic orientation, orientation gradients, grain morphology, strain tensor, and lattice structure, with spatial resolution of less than 300 nm and angular resolution of 0.01°. It is applicable to single crystal, polycrystalline, composite, deformed, and functionally-graded materials. Applications include studies of fundamental deformation processes, basic grain-growth behavior, electromigration, solid-solution precipitation, structural phase transformation, and high-pressure mineral physics, etc.

**2018 Neutron Experiment descriptions:**

**N1: Triple-Axis Spectrometers, HFIR HB-1A & HB-3**

*Spin wave and phonon dispersion in Fe-Ga solid solutions*

Fe-Ga alloys with appropriate composition and heat treatment, exhibit giant magnetostriction in a polycrystalline and ductile form. The tetragonal magnetostriction coefficient, λ100, of Fe-Ga can be up to 15 times that of pure Fe. This makes these materials of tremendous scientific and technological interest for use in devices such as actuators, transducers and sensors. Elastic constant measurements show that the shear elastic constant 1/2(C11-C12) decreases with increasing gallium concentration and extrapolates to zero at approximately 26 at.% Ga. The slope of the phonon dispersion curve at low-q of the T2[110] branch is a measure of that elastic constant and hence the interest in measuring phonons in these materials. With the large magnetoelastic interactions in such a material, it is also of interest to measure the spin wave dispersion. The triple-axis spectrometers HB-1A and HB-3 will be used to measure both phonon and spin waves of two compositions of Fe-Ga alloys.

**N2: Powder Diffractometer, HFIR HB-2A**

*Magnetic structure of NiO*

Neutron diffraction measurements will be performed to investigate the onset of long-range magnetic order in NiO. Data will be collected at various temperatures, ranging from 600K to 288K, using the Neutron Powder Diffractometer at the HFIR. Rietveld analysis of the crystal and low-temperature magnetic structure will be carried out using FullProf Suite software. The results obtained will be discussed and compared with those reported in earlier studies.

**N3: WAND powder/single-crystal diffractometer, HFIR HB-2C WAND**

*Crystallographic superstructures Ho2PdSi3 and Pr2PdSi3*

The intermetallic compound series R2PdSi3 (R = rare earth metal) exhibits some interesting magnetic properties as giant magneto-resistance effect, strong anisotropy in the electronic properties and a generic field induced phase. The magnetic structures are quite complex with large magnetic unit cells due to the delicate interplay between competing crystal electric field effect and magnetic exchange interaction and the addition of geometric frustration. The hexagonal crystallographic structure is formed from the sequence of triangular rare earth layers and Pd/Si layers stacked along the c-axis. The c/a ration is close to one and gives raise to the expectation of a strong anisotropy between hexagonal axis and plane expressed in large values of the second order of Stevens’s operator B20. However, the Pd/Si layers obey site occupation rules of its ions and the stacking of the layers yields a crystallographic superstructure. In the R2PdSi3 with heavy rare earth ions (R = Gd, Tb, Dy, Ho, Er, Tm) the connection between the crystallographic superstructure and the magnetic structure has been studied extensively, but it is unknown if a superstructure for compounds with light rare earths (for instance Pr) exists, due to the lanthanide contraction.

We hereby propose to investigate the structure of Pr2PdSi3 using WAND². We will orient the crystal in HK0 scattering plane and collect a full reciprocal map. With the out-of plane coverage we should be able to cover up to HK0.5 reciprocal space and be able to search for additional reflections.

**N4: Four-Circle Diffractometer, HFIR HB-3A**

*Structure and lithium-ion motion in the triphylite LiFePO4 studied by single crystal diffraction*

Triphylite, Li(Fe,Mn)PO4, is a candidate cathode material for lithium ion batteries due to its virtues of low cost, better safety characteristics and environmental friendliness. But it also faces a significant challenge to achieve both high reversible lithium storage capacity and rapid ion and electron transport capabilities for large-scale EV applications. Studies on the lithium-ion motion properties will help to understand the lithium conduction mechanisms in a lithium ion battery. Using single crystal neutron diffraction, we will resolve the structure of a natural triphylite single crystal at several selected temperatures. Besides the nuclear structure, we are also able to give the magnetic structure at the temperatures lower than its transition temperature. Fullprof and Shelx will be used to refine both nuclear and magnetic structures.

**N5: Neutron Imaging Station, HFIR CG-1D**

*Hemi-wicking of water on rock fracture surfaces (3 experiments)*

Spreading of water over rough surfaces is a capillary process that is well understood in engineered materials. However, it is not well understood in geomaterials. We plan to study in-situ kinetic hemi-wicking on rock fracture surfaces (of a split cylindrical shape rock). These measurements will require the acquisition of neutron radiographs at a rate of 30 frames per second. The core samples have low porosity and their dimensions are 2.5 cm dia and 5 cm long. They are fractured by compression loading prior to the experiments at the neutron imaging beamline. The fracture is oriented perpendicular to the detector and water is brought up to touch the bottom surface of the core sample.

*Sphere packing (1 experiment)*

Metallic sphere packing are substitutes for lattice arrangements in crystalline materials. In this experiment, we will use a piezoelectric motor to change the packing arrangement of the spheres (from disordered to ordered close-packing, the highest density achievable) and use neutron computed tomography to map the spheres packing arrangement in 3D. Different-diameter spheres are more representative of interstitial packing in crystalline materials. These measurements will also be performed depending on time availability.

**N6: IMAGINE, HFIR CG-4D**

*Laue white beam diffraction in a diamond pressure cell*

The IMAGINE CG4-D beamline is designed for rapid survey of the reciprocal space. The beamline optics select a quasi-Laue bandpass, stimulating multiple reflections in a single exposure. The instrument is equipped with a large 2D detector that simultaneously record stimulated Bragg spots.  Neutron single crystal diffraction will be performed using a new neutron diamond anvil cell. The students will load small crystals into the diamond cell together with an appropriate pressure transmitting medium. Initially, single crystals will be measured under ambient pressure. Pressure will then be applied, and the crystals will be re-measured under pressure. Diffraction patterns under ambient and high pressure will be indexed and analyzed.​

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

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

Contrast-matching SANS has been widely used to characterize structure of soft and biological matter as well as pore accessibility in porous materials. The particular advantage of this technique is attributed to the large difference in coherent scattering lengths of hydrogen and deuterium. By changing composition of protonated and deuterated solvent (i.e. varying the volume fraction of H2O in the mixture of H2O and D2O), 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 H2O 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.

**N8: Small Angle Neutron Scattering, HFIR CG-3 Bio-SANS**

**SNS, EQ-SANS, BL-6**

*Micellar morphologies in self-associated triblock copolymer solutions: effects of concentration and contrast matching in porasils*

The PEO-PPO-PEO triblock copolymers have important applications in industry and medicine. Because of the different solubilities of PEO and PPO in water, these copolymers exhibit a rich phase behavior that is sensitive to polymer concentration, solvent ionic strength, temperature, and pressure. These phase changes occur by the self-assembly of the polymer chains into structures with characteristic length scales of the order of few nanometers. Thus, small-angle neutron scattering (SANS) is a technique uniquely well-suited to studying this phase behavior. In these experiments we will study the effects of concentration and ionic strength on block copolymer self-assembly using solutions of 1,2, and 5 wt% Pluronics F108 triblock copolymer in D2O 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.

**N9: NOMAD Nanoscale-Ordered Materials Diffractometer, SNS BL-1B**

*Introduction to Pair Distribution Function analysis*

The Nanoscale Ordered Materials Diffractometer (NOMAD) is designed for the determination of pair distribution functions (PDF). The PDF is a measure of the probability to find an atom B at a distance r away from arbitrarily chosen central atom A relative to a random arrangement. As such it is a measure of the atomic arrangement of the sample independent of periodicity and therefore the PDF formalism can be applied equally to liquids, glasses, nanomaterials and long range ordered crystalline materials.  We will determine the PDF of glassy SiO2 and fit a Continuous Random Network model to it. We will perform an isotope substitution experiment for BaTi2O5. We will introduce real-space fitting using the ‘small-box’ refinement program PDFgui, modeling the PDF of diamond, crystalline SnO2, and SnO2 nanoparticles.  We will also introduce the levitation sample environments at NOMAD for container-less and high temperature neutron scattering, performing a laboratory experiment with a melt.

**N10: BASIS Backscattering, SNS BL-2**

*Diffusion dynamics of protons in a novel ionic liquid designed for proton-exchange membranes*

Protic ionic liquids show great potential for mobile fuel cell applications. They possess appealing features such as almost negligible vapor pressure, the characteristic electrical conductivity of an ionic conductor, and a sizable temperature gap between the melting and decomposition points. The diffusion dynamics of protons in these complex liquids are closely tied to their performance as electrolytes. Quasielastic neutron scattering (QENS) is a technique of choice for studying the details of diffusion dynamics of hydrogen because of (1) the large incoherent scattering cross-section of hydrogen compared to other elements and (2) capability of probing spatial characteristics of diffusion processes through dependence of the scattering signal on the momentum transfer, Q. The latter is a clear advantage of QENS compared to, for instance, NMR. In our QENS experiment to be performed on the SNS backscattering spectrometer, BASIS, we will utilize the Q-dependence of the scattering signal to identify and analyze several dynamic processes involving diffusion motions of hydrogen atoms in a synthesized ionic liquid [H2NC(dma)2][BETI].

**N11: SNAP Spallation Neutrons at Pressure, SNS BL-3**

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

The SNAP instrument is ORNL’s dedicated high pressure diffractometer. It is a highly versatile instrument optimized for the small samples sizes present in neutron pressure cells. SNAP employs standard gas pressure and clamp cells and also the entire range of Paris-Edinburgh cells, cells that pushed standard pressures for neutron scattering to 20 GPa. Furthermore, SNAP has been leading the development of record breaking neutron diamond cells which have achieved the highest pressure recorded with neutron scattering as well as the large volume of any sample held at static pressures above 60 GPa.

During this experiment, students will be introduced to the various pressure cells and the opportunities and challenges available at SNAP. Furthermore, they will conduct a hands-on experiment on water ice. Water ice has one of the most diverse phase diagrams of any substance known. We all know ice I, the hexagonal form that freezes at 0 oC 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 the their counterpart liquid and sink under equilibrium conditions. At room temperature, upon compression, water 'freezes' at about 1.3 kbar into ice VI, further transforming at 2 kbar to ice VII, both with inter-penetrating hydrogen bond networks. Over all, the tetrahedral and highly directional nature of the hydrogen bond leads to a fascinatingly diverse P-T structural phase diagram. Students will load a sample of liquid water into a Paris-Edinburgh pressure cell. They will increase the pressure on the sample first to 1.5 GPa and then to 3 GPa, collecting data at each point. Once analyzed, the data will reveal that the sample has undergone two phase transitions: first from liquid water at ambient pressure to ice VI at 1.5 GPa and second from ice VI to ice VII at 3 GPa.

**N12: Magnetism Reflectometer, SNS BL-4A**

*Revealing magnetism in thin films of normally non-magnetic materials*

Understanding the magnetic properties of complex materials near surfaces and interfaces critically important for the development of functional nanostructures and devices. To investigate such structures, where the magnetic layer is only a few unit cells thick and buried within a material, polarized neutron reflectometry is clearly the method-of-choice. During the last two decades Polarized Neutron Reflectometry (PNR) has become a powerful and popular technique in the study of properties of thin films and multilayers. Recent studies show a strong influence of interfaces on the magnetic properties of thin films, leading to behaviors that are radically different from those of bulk materials. Students will apply polarized neutron reflectometry to study interfacial magnetism in a LaMnO3-thin film epitaxially grown on a SrTiO3 substrate. They will mount the sample in the Displex and will learn how to align the sample in the neutron beam of only 50 microns thick. First PNR measurement will be performed at room T. Then the sample will be cooled to 5K and the measurement will be repeated. The students will process the data using the data reduction programs and will compare the results of the two experiments. With this practice, students will learn polarized neutron reflectometry set-up, in-situ data reduction from 2-D intensity maps, and understand the evolution of properties in thin films with temperature.

**N13: VULCAN Engineering Materials Diffractometer, SNS BL-7**

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

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

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

*Introduction to diffuse scattering analysis based on single crystal measurement*

CORELLI is a statistical chopper spectrometer with energy discrimination located at beam-line 9 at the SNS. CORELLI is designed and optimized to probe short-range correlation of crystalline materials through single-crystal diffraction and elastic diffuse scattering. CORELLI combines the high efficiency of white-beam Laue diffraction with energy discrimination by modulating the beam with a unique statistical chopper.  We will practice the experimental setup, data collection, data reduction on the single crystal Zr0.85Ca0.15O1.85 on Corelli. Data collection strategy will be optimized based on initial sample orientation determination. Data reduction and visualization (including the comparison of total and elastic-only spectrum) will be performed using Mantid. The normalized data will be used to perform three dimensional (3D)-PDF using the punch-fill method to reveal the short-range correlation in the system.

**N15: POWGEN Powder Diffractometer, SNS BL-11A**

*Powder Neutron Diffraction for crystal structure refinement and quantitative phase analysis*

The student groups will have the opportunity to fill a sample holder with sample powder and perform a helium gas pump-purge of the holder, readying it for neutron diffraction with our POWGEN Automatic Changer (PAC) sample changer. They will learn how to set up a run using the Data Acquisition System (DAS). Afterwards they will learn Rietveld refinement using Powgen time-of-flight (TOF) neutron diffraction data. Exercises will include

* Sample 1: A simple structure (Ni or LaB6) to introduce TOF refinement concept.
* Sample 2: Quantitative phase analysis (NIST standard 674b: a mixture of ZnO, TiO2, Cr2O3 and CeO2).
* Sample 3: For those who want to refine a more complex structure, we will look at several models to determine the true crystal structure of Ba2CuWO6, which shows a Jahn-Teller distortion.
* Sample 4: Finally, those who get through the first three examples will be able to learn how to do sequential refinement for temperature scans of ZrW2O8.

If the students would like to analyze POWGEN data on their own samples that will be possible during the neutron school session provided the students use the mail-in proposal program. They should specify in the proposal that this is related to the 2018 NXS. The proposal should be submitted no later than June 25, 2018.  Once the proposal is submitted the beamline team will be in touch to work out the logistics.

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

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

TOPAZ is a high-resolution single crystal diffractometer for the study of nuclear and magnetic structures of materials at sub-atomic resolution. It uses a large array of neutron time-of-flight detectors for data collection in wavelength-resolved Laue mode to cover a large 3D volume of reciprocal space, or Q-space (after unit conversion from neutron events recorded in detector x, y and a band of neutron wavelengths). We will practice the experimental setup, data collection, data reduction and perform a structure refinement of a single crystal dataset of scolecite (CaAl2Si3O10·3H2O) measured on TOPAZ to locate the missing hydrogen atoms on the water molecules. Scolecite is the calcium member of the natrolite family within the zeolite group.  The cation and hydrogen bonding interaction of the water molecules with the framework plays an important role in fine-tuning the adsorption and electrostatic properties of the porous zeolite channels, which is fundamental for applications in separation science and energy storage materials. Single crystal data collection strategy will be optimized with the CrystalPlan program; peak integration will be performed in 3D Q-space in Mantid. Data reduction including neutron TOF spectrum, detector efficiency, and absorption corrections will be carried out with the ANVRED3 program. The structure will be refined using GSAS. The option to refine the neutron structure in SHELX 2014 and JANA2006 will also be explored.

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

*Dynamics of Surfactant Micelles*

We will investigate the dynamics of sodium dodecyl sulfate (SDS) micelles. The goal of the experiment is to measure the effective diffusion coefficient of the SDS micelles suspended in heavy water. This "classic" NSE experiment will allow us to illustrate the basic principles of the NSE technique and the required measurements and corrections. We will go through the reduction process starting from raw data to the intermediate scattering function. Finally, by comparing the results with model calculations, we will show the link between the structure and the dynamics in colloidal fluids.

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

*Proton dynamics in phosphoric acid*

Phosphoric acid, H3PO4, is a tribasic acid commercially available an 85% aqueous solution. The annual production of phosphoric acid is in the tens of megatons range. It is used mainly in the production of fertilizers, but also in the food and cleaning agents industry. In its anhydrous form it crystallizes as a monoclinic solid with a complex network of hydrogen bonds. We will use VISION to examine proton dynamics phosphoric acid. Use will be made of the diffraction detector on the beam line, which permits the simultaneous collection of diffraction and inelastic data. VISION has a dedicated computer cluster for data analysis. In parallel with the experiment, we will calculate the vibrational spectrum of phosphoric acid with Density Functional Theory (DFT) to show how these calculations support spectral interpretation. Use of the new software, O'climax to convert computed vibrational modes to a density of vibrational states directly comparable with VISION data will be demonstrated.

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

*Dynamics of metal hydride systems: Harmonic oscillators and beyond*

The hydrogen in zirconium hydride (ZrH2) 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 ZrH2 as a function of energy and wavevector transfer, and determine how well it conforms to the predictions of the scattering from a harmonic oscillator. Practical applications of sample preparation, data collection and analysis will be given to generate the scattering function S(Q,ω) from the data. This will be compared to theoretical predictions based on the harmonic oscillator description, with a discussion of what may cause any discrepancies found. As time permits, other samples or experimental conditions (temperature, incident energy) will be measured to highlight differences in the energy spectra.

**N20:  SNS Sample Environment**

*High pressure synthesis in a neutron diamond anvil cell*

The Sample Environment Group in ORNL's Neutron Sciences Directorate supports experiments across all beamlines at SNS and HFIR that require dedicated environments for cooling, pressure, heating or also magnetic and electric fields. Furthermore, the Sample Environment Group is also actively involved in the development of new technologies and scientific efforts. A recent example thereof is the newest generation of neutron diamond anvil cells, a cell that allows record pressures and sample volumes. In this hands-on experiment, students will prepare and load such a neutron diamond cell with a silicon sample. They will pressurize the sample to ~15 GPa to metallize the silicon into the same structure as white tin. They will then recover the sample upon pressure release which will have transformed into a kinetically stable new phase. This will be verified by Raman spectroscopy.

**N21:  Low temperature,  HFIR Sample Environment**

*Operation of Liquid Helium Cryostats and Closed Cycle Refrigerators*

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