Experiments for Neutron X-ray School 2013

X-ray Experiments:

	Technique	**Beamline**
X1	Tomography	2-BM
X2a	X-ray Fluorescence Microscopy	2-ID-E
X2b	X-ray Fluorescence Microscopy	8-BM
X3	Inelastic X-ray Scattering	3-ID
X4a	X-ray Magnetic Circular Dichroism	4-ID-C
X4b	X-ray Magnetic Circular Dichroism	4-ID-D
X5a	X-ray Absorption Spectroscopy	5-BM
X5b	X-ray Absorption Spectroscopy	9-BM
X5c	X-ray Absorption Spectroscopy	20-BM
X6	Magnetic X-ray Scattering	6-ID
X7	Radiography	7-BM
X8	Time Resolved X-ray Diffraction	7-ID
X9	Grazing Incidence SAXS	8-ID-E
X10	Powder Diffraction	11-BM, 17-BM
X11	Pair Distribution Function	11-ID-B
X12	SAXS/WAXS	12-ID-B
X13	Liquid Surface Scattering	15-ID-C
X14	High Pressure Scattering	16-BM-D
X15	Nanoprobe X-ray Flourescence	26-ID
X16	Grazing Incidence Interface Diffraction	33-BM
X17	Coherent X-ray Diffraction	34-ID-C
X18	Micro-Laue Diffraction	34-ID-E

Neutron Experiments:

	Instrument	Beamline
N1-a	Triple-Axis Spectrometers	HFIR HB1
N1-b	Triple-Axis Spectrometers	HFIR CTAX
N2	Powder Diffractometer	HFIR HB2A
N3	Four-Circle Diffractometer	HFIR HB3A
N4	Neutron Imaging Station	HFIR CG1D
N5-a	Small Angle Neutron Scattering	HFIR CG2 General Purpose SANS
N5-b	Small Angle Neutron Scattering	HFIR CG3 Bio-SANS
N5-c	Small Angle Neutron Scattering	SNS BL6 EQ-SANS
N6	NOMAD Nanoscale-Ordered Materials Diffractometer	SNS BL1B
N7	BASIS Backscattering Spectrometer	SNS-BL2
N8	SNAP Spallation Neutrons at Pressure	SNS BL-3
N9	Magnetism Reflectometer	SNS BL4A
N10	Liquids Reflectometer	SNS BL4B
N11	VULCAN Engineering Materials Diffractometer	SNS BL7
N12	POWGEN Powder Diffractometer	SNS BL11A
N13	SEQUOIA Fine-Resolution Fermi Chopper Spectrometer	SNS BL17
N14	HYSPEC Hybrid Spectrometer	SNS BL-14B

X-ray Experiment descriptions:

X1: X-ray Tomography, 2-BM

"X-ray computed microtomography (CAT scans) of porous media and corrosion in Al"
Francesco Decarlo and Xanghui Xiao

Aluminum alloys are susceptible to intergranular corrosion in wet environments. This is a problem since the narrow regions of attack along grain boundaries can be sites where cracks initiate, leading to structural failure. X-ray microtomography at a synchrotron facility allows to monitor the development of intergranular corrosion in situ in an aqueous environment in real time offering a remarkable insight into the evolution of corrosion enabling us to measure the rate of attack along individual grain boundaries and the rate of growth of the width of the cavities compared with the length, and assess the interaction with microstructural features such as intermetallic particles. In this experiment we will describe how x-ray tomography is performed including basic tomography principles, sample mounting and alignment, data collection, data analysis and 3D rendering. Tomographic data will be collected to look at corrosion in Aluminum structures.

X2a,X2b: X-ray micro-fluorescence imaging of bio-samples, 2-ID-E & 8-BM

"Trace element micro-analysis of biological cells by X-ray fluorescence microscopy"
Sophie-Charlotte Gleber, Lydia Finney, and Stefan Vogt

Metals and other trace elements are essential for the existence of life as we know it. In any organism, there are only few intracellular processes that do not depend on the presence of metals or other trace elements. Hard x-ray fluorescence microscopy is a powerful technique to study the distribution and chemical state of the elements from Al, P to Cu, Zn and above, with high spatial resolution and very high sensitivity. Due to its inherent low background, x-ray fluorescence is particularly well suited to detect elements present only in trace quantities, down to the level of attograms. The elemental content is measured directly by using the characteristic fluorescence of atoms excited by the microfocused X-ray beam, without the need for element-sensitive dyes. In this experiment, we will map and quantify the elemental distributions of elements from Si to Zn in single cells, in mouse tissue sections, and correlate these with visible light micrographs obtained from the same samples.

X3: Inelastic X-ray Scattering 3-ID

"Inelastic X-ray Scattering on Phonons in Single Crystals"
Ahmet Alatas, and Ercan Alp

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 transfered energy.
During the NX-school, inelastic x-ray scattering experiments on single crystal aluminum will be demonstrated. We will determine sound velocity and elastic constant along [00L] direction from

dispersion curve and compare the results with the values found in the literature. Experiment will involve aligning sample to the beam and orienting single crystal before collecting energy spectrum.

X4a, X4b: X-ray magnetic circular dichroism - 4-ID-C or 4-ID-D

"Element selective magnetization measurements using XMCD"
Yong Choi, David Keavney and Daniel Haskel

X-ray magnetic circular dichroism (XMCD) measures the difference in absorption of circularly polarized x-rays by a magnetic material. This technique can be used to extract element and orbital specific magnetic information. In this experiment spectra will be taken at either the soft (C) or hard (D) x-ray beamlines on APS-4-ID. Most of the absorption edges that probe the primary magnetic electrons (3d and 4f) lie in the soft x-ray portion of the spectrum, which requires a windowless UHV (soft x-ray) beamline. Using soft x-rays, XMCD spectra will be taken of a trilayer film. The XMCD spectra as a function of applied magnet field will be taken for different elements to determine the field required to switch individual layers in the material. Using hard x-rays (~8000 eV), XMCD spectra will be taken of a rare-earth/transition-metal compound at several temperatures to determine the compensation temperature in the material.

X5a, X5b, X5c: X-ray Absorption Fine Structure - 5-BM, 9-BM, & 20-BM

"Polarization Dependent XAFS in High Tc Superconductors"
Denis Keane, Trudy Bolin, Tianpin Wu, Steve Heald, Mali Balasubramanian, Chugjun Sun

The polarization dependence of the XAFS can be very powerful in separating contributions from various bonds in layered materials. This will be demonstrated with measurements on cuprate-based High Tc superconductors. Oriented powder samples will be used along with polarized synchrotron radiation to isolate the XAFS signals from in-plane and out-of-plane bonding. This data will be analyzed using simple linear combination fitting for the near edge region, and first shell analysis for the EXAFS.

X6: Magnetic X-ray Scattering, 6-ID

"Resonant magnetic x-ray scattering from a rare-earth compound"
Zahir Islam and Jong-Woo Kim

This experiment will go over the basics of aligning a single crystal in a diffractometor. Magnetic Bragg diffraction peaks from a single crystal of a rare-earth compound will be measured and their intensity 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.

X7: Radiography, 6-ID

"Time resolved radiography of liquid fuel sprays"
Alan Kastergren

This experiment will demonstrate how to image the microsecond dynamics of liquid fuel sprays using fast x-ray imaging techniques.

X8: Time-resolved x-ray diffraction, 7-ID

"Time-resolved x-ray diffraction"
Don Walko

This experiment will consist of laser-pump/x-ray diffraction-probe measurements of crystalline solids. An ultrafast Ti:sapphire laser can be used to excite a variety of materials systems. X-ray Bragg diffraction is used to probe 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. In this measurement, the laser will be used to heat a thin metal film grown on a transparent substrate. The time-dependent shift of the film Bragg peak will act as a thermometer for the film, from which the conductance of the film/substrate interface will be measured.

X9: Grazing Incidence Small-Angle X-ray Scattering (GISAXS), 8-ID

"GISAXS from organic photovoltaic thin films"
Zhang Jiang, Joseph Strzalka

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% to over 8 %, 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 Angstroms to tens or 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.

X10: Powder diffraction – High and Low Resolution, 11-BM & 17-BM

" Hands-on high resolution and in-situ powder diffraction measurements & analysis "
Greg Halder, Matthew Suchomel, Bryan Toby, & Bob VonDreel

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 a modern synchrotron powder diffraction experiment, from sample preparation and data collection to 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 detectors and a wide range of in-situ sample environments. They will learn how to access and use these tools for their own science. The second day of this experiment will include an interactive tutorial on Rietveld Analysis methods

using the software package EXPGUI-GSAS to extract crystallographic structural details from their powder diffraction data.

X11: Pair Distribution Function, 11-ID-B

"Pair-Distribution-Function measurements with High-Energy X-rays."
Oleg Borkiewicz and Karena Chapman

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

X12: Small Angle X-ray Scattering, 12-ID

" Small Angle Scattering (SAXS) of biological, organic and inorganic systems."
Xiaobing Zuo and Byeongdu Lee

SAXS provides valuable structural information such as size, shape and particle interaction, and has been widely used in material sciences and structural biology. In this experiment, a SAXS pinhole apparatus will be introduced to perform measurements on a variety of different samples like proteins, polymers, nano-particles. The data will be analyzed and interpreted.

X13: X-ray liquid surface scattering, 15-ID-C

"Biomolecules at air-water interface"
Binhua Lin, Mati Meron and Mrinal Bera

Many biochemical processes and reactions occur at surfaces and interfaces. These include interactions between cells and the extracellular matrix, protein interactions at cell and organelle membranes, gas transfer at the lung tissue-air interface, and drug intake by cell membranes. Synchrotron x-ray surface scattering techniques are used to determine structure on the sub-nanometer length scale at soft, hydrated interfaces of biological interest. The goal of this experiment is to determine the structure and ordering of a Langmuir monolayer of phospholipid molecules, Dipalmitoylphosphatidylcholine (DPPC), which is the major constituent of lung surfactant (a Langmuir monolayer consists of a single layer of amphiphilic molecules supported at the air-water interface). We use Langmuir trough method to prepare the monolayer of DPPC at the surface of water. X-ray reflectivity (XR) technique will be used to measure the electron density profile (or structure of the monolayer) normal to the surface of water, and grazing incident x-ray diffraction (GIXD) will be used to measure the packing of the lipid molecules along the water surface. Results of those measurements will then be analyzed through model-fitting routines to determine the molecular structure and packing of the lipids at the surface of water.

X14: High-Pressure Powder Diffraction, 16-BM-D

"Pressure-induced structure phase transition in ZnO"
Changyong Park and Dmitry Popov

In this experiment, students will get familiar with the high-pressure XRD experiment procedure, observe the pressure-induced structural phase transition in ZnO using angle dispersive x-ray diffraction technique, and refine unit cell parameters of the low- and high-pressure phases of ZnO at high pressure.

X15: X-ray nano-fluorescence, 26-ID

"Nanoprobe X-ray Fluorescence Studies of Metal Impurity Decoration of Dislocations in Large-Area Solar Cells"
Volker Rose, Martin Holt, Robert Winarski, Ian McNulty, Mariana Bertoni

It is accepted throughout the photovoltaic community that the overall performance of entire modules is regulated by inhomogeneously distributed nanoscale defects inside the wafers. Over the years a variety of techniques have been used to map and characterize precipitates, grain boundaries and dislocations. However, in the race to achieve higher and higher resolutions, while studying industry-relevant material, many of these techniques fall short either due to the inherent resolution limitations of the equipment or because the combination of low defect spatial densities and strong heterogeneity, present a challenge to sample preparation and characterization.
In this experiment, we will demonstrate the future of synchrotron-based nanoprobe techniques for identifying defects in large volumes of commercial solar cell materials. We are going to utilize a state-of-the-art x-ray fluorescence nanoprobe beamline to identify the precise nature of performance limiting defects in commercial mc-Si solar cells. X-ray fluorescence with a beam spot size < 60 nm is used to characterize the contamination levels in solar cell materials.

X16: Grazing incidence interface diffraction, 33-BM

"Grazing incidence x-ray diffraction study of atomic modulations in ordered oxide films"
Phil Ryan and Jenia Karapetrova

The synthesis of complex oxide superlattices with single unit cell control and atomically sharp interfaces has opened new routes to stabilizing collective ordering phenomena in materials. Heterostructures of dissimilar complex oxides have received considerable interest due to the novel interfacial properties that emerge resulting from the competition between the spin, charge, or orbital ground states of the adjoining compounds. Superlattices can exhibit magnetic ordering temperatures that much higher than those measured in compositionally equivalent alloys. This experiment will use grazing incidence x-ray scattering to measure the structural properties of the superlattice and how it is related to the magnetic order.

X17: Coherent X-ray Diffraction Imaging, 34-ID-C

"Coherent X-ray Diffraction Imaging of Nanocrystals"
Ross Harder

The high brightness, and hence high degree of coherence of modern syncrotron 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 produce images from coherently scattered x-ray. This imaging method allows one to surpass the resolution limits of modern x-ray optics and also provides for an unencombered 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.

X18: X-ray micro-Laue diffraction, 34-ID-E

"Measurements of the crystal microstructure using X-ray Laue diffraction"
Ruqing Xu, Wenjun Liu, Jon Tischler

The X-ray Laue diffraction 3D microscopy takes advantages of high brightness of the source, advanced focusing K-B mirrors, depth profiling technique, and high speed area detector. It is a scanning diffraction microscopy developed at 34-ID beamline. It can provide detailed local structural information of crystalline materials, such as crystallographic orientation, orientation gradients, grain morphology, strain tensor, and lattice structure, with high spatial resolution of 300 nm and angular resolution of 0.01 degree. It is general and applicable to single crystal, polycrystalline, composite, deformed, and functionally-graded materials. Applications include studies of fundamental deformation processes, basic grain-growth behavior, electro-migration, solid-solution precipitation, and high-pressure mineral physics.

Neutron Experiment descriptions:

N1: Triple-Axis Spectrometers, HFIR HB1 & CTAX
Magnetic excitation and anisotropy in multiferroic $BiFeO_3$

Multiferroic materials, in which spontaneous ferroelectric polarization and magnetic order coexist, have been investigated intensively due to their potential industrial applications. Because the Néel temperature T_N~640 K is much higher than room temperature and also because of the large spontaneous electronic polarization (P~100 $\mu C/cm^2$), $BiFeO_3$ has attracted a lot of attention. We will measure the magnetic excitation in $BiFeO_3$ at room temperature. The excitation energy below 11 meV will be measured at CTAX. In combination with higher excitation energy measured at HB-1, the full magnetic dispersion relation will be determined. In particular, the low-energy gapped excitations allow the determination of the Dzyaloshinskii-Moriya interaction and single ion anisotropy.

N2: Powder Diffractometer, HFIR HB2A
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: Four-Circle Diffractometer, HFIR HB3A
Structure and lithium-ion motion in the triphylite LiFePO4 studied by single crystal diffraction

Triphylite, $Li(Fe,Mn)PO_4$, is a promising 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.

N4: Neutron Imaging Station, HFIR CG1D
Dynamics of fluid flow in permeable rock

The principle of neutron imaging is based on the attenuation from both absorption and scattering, of a directional neutron beam by the matter through which it passes. Neutron imaging is complementary to other imaging techniques such as X-rays. X-rays are scattered and absorbed by electrons, so absorption and scattering increase monotonically with atomic number. Neutrons, on the other hand, interact with nuclei and their scattering power does not vary in any regular way with atomic number. Several areas of research already benefit from neutron imaging, such as engineering, advanced material characterization, fluid-flow and/or two-phase flow devices, automotive technology, advanced manufacturing technology, applied sciences, aerospace, life and biological sciences, national security applications, etc. Neutrons are specifically well suited for imaging light atoms (hydrocarbons for example) buried in heavy atoms, and are capable of characterizing fluid flow (dynamics). Time resolved water uptake and flow in permeable rocks will be spatially mapped and measured via neutron imaging.

N5: Small Angle Neutron Scattering, HFIR CG2 General Purpose SANS
HFIR CG3 Bio-SANS
SNS BL6 EQ-SANS

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 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 (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.

N6: NOMAD Nanoscale-Ordered Materials Diffractometer, SNS BL1B
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 liquid water. We will look at the PDF of diamond and compare the PDF of bulk and nanocrystalline NiO.

N7: BASIS Backscattering, SNS BL2
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 new 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 recently synthesized ionic liquid $[H_2NC(dma)_2][BETI]$.

N8: SNAP Spallation Neutrons at Pressure, SNS BL3
Pressure-induced phase transitions of water at room temperature

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.

N9: Magnetism Reflectometer, SNS BL4A
Revealing magnetism in thin films of normally non-magnetic materials

Understanding the magnetic properties of complex materials near surfaces and interfaces is of critical importance 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 the study of interfacial magnetism in $LaMnO_3$-thin film epitaxially grown on $SrTiO_3$ 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 down 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.

N10: Liquids Reflectometer, SNS BL4B
Polymer self-diffusion studied by specular reflectivity

Isotopic substitution is a powerful tool in neutron scattering studies. In this experiment we will observe the self-diffusion of polystyrene (PS) by means of a 500-Å-thick deuterated (dPS) layer float-deposited atop a spin-coated 500-Å-thick protonated PS layer on a silicon substrate. Students will prepare the film in the beamline 4B wet lab and measure specular reflectivity. We will then anneal the sample for ~30 min in a vacuum oven and re-measure the reflectivity. Students will fit the data from the two runs to observe changes in the interfacial width of the dPS/PS.

N11: VULCAN Engineering Materials Diffractometer, SNS BL7
Non-destructive residual stress/strain measurement of weld by neutron diffraction

Residual stresses in engineering component are important to structure lifetime reliability and durability. During welding, severe residual stresses are commonly built up across the weld metal (WM), heat affected zone (HZ) and base metal (BM). The variation of chemical composition and microstructure will also affect the accurate measurement residual stress. Using Time-of-Flight neutron diffraction on VULCAN instrument, the residual stress/strain and the phase distribution can be spatially resolved by engineering diffraction. In the experiment, a weld-bead-on-plate sample will be used for demonstrating the non-destructive residual stress measurement. A stress-free coupon sample, which has similar chemistry of the weld sample, will be used as the reference. Single peak and Rietveld refinement will be used to determine the residual strain and phase concentration of each measurement location, respectively.

N12: POWGEN Powder Diffractometer, SNS BL11A
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) and also reduce data using MantidPlot generating GSAS & Fullprof normalized diffraction data files. 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 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: Finally for those who want to refine a more complex structure, we will look at several models to determine the true crystal structure of Ba_2CuWO_6, which shows a Jahn-Teller distortion.

N13: Fine-Resolution Fermi Chopper Spectrometer (SEQUOIA), SNS BL17
Dynamics of metal hydride systems: Harmonic oscillators and beyond

The hydrogen in zirconium hydride (ZrH2) sits at the interstitial positions between the zirconium. At the simplest description, the energy levels 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 sample preparation, data collection and analysis will be given to generate the scattering function $S(Q,\omega)$ from the data. This will be compared to theoretical predictions based on the harmonic oscillator description, with a discussion of what may cause any discrepancies found. As time permits, other metal hydrides will be measured to highlight differences in their energy spectra.

N14: Hybrid Spectrometer (HYSPEC), SNS BL14A
Spin-wave excitations in the orbital-spin coupled system MnV_2O_4

The Hybrid Spectrometer (HYSPEC) is a unique instrument whose concept combines the time-of-flight spectroscopy with the focusing Bragg optics by using the TOF for selecting the neutron energy and a vertically-curved crystal monochromator for concentrating the neutron flux on sample. It is optimized for detailed investigations of the low-energy atomic-scale dynamical properties of crystalline solids. Transition-metal spinels, AB_2X_4, have been for many years the subject of intense experimental and theoretical activity. Structurally, the most interesting feature of these systems is the fact that the B cation occupies the nodes of a pyrochlore lattice, which is known to be geometrically frustrated. In the spinel MnV_2O_4, the octahedral site is occupied by the V^{3+} ion having two 3d

electrons in threefold t_{2g} levels. This compound exhibits two transitions to long-range ordered ferromagnetic states, the first collinear and the second noncollinear. The lower temperature magnetic transition is accompanied by a structural distortion to an orbitally ordered tetragonal phase. The HYSPEC spectrometer will be used to investigate the low-energy spin-wave excitations in a MnV_2O_4 single crystal. The exercise will enable students to get hands-on experience with crystal alignment procedures, as well as on data processing using data reduction and plotting programs.