X-ray Experiments:

	Technique	**Beamline**
X1-a	Powder diffraction – High and Low Resolution	1-BM
X1-b	Powder diffraction – High and Low Resolution	11-BM
X2	High Energy Diffraction – Stress & Strain	1-ID
X3	X-ray Tomography	2-BM
X4	X-ray micro-fluorescence imaging of bio-samples	2-ID-E
X5	Inelastic X-ray Scattering	3-ID
X6-a	X-ray magnetic circular dichroism	4-ID-C
X6-b	X-ray magnetic circular dichroism	4-ID-D
X7	Magnetic X-ray Scattering	6-ID
X8	Time-resolved x-ray diffraction	7-ID
X9	Grazing Incidence Small-Angle X-ray Scattering	8-ID
X10	Pair Distribution Function	11-ID-B
X11	Small Angle X-ray Scattering	12-ID
X12	Single crystal diffraction	13-BM-C
X13	X-ray liquid surface scattering	15-ID-C
X14	High-Pressure Powder Diffraction	16-BM-B
X15	Fiber Diffraction	18-ID
X16-a	X-ray Absorption Fine Structure	5-BM-D
X16-b	X-ray Absorption Fine Structure	20-BM-B
X17	X-ray nano-fluorescence	26-ID
X18	Grazing incidence interface diffraction	33-BM-C
X19	Coherent X-ray Diffraction Imaging	34-ID-C
X20	X-ray micro-Laue diffraction	34-ID-D

X-ray Experiment Schedule:

	Week 1 (X-ray)			
Group	12-Aug	14-Aug	15-Aug	16-Aug
A	X20	X20	X2	X3
B	X19	X19	X7	X6-a
C	X12	X16-a	X20	X20
D	X8	X6-b	X19	X19
E	X13	X13	X11	X16-a
F	X3	X10	X10	X9
G	X17	X10	X10	X16-b
H	X11	X1-a	X6-b	X1-a
I	X4	X1-b	X16-b	X1-b
J	X14	X14	X5	X18
K	X18	X8	X5	X7
L	X15	X4	X9	X11
M	X17 (M1) X12 (M2)	X2	X3	X16-b

Two day experiment

X-ray Experiment descriptions:

X1: Powder diffraction – High and Low Resolution, 1-BM & 11-BM
Hands-on high resolution and in-situ powder diffraction measurements & analysis
Greg Halder, John Okasinski, Matthew Suchomel, Bryan Toby, Lynn Ribaud, & 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.

X2: High Energy Diffraction – Stress & Strain, 1-ID
Strain and texture measurements in polycrystalline bulk samples using high-energy X-rays
Jon Almer

Polycrystalline materials encompass large groups of materials such as metals, ceramics and minerals. For many applications it is crucial to understand the structure-performance relationships of such materials under thermo-mechanical processing, e.g. rolling, annealing. We concentrate here on the internal strains and stresses as well as grain orientation aspects (texture). The properties often depend on the local position within the sample and may be mapped if the spatial resolution of the probe is sufficient. The dynamical behavior at surfaces is often not representative of the bulk due to effects such as stress relaxation or abnormal grain growth. Therefore a bulk penetrating probe is required such as high energy X-rays (40 to 100 keV). Third generation high-energy synchrotrons like the APS provide high energy X-rays of unprecedented brilliance enabling high spatial resolution and, in combination with 2D-detectors, fast data acquisition. High energy X-rays are therefore particularly suited for in-situ investigations and rather complementary to neutrons, which in general provide even higher penetration power but substantially coarser spatial resolution and slower data acquisition. In this experiment we will use high-energy x-rays to monitor strain and texture in a polycrystalline sample under in-situ mechanical loading.

X3: 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.

X4: X-ray micro-fluorescence imaging of bio-samples, 2-ID-E
Trace element micro-analysis of biological cells by X-ray fluorescence microscopy
Sophie-Charlotte Gleber, Stefan Vogt, and Lydia Finney

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.

X5: Inelastic X-ray Scattering, 3-ID
Inelastic X-ray Scattering on Phonons in Single Crystals
Ahmet Alatas, Ayman Said, Bogdan Leu, 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.
Advanced Photon Source has two partially dedicated beamlines (Sector 3 and 30), with very high-energy resolution (1.5-2 meV), specilized for studying collective excitations

(phonons) where their energies lie in the order of milli-electronvolts (meV). IXS is very important technique in aplications ranging from condensed matter physics to life science and mineral physcis to geophysics.
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.

X6: X-ray magnetic circular dichroism, 4-ID-C or 4-ID-D
Element selective magnetization measurements using XMCD
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.

X7: 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.

X8: Time-resolved x-ray diffraction, 7-ID
Time-resolved x-ray diffraction
Haidan Wen and Bernhard Adams

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
Wei Chen, 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: Pair Distribution Function, 11-ID-B
Pair-Distribution-Function measurements with High-Energy X-rays.
Peter Chupas 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.

X11: 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.

X12: Single crystal diffraction, 13-BM-C
Single crystal monochromatic diffraction on minerals.
Przemek Dera, Sergey Tkachev, Tony Lanzerotti

Single-crystal X-ray diffraction (SXD) is one of the principal methods for characterization of structure of crystalline solids, and has been extensively used in Earth science research. SXD can provide structural information of very high precision and accuracy. The information is averaged over the volume of the illuminated sample and is not element-selective. The combination of diffraction angles and intensities provides a characteristic of a mineral, and therefore constitutes a powerful tool for phase identification through search/match routines using crystallographic databases, etc. From the geometrical distribution of diffraction effects (diffraction angles) the geometry of the crystal lattice, its orientation, and the unit cells parameters can be reliably determined. Lattice parameters not only represent a fundamental component in the structural characterization of a material, but from these a wealth of geologically relevant information may be derived. Provided the composition is known, the mineral density may be calculated, a parameter essential to the modeling of the Earth's interior and of processes such as the segregation of crystals in magma and planetary differentiation. Experimental site occupancies give the intracrystalline atomic distribution of binary solid solutions as long as atomic species differ sufficiently in atomic number. The systematic characterization of mineral structures leads to the development of predictive models of the crystal chemistry of minerals, and empirical trends in the behavior of minerals. Structural parameters from diffraction analysis are often indispensable information in the interpretation of spectroscopic results.
In this experiment, students will gain hands-on experience with all aspects of a modern synchrotron monochromatic single crystal diffraction experiment with area detector from sample preparation (natural mineral sample will be used) through data collection to structure determination.

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-B
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: Fiber Diffraction , 18-ID

Microfocus fiber diffraction. Refinement of collagen macromolecular structure within fibrils.
Olga Antipova, Joseph Orgel, Tom Irving, Raul Barrea

X-ray fiber diffraction is a method that provides information about specific molecular arrangements within fibrils. Fibril-forming proteins have unique structure that cannot be analyzed by conventional X-ray crystallographic methods which require protein extraction and crystallization in special conditions that may be far from native. In this experiment students will participate in tissue sample handling, data collection and initial analysis. Students will observe nanoscale tissue heterogenity detected by micro-focused beam and will learn how to use Fit2D software to analyze diffraction patterns and extract information about molecular arrangements and interactions within collagen fibrils.

X16: X-ray Absorption Fine Structure - 20-BM-B & 5-BM-D
Polarization Dependent XAFS in High Tc Superconductors
Steve Heald and Denis Keane

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.

X17: 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.

X18: Grazing incidence interface diffraction, 33-BM-C

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.

X19: 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.

X20: X-ray micro-Laue diffraction, 34-ID-D
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.