

X-ray Experiments descriptions:

X1: High Energy Diffraction – Stress & Strain, 1-ID

“Strain and texture measurements in polycrystalline bulk samples using high-energy X-rays”

Jon Almer & Dean Haeffner

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.

X2: High Resolution Powder Diffraction, 11-BM

“Pocket Full of Kryptonite”

Matthew Suchomel, Brian Toby, Lynn Ribaud, & Bob VonDreele

Students will be given a chance to participate in robotic-assisted data collection on the 11-BM high-resolution 12-analyzer powder diffractometer. They will use customized beamline software to prepare and perform scans on a sample of the mineral Jadarite. Students will be given the opportunity to learn about the Rietveld method, and will use GSAS to perform their own Rietveld refinement of collected 11-BM data. In the end, a three-dimensional atomic structure of the Jadarite sample will be determined; a compound that has the identical chemical formula as marked on the crate of 'kryptonite' Lex Luthor obtained in “Superman Returns”. This 'kryptonite', however, should only help your x-ray vision. Instructional material on GSAS and the Rietveld method will be provided.

X3: 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 it 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.

X4: High-Pressure Powder Diffraction, 16-ID

"Pressure-induced structure phase transition in ZnO"

Yue Meng

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.

X5: X-ray micro-Laue diffraction, 34-ID-E

"Measurements of the x-ray microstructure using Laue diffraction"

Wenjun Liu

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 multiple area detectors. It can provide detailed local structural information of crystalline materials, such as crystallographic orientation, orientation gradients, and strain tensors. Applications include studies of fundamental deformation processes, basic grain-growth behavior, electro-migration, solid-solution precipitation, and high-pressure mineral physics.

X6: 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.

X7: Coherent X-ray Diffraction Imaging, 34-ID-D

"Coherent X-ray Diffraction Imaging of Nanocrystals"

Ross Harder

Synchrotron sources produce a high coherent flux enabling new imaging techniques to be developed. Coherent X-ray Diffraction (CXD) imaging can be used to produce wavelength limited resolution images. Though not yet at angstrom resolution images of nanocrystalline objects in three dimensions at resolution equal to or better than typical x-ray lenses have been shown. Further, the CXD around the Bragg spots of crystalline samples can be used to image the distortions of the lattice planes of a crystal due to strain in the sample. In this experiment the CXD from small (typically 500nm) gold crystals will be measured. The three dimensional CXD pattern around a Bragg spot of the crystal, will then be inverted to a three dimensional image of the density of the crystal using computational algorithms.

X8: X-ray Tomography, 2-BM

"X-ray computed microtomography (CAT scans) of corrosion in Al"

Francesco Decarlo and Xanghui Xiao

In this experiment, the basics of how to perform x-ray computed micro-tomography will be covered. This includes basic principles, sample mounting and alignment, data collection, data analysis and 3D rendering will be covered. A tomographic data will collected to look at the corrosion in Aluminum structures.

X9: X-ray micro-fluorescence imaging of bio-samples, 2-ID-E

"Trace element micro-analysis of biological cells by X-ray fluorescence microscopy"

Jesse Ward, Gin-Fu Peter Chen, 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.

X10: X-ray nano-fluorescence imaging of Geopolymers, 26-ID

"Hard x-ray nanoprobe fluorescence characterization of geopolymers"

Volker Rose, Martin Holt, Robert Winarski, and Jorg Maser

Geopolymers are currently being developed as an environmentally beneficial replacement to Portland cement for concrete production, offering comparable performance and cost while reducing greenhouse emissions by approximately a factor of 5. Given that cement production is responsible for up to 8% of global anthropogenic CO₂ emissions, this equates to the opportunity to reduce CO₂ by at least tens of millions of tons per annum worldwide. The spatial resolution and penetration power of the hard x-ray nanoprobe (HXN) on 26-ID permit the study of the nanoscale heterogeneity of the geopolymer, which is critical in order to optimize their performance. We will utilize the HXN to obtain fluorescence maps of a hydroxide, an activated geopolymer, in order to map the Chromium contamination which is a potential issue in the valorization of the geopolymers. This experiment intends to study the correlation between chromium and iron, which will help to understand if chromium is likely to be released problematically into the environment.

X11. X-ray Absorption Fine Structure - 20BM

"Polarization Dependent XAFS in High Tc Superconductors"

Steve Heald

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.

X12: X-ray magnetic circular dichroism - 4-ID-C or 4-ID-D

"Element selective magnetization measurements using XMCD"

John Freeland, David Keavney, Daniel Haskel, and Jonathan Lang

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.

X13: Magnetic X-ray Scattering, 6-ID

"Resonant magnetic x-ray scattering from a Ho single crystal"

Zahir Islam and Jong-Woo Kim

This experiment will go over the basics of aligning a single crystal in a diffractometer. Magnetic Bragg diffraction peaks from a Ho single crystal will be measured and their intensity compared to that of the structural charge peaks on and off resonance. The size of the moment and wave vector of the magnetic peak will be measured as a function of temperature.

X14: Nuclear Resonant Inelastic X-ray Scattering, 3-ID

"Measurement of the phonon density of states using NRIXS from Iron compounds "

Ercan Alp and Ahmet Alatas

Nuclear resonant inelastic x-ray scattering (NRIXS) is a method that uses nuclei with suitable resonances to measure the vibrational density of states of solid materials. The method is very sensitive to small amounts of material and takes advantage of the high brilliance of synchrotron radiation, which makes micrometer-sized x-ray beams with high intensity possible. These properties together with the isotope selectivity allowed NRIXS investigations on materials under pressures in the Mbar regime using diamond anvil cells and Laser heating, on proteins and enzymes to identify vibrational modes specific to their active sites. In this experiment, nuclear resonance concepts and time discrimination techniques will be explained and used to extract thermodynamic properties like specific heat, vibrational entropy, as well as the sound velocity in Iron compounds.

X15: Small Angle X-ray Scattering , 5-ID or 18-ID

"Small-Angle X-ray Scattering from Dilute Monodispersed Proteins or RNA"

Steven Weigand (DND-CAT) or Liang Guo (Bio-CAT)

Students will do SAXS measurements on either of two beamlines. On beamline 5-ID (DND-CAT), small-angle X-ray scattering data will be collected and analyzed from several dilute protein solutions using the in-vacuum capillary flow-cell. Students will also get experience with

the ATSAS software package, as they construct dummy-atom models based on the data collected. On 19-ID (Bio-CAT), students will have chance to collect small-angle x-ray scattering data of protein or RNA samples in solution, reduce data from 2D to 1D format and perform basic analysis on the reduced data. Students will also gain experience in using available modeling programs to construct low-resolution models of proteins and RNAs.

X16: Grazing Incidence Small-Angle X-ray Scattering (GISAXS), 8-ID

“Self-Assembly of Two-Dimensional Nanostructures Studied by GISAXS”

Zhang Jiang, Xiao-Min Lin, Joseph Strzalka

Grazing-incidence small-angle x-ray scattering (GISAXS) is a tool to probe nanostructures at surfaces, interfaces, and within thin films. In this experiment, students will apply in-situ GISAXS to observe the self-assembly of colloidal nanoparticles at the liquid/vapor interface. Students will gain familiarity with GISAXS principles, instrument operation procedure, and data analysis.

X17: Time-Resolved Macromolecular crystallography, 14-ID

“Time-resolved pump-probe experiments of protein crystals”

Vukica Srajer

This experiment will provide hands-on experience in conducting pump-probe time-resolved crystallographic experiments involving protein crystals. Picosecond laser pulses will be used to initiate a reaction in a protein crystal, while a synchronized 100ps X-ray pulses will probe structural changes in the crystal at a given time delay following the laser pulse. Collected time-resolved Laue data will be processed using Precognition software package

X18: Time-resolved x-ray diffraction, 7-ID

“Time-resolved x-ray diffraction from semiconducting materials”

Don Walko and Eric Dufresne

This experiment will consist of laser-pump/x-ray diffraction-probe measurements of crystalline solids. An ultrafast Ti:sapphire laser will 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. The first part of this experiment will be to use the laser to excite coherent acoustic strain waves in a semiconductor sample; the strain-induced deformation of the Bragg peak will be observed. The second part of this experiment will be to use the laser 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.