National School of Neutron and X-ray Scattering
X-ray Experiments 2007

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X1: EXAFS

"Temperature dependent EXAFS of Cu and determination of the valence of Cr compounds"

The EXAFS of Cu metal will be measured as a function of temperature to illustrate the types of information available in an EXAFS experiment. This will be analyzed to determine the Cu vibrational properties. A XANES experiment will be conducted on some Cr compounds to illustrate how such measurements can be used to determine chemical valence.

EXAFS, 20BM

X2: Liquid Scattering

"Liquid Surface Scattering Study of a Gold Nanosphere Monolayer on the Surface of Water"

We measure the ordering and morphology of the gold nanospheres (7nm in diameter) floating on the surface of water using x-ray reflectivity (XR) and grazing incident X-ray diffraction (GIXD). We obtain information on the morphology of the film of the spheres normal to the surface using XR, and the packing pattern of the spheres along the surface using GIXD.

Liquid Scattering, 15ID

X3: Pair Distribution Function

"Pair-Distribution-Function measurements with High-Energy X-rays."

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.

PDF, 11ID-B

X4: Microtomography

"Micro tomography of a sea urchin spine."

During this experiment we will perform a full tomography system alignment, collect and analyze multiple sea urchin spines at 1 um resolution and perform a 3-D rendering of the volumetric data.

Tomography, 2BM

X5: USAXS

"USAXS studies of structure of common materials"

We will use ultra-small angle X-ray scattering technique to study microstructure of common household materials from nanometers to microns. Examples of materials will be provided, but safe and appropriate materials brought by students will be considered for experiments also. Students will collect, reduce and analyze data. During this experiments students will be provided with introduction to "Irena" small-angle scattering analysis and modeling software and handout for different methods of SAS data analysis will be provided. If students bring their own results, they can analyze them under supervision of "Irena" author.

USAXS, 32ID

X6: High Energy Diffraction

"Texture and internal strain measurements using high-energy x-rays"

High-energy x-ray diffraction will be used to determine the microstructure, average grain orientation (texture) and internal strain in a multi-phase polycrystalline material. These properties will be measured in-situ, as a function of applied tensile load.

High Energy Diffraction, 1ID

X7: High Pressure Powder Diffraction

"Pressure-induced structure phase transition in ZnO"

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.

High Pressure powder, 16ID-B

X8: Biomaterials

"Microprobe x-ray fluorescence and x-ray spectroscopy on biological samples"

X-ray fluorescence on biological samples will be performed at the 18ID BioCAT microprobe. A 5 micron size x-ray beam will be use to probe normal and tumor tissue samples to generate the elemental mapping composition of the samples. In particular we will be looking for maps of Ca, Fe, Cu and Zn.
On the second day, X-ray absorption spectroscopy will be performed on the same samples. Based on the mapping results, selected sample spots will be scanned to obtain XANES spectra at the Cu k edge. Comparison with Cu standards will be also performed to demonstrate the ability of the technique to identify different Cu species.

BIOCAT, 18ID

X9: XMCD

"Single crystal magnetic x-ray scattering"

This experiment will go over the basics of aligning a single crystal in a diffractometor. Magnetic Bragg diffraction peaks will be measured and their intensity compared to that of the charge scatting peaks. The intensity and wave vector of the magnetic peak will be measured as a function of temperature.

XMCD (6ID)

X10: High Resolution Powder Diffraction
"High resolution powder diffraction and Rietveld analysis"

Students will be given a chance to participate in robotic data collection on the newly completed 11-BM high resolution 12-analyzer powder diffractometer. They will then preprocess that data to a pseudo-single scan dataset. Finally, students will be given the opportunity to perform a Rietveld refinement to fit a structural model to a set of 11-BM data using GSAS. A menu of self-guided instructional materials on the Rietveld method will be provided.

Powder, 11BM

X11: Inelastic X-ray Scattering
"Inelastic X-ray Scattering on Phonons in Single Crystals and Liquids"

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 sample, 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 – information on correlations in time is obtained. One can observe scattering from thermally excited phonons in solids or in liquids diffusion processes or viscous damping of sound waves when these phonon excitations are much closer to the elastic line and with much better energy resolution in the order of milli-electronvolts (meV). Advanced Photon Source has two dedicated beamlines (Sector 3, newly operational Sector 30) for high energy resolution inelastic x-ray scattering experiments. It is also becoming very important tool in the geophysic community since the determination of sound velocity under extreme condition (high pressure – high temperature) of the sample becomes available in this method.

During the NX-school, students will be separated into two groups and two inelastic x-ray scattering experiments will be demonstrated. These experiments are as follow:

Determination of an elastic constant in an aluminum single crystal: Orient the crystal in transmission geometry to the beam with the 001 direction in the scattering plane. Make a quick energy scan at zero scattering angle to find the energy zero point. Measure the phonon positions at (0/0/1.8) and (0/0/1.7). Draw a dispersion curve and calculate the longitudinal sound velocity in this direction. Compare the sound velocity and the elastic constant with values found in the literature.

Measurement of the high frequency sound velocity in water: Prepare a sample container filled with water with about 10 mm diameter. Align it to the beam. Go to momentum transfers of Q = 3 nm^{-1}, 5 nm^{-1} and measure the dynamic structure factor S symmetrically around the elastic energy. Determine the phonon position. Calculate the so-called current-current correlation function and determine the peak position here. Plot a dispersion relation and determine the sound velocity. Compare it to the macroscopic sound velocity of water.

IXS, 3ID

X12: X-ray Fluorescence

"Trace element micro-analysis in biological cells using scanning X-ray fluorescence microscopy"

Scanning X-ray fluorescence microscopy uses focused hard X-rays (e.g., 10keV) to measure & map the (trace) element content of samples. In this experiment, we will map and quantify the elemental distributions of elements from Si to Zn in single cells, and correlate these with visible light micrographs obtained from the same cells.

X-ray fluorescence 2ID-E

X13: SAXS

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

A SAXS pinhole apparatus will be introduced to perform measurements on a variety of different samples like cytochrome c, polyethylene and Au nano particles. The data will be analyzed and interpreted. Other examples for SAXS measurements can be suggested.

SAXS, 12ID