Neutron Experiments descriptions:

Sept. 29, Sept. 30, Oct. 1

1. Triple-axis spectrometers, HB-1A, HB-1, and HB-3:
"Spin wave and phonon dispersion in Fe-Ga solid solutions"
(Jaime Fernandez-Baca, Mark Lumsden, Andre Zheludev, Jerel Zarestky)

Fe-Ga alloys with appropriate composition and heat treatment, exhibit giant magnetostriction in a polycrystalline and ductile form.[1,2] The tetragonal magnetostriction coefficient, λ_{100}, of Fe-Ga can be up to 15 times that of pure Fe. This makes these materials of tremendous scientific interest as well as technological interest for use in devices such as actuators, transducers and sensors. Elastic constant measurements[3] show that the shear elastic constant $1/2(C_{11}\text{-}C_{12})$ 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 $T_2[110]$ 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.

In the neutron school experiments at HB1, HB1A and HB3, we will use samples of three compositions of Fe-Ga alloys in to measure both phonon and spin wave neutron groups at room temperature.

[1]A. E. Clark, K. B. Hathaway, M. Wun-Fogle, J. B. Restorff, T. A. Lograsso, V. M. Keppens, G. Petculescu, and R. A. Taylor, *J. Appl. Phys.*, **93**, 8621 (2003).
[2] G. Petculescu, K. B. Hathaway, T. A. Lograsso, M. Wun-Fogle, and A. E. Clark, *J. Appl. Phys.*, **97**, 10M315 (2005).
[3] M. Wuttig, L. Dai, and J. R. Cullen, *Appl. Phys. Let.*, **80**, 1135 (2002).

2. Wide Angle Neutron Diffraction:
"Hausmannite Mn_3O_4: Magnetic Structure Revisisted"
(Ovidiu Garlea)
Neutron diffraction measurements will be performed to investigate the onset of long-range magnetic order in the Mn_3O_4 hausmannite [1,2]. Data will be collected at various temperatures, ranging from 300 K to 5 K, using the Wide-Angle Neutron Diffractometer (WAND) at the HFIR. Rietveld analysis of the crystal and low-temperature magnetic structure will be carried out using FullProf Suite software. The obtained results will be discussed and compared with those reported in earlier studies [1,2].

1. B. Boucher, R. Buhl and M. Perrin, J. Phys. Chem. Solids 32, 2429 (1971).
2. G. B. Jensen and O. V. Nielsen, J. Phys. C: Solid State Phys. 7, 409 (1974).

Sample description: Mn_3O_4 powder, Volume of sample ~ 1.5 cc
Sample environment: 4 K Displex

3. Small angle Nuetron Scattering:
"*Micellar Morphologies in Self-Associated Triblock Copolymer Solutions: Effects of Concentration*"
(Ken Littrell)

The PEO-PPO-PEO triblock copolymers have important applications in industry and medicine. Because of the differing 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 most appropriately measured in nanometers. Thus, small-angle neutron scattering (SANS) is a probe uniquely well-suited to studying this phase behavior. In these experiments we will probe the effects of concentration and ionic strength on block copolymer self-assembly using solutions of 1,2, and 5 weight% Pluronics F108 triblock copolymer in D20 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.

4. Quasi-Elastic Neutron Scattering - BASIS:
"Diffusion dynamics of protons in a novel ionic liquid designed for proton-exchange membranes"
(Eugene Mamontov)

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 $[H_2NC(dma)_2][BETI]$.

Sample:
Ionic liquid $(NH_2)(CN_2)(CH_3)_4$-$(CF_3)(CF_2)(S(O)_2)N(S(O)_2)(CF_2)(CF_3)$ in a sealed can in a closed cycle refirgerator

5. Liquids Reflectometer:
"Polymer self-diffusion studied by specular reflectivity"
(John Ankner)

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 mins 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. We will have backup samples ready in case deposition fails for some reason.

6. ARCS:
"Dynamics of metal hydride systems: Harmonic oscillators and beyond"
(Doug Abernathy, Matt Stone)

The hydrogen in zirconium hydride (ZrH_2) 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.
Samples: Zirconium hydride (ZrH_2); Titanium hydride (TiH_2); Sodium aluminum hydride ($NaAlH_4$)
Sample environment: Room temperature holder or CCR

7. Bio-SANS
"Protein unfolding studied by small-angle neutron scattering"
(Volker Urban, William Heller)

Small-angle neutron scattering (SANS) is a powerful tool for looking at the conformation of biological macromolecules in solution. SANS is particularly sensitive to conformational changes of proteins and nucleic acids in response to applied stimuli, such as temperature, pressure or small molecules. We will study the solution conformation of human serum albumin, a multifunction protein found in the blood, and how it changes in response to urea, a protein denaturant, using the BioSANS instrument at HFIR. Various methods of fitting the data will be employed to extract the molecular weight of the scattering particle, the radius of gyration, the distance distribution function P(r) and the maximum linear dimension. Methods for developing models of the protein from SANS data will also be discussed.

Samples to be measured (4 total):
Human Serum Albumin in aqueous solution and associated buffer solution
Human Serum Albumin in aqueous solution with 6 M urea and associated buffer solution

X-ray Experiments descriptions:

October 5,6

X1: High Resolution Powder Diffraction – 11-BM
"High resolution powder diffraction and Rietveld analysis"
Brian Toby

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.

X2: X-ray magnetic circular dichroism - 4-ID-C & 4-ID-D
"Element selective magnetization measurements using XMCD"
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, XMCD spectra will be taken of a rare-earth/transition-metal compound at both soft x-ray (~800 eV) and hard x-ray (~8000 eV) resonances.

X3: Pair Distribution Function, 11-ID-B
"Pair-Distribution-Function measurements with High-Energy X-rays."
Peter Chupas

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.

X4: Small Angle X-ray Scattering, 12-ID
"Small Angle Scattering (SAXS) of biological, organic and inorganic systems"
Soenke Seifert

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.

X5: X-ray Tomography, 13-BM
"X-ray computed microtomography (CAT scans) of porous media"
Mark Rivers

X-ray computed microtomography will be performed on a sample containing solids, water, and oil. The water will be doped with cesium and the oil doped with iodine. By collecting tomographic data sets above and below the K x-ray absorption edges of these elements the complete 3-D distribution of the liquids and solids will be determined. Such information can be used, for example, to study the efficiency of environmental remediation techniques, or of methods for enhanced petroleum extraction.

October 8,9

X6. X-ray Absorption Fine Structure - 20BM
"XANES of Cr compounds and temperature dependent XAFS of Cu metal
Steve Heald

Near edge spectra (XANES) of a Cr containing sample will be compared to standard materials to determine the Cr valence. In environmental studies it is important to distinguish highly toxic chromate from less toxic Cr(III). For a Cu metal sample temperature dependent measurements of the EXAFS will be used to determine the vibrational properties of the Cu atoms. This will demonstrate the detailed analysis of EXAFS to determine structural and vibrational parameters.

X7. Surface X-ray Scattering - 33ID
"In-situ surface x-ray diffraction to search quantum size effects in metal films on oxide surfaces"
Hawoong Hong

XOR-UNI sector-33 has a surface diffractometer, which is specially designed for diffraction works to study films and surfaces in UHV. Ultra thin films can exhibit unique structural behavior due to the quantum confinement of electrons in films along its thickness direction. This quantum size effect has not been reported on oxide surfaces. We have been gathering evidences toward quantum size effects of metal films on oxides, particularly on sapphire surfaces. The sapphire surface is not very stable as one might guess. The surface can be significantly modified by high temperature annealing, and can be etched by water. Also vacuum heating can drive out oxygen and change the surface composition. Students will pick one of the surface treatment options for the start and then grow a Pb film to a selected thickness (< 20 ML). The reflectivity (the specular rod) and/or crystal truncation rods will be measured before and after the film deposition. Analysis will be attempted to figure out the starting surface structures, and the film structure and morphology. We will try to find the evidence of quantum size effects from the film structure.

X8: X-ray fluorescence imaging, 2-ID-E
"Imaging Trace Elements in Cells with X-Ray Fluorescence Microscopy"
Stefan Vogt

The presence of metals and trace elements is essential for the existence of life as we know it. In any organism, there are very few intracellular processes that are not dependent on the presence of metals or other trace elements; trace metals are also increasingly recognized as having a critical impact on human health both in their natural occurrence, via therapeutic drugs, and in diseases such as Alzheimer's.
Hard x-ray fluorescence microscopy is a powerful technique to map and quantify element distributions in biological specimens such as cells and bacteria. Due to its inherent low background, x-ray fluorescence is particularly well suited to detect metals 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. Typically, 10-15 elements are mapped simultaneously, leading to precise elemental colocalization maps.
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.

X9: High Pressure Powder Diffraction, 16ID,
"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.

X10: X-ray Photon Correlation Spectroscopy, 8ID
"X-Ray Photon Correlation Spectroscopy from Colloidal Suspensions"
Alec Sandy

X-Ray Photon Correlation Spectroscopy (XPCS) is the x-ray analog of Dynamic Light Scattering (DLS) (which is performed with visible-light lasers). XPCS measures sample dynamics over length scales from approximately 10 to 1,000 nanometers and time scales from 10 milliseconds to 100 seconds. In this experiment we will measure and compare the length-scale dependent diffusion properties of dense and dilute suspensions of colloidal particles.