2016 Neutron Experiment descriptions:

N1: Triple-Axis Spectrometers, HFIR HB-1A & HB-3

*Spin wave and phonon dispersion in Fe-Ga solid solutions*

Fe-Ga alloys with appropriate composition and heat treatment, exhibit giant magnetostriction in a polycrystalline and ductile form. The tetragonal magnetostriction coefficient, $\lambda_{100}$, of Fe-Ga can be up to 15 times that of pure Fe. This makes these materials of tremendous scientific and technological interest for use in devices such as actuators, transducers and sensors. Elastic constant measurements show that the shear elastic constant $1/2(C_{11}-C_{12})$ 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. The triple-axis spectrometers HB-1A and HB-3 will be used to measure both phonon and spin waves of two compositions of Fe-Ga alloys.

N2: Powder Diffractometer, HFIR HB-2A

*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 HB-3A

*Structure and lithium-ion motion in the triphylite LiFePO₄ studied by single crystal diffraction*

Triphylite, Li(Fe,Mn)PO₄, 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: WAND powder/single-crystal diffractometer, HFIR HB-2C WAND**

*Diffuse magnetic scattering in Ho$_2$PdSi$_3*

When a neutron beam is diffracted by a sample without translation symmetry the resulting diffraction pattern still carries information. For instance scattering form a liquid will have a maximum in intensity on the average distance between two particles. In solids, regular stacking faults, intercalated atoms or defects can result in diffuse scattering and the analysis of these scattering patterns is important for understanding the real structure. Diffuse scattering in magnetism can occur in systems where the magnetic exchange interactions allow only degenerate ground states, or where the minimization of energy cannot be achieved for all related magnetic moments. Still, the diffuse scattering can be analyzed to understand the underlying exchange interactions. In this experiment we will investigate the diffuse scattering pattern of a geometrically frustrated rare-earth intermetallic Ho$_2$PdSi$_3$. From the diffuse scattering pattern, the spin-spin correlation function is deduced using a reverse Monte-Carlo approach. The spin-spin correlation function is then used to determine the ordered magnetic ground state. Results between simulation and magnetic ground state are used to evaluate the advantages and limitations of this technique. The experiment will include the careful hands-on set-up of the sample at the experiment using a neutron camera and goniometer, and the data treatment for the Monte-Carlo simulation.

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**N5: Neutron Imaging Station, HFIR CG-1D**

*Neutron tomographic investigation of sphere packing*

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. Additionally, x-rays have limited penetration depth for high atomic number materials. Neutrons, on the other hand, interact with nuclei and their scattering power does not vary in any regular way with atomic number, and can penetrate the bulk samples. 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. Neutron tomography is a unique tool where we can glimpse in the bulk of the assembly of monodispersive precise spherical particles made out of steel. It is of interest to do systematic investigation of the sphere packing and possible demonstration of the order-disorder transition from the random close packing to the crystalline close packing. We address the following questions: What are the highest packing densities dynamically reachable by shaken sphere packing? Does spontaneous crystallization occur? If so, what are the dynamical regimes facilitating this?
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$_2$O 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$_2$O and D$_2$O), 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$_2$O/D$_2$O ratios (empty pores, i.e., full neutron contrast), pores filled with 71% H$_2$O + 29% D$_2$O (intermediate neutron contrast) and 42%H$_2$O + 58%D$_2$O (zero-average contrast)) will be measured to demonstrate the power of contrast matching SANS technique.

**N7: NOMAD Nanoscale-Ordered Materials Diffractometer, SNS BL-1B**

*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 BaTi$_2$O$_5$. We will introduce real-space fitting using the ‘small-box’ refinement program PDFgui,
modeling the PDF of diamond, crystalline SnO₂, and SnO₂ nanoparticles. We will also introduce the levitation sample environments at NOMAD for container-less and high temperature neutron scattering, performing a laboratory experiment with a melt.

If the students would like to analyze NOMAD data on their own samples (~100 mg minimum size needed), that will be possible during the neutron school session provided the students use the mail-in proposal program by July 17st (https://neutrons.ornl.gov/nomad/mail-in). They should specify in the proposal that this is related to the 2016 NXS. Once the proposal is submitted the beamline team will be in touch to work out the logistics.

**N8: BASIS Backscattering, SNS BL-2**  
*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 [H₂NC(dma)₂][BETI].

**N9: Inelastic Neutron Spectroscopy - INS (VISION), SNS BL-16B**  
*High-resolution vibrational spectroscopy with neutrons*  

The spectroscopic technique implemented at the VISION beam line will be discussed and related to other neutron scattering methods and to Raman- and IR- spectroscopy, the experimental procedures at VISION will be introduced. We will prepare two samples for use at VISION - Zirconium hydride (ZrH₂) and Toluene. Vibrational data will be collected at low temperature (5K). The raw data will be reduced and normalized with respect to the incident beam spectrum with python based script running in the Mantid framework. The resulting energy transfer spectra will be compared with Raman and/or IR data and data from BL18 (ARCS) if time permits. The spectra will also be compared to theoretical spectra obtained with CASTEP (first-principles quantum mechanical calculations based on plane-wave basis sets and pseudopotential). The expected neutron data can be predicted based on CASTEP results using the a-Climax software.
N10: Magnetism Reflectometer, SNS BL-4A

Revealing magnetism in thin films of normally non-magnetic materials

Understanding the magnetic properties of complex materials near surfaces and interfaces critically important 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 study interfacial magnetism in a LaMnO$_3$-thin film epitaxially grown on a 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 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.

N11: 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.

N12: VULCAN Engineering Materials Diffractometer, SNS BL-7

In-situ neutron diffraction measurement of intergranular strain evolution in 316 stainless steel under uniaxial loading

Anisotropic materials such as stainless steels will develop strong intergranular strains in the regime of plastic deformation. Neutron diffraction allows strain/stress measurement at depth by its high penetration through most engineering materials. The lattice strains of different lattice plane can be calculated by Bragg peak shift with respect to zero strain/stress a reference. At the Spallation Neutron Source, VULCAN can probe changes of lattice strain of all possible $hkl$ directions under in-situ loading. In this experiment,
cubic fcc stainless steel dog-bone sample of 6 mm in diameter will be applied tensile loading continuously up to 5% engineering strain by using the VULCAN MTS load-frame, in the meantime neutron diffraction pattern of the steel sample will be collected. The neutron data will be separated and reduced based on the load intervals. Single peak refinement will be used for analyzing the intergranular strains of (111), (200), (220) and (311) lattice planes in the material under uniaxial loading. Through this practice, students will learn in-situ loading neutron diffraction measurement set-up at VULCAN; lattice strain data calculation from diffraction pattern using VDRIVE software, and understand the nature of intergranular strain evolution of material under loading.

N13: POWGEN Powder Diffractometer, SNS BL-11A

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). 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 LaB₆) to introduce TOF refinement concept.
- Sample 2: Quantitative phase analysis (NIST standard 674b: a mixture of ZnO, TiO₂, Cr₂O₃ and CeO₂).
- Sample 3: For those who want to refine a more complex structure, we will look at several models to determine the true crystal structure of Ba₂CuWO₆, which shows a Jahn-Teller distortion.
- Sample 4: Finally, those who get through the first three examples will be able to learn how to do sequential refinement for temperature scans of ZrW₂O₈.

N14: Wide-Angular Range Chopper Spectrometer (ARCS), SNS BL-18

Dynamics of metal hydride systems: Harmonic oscillators and beyond

The hydrogen in zirconium hydride (ZrH₂) sits at the interstitial positions between the zirconium. In 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 ZrH₂ 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,ω) 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 samples or experimental conditions (temperature, incident energy) will be measured to highlight differences in the energy spectra.
N15: TOPAZ Single-crystal Diffractometer, SNS BL-12

High-resolution single crystal structure analysis from 3-D mapping of reciprocal space using TOF Laue diffraction

We will practice the experimental setup, data collection, data reduction procedures and perform a structure refinement of a high-resolution single crystal data set of scolecite measured on TOPAZ using neutron wavelength-resolved TOF Laue technique. Scolecite (CaAl$_2$Si$_3$O$_{10}$·3H$_2$O) is the calcium member of the natrolite family within the zeolite group. The cation interaction with the framework oxygen bonding plays an important role in fine tuning the adsorption and electrostatic properties of the porous zeolite channels, which is fundamental for applications in separation science and energy storage materials. Single crystal data collection strategy will be optimized with the locally developed CrystalPlan program; peak integration will be performed in 3D Q-space (reciprocal space) in Mantid. Data reduction including neutron TOF spectrum, detector efficiency, and absorption corrections will be carried out with the ANVRED3 program. The structure will be refined using GSAS. The option to refine the neutron structure in SHELX 2014 will also be explored.