SENSE Workshop Abstracts
September 24-26, 2003
Turnbull Conference Center, Florida State University
Tallahassee, Florida

Sponsors

National Science Foundation
University of Tennessee / Joint Institute for Neutron Sciences
Florida State University
Oak Ridge National Laboratory / Spallation Neutron Source
Oak Ridge National Laboratory / Center for Nanophase Materials Sciences
Oak Ridge Associated Universities

Nanomagnetism and Neutron Scattering

Ivan K. Schuller, Physics Department, UCSD, La Jolla, CA. 92093-0319

Magnetism at short length scales is one of the interesting and most active areas in solid state physics and materials science. Modern thin film and lithography techniques allow preparation of artificially structured magnetic materials that have dimensions comparable to interesting magnetic sizes such as dipolar, exchange, spin diffusion, domain wall, etc. length scales. I will describe several examples, from our work, where neutron scattering has been particularly useful in settling key issues in nanostructured magnetism. This includes interfacial magnetic and structural roughness and interdiffusion, coupling in magnetic multilayers and reversal mechanisms in exchange biased systems. I will also describe possible new directions in which neutron scattering could provide answers to key issues in the field.
Work supported by DOE and done in collaboration with J.W. Cable, M.R. Khan, G.P. Felcher, M. J. Pechan, J.F. Ankner, D. M. Kelly, C.F. Majkrzak, M.R. Fitzsimmons, P. Yashar, C. Leighton, J. Nogues, J.A. Dura, A. Hoffmann, H. Fritzsche, S. Kim, S.G.E. te Velthuis.

Materials Science and Engineering Studies using Neutron Diffraction

D. W. Brown, M. A. M. Bourke, B. Clausen, S.C. Vogel., Los Alamos National Laboratory

In the past five years, several neutron diffractometers have been designed and built around the globe specifically for engineering neutron diffraction measurements. While the neutronics of purpose built engineering diffractometers has not greatly changed from the workhorse diffractometers that preceded them, major advances have been achieved in the available sample environment. Specifically, the newly commissioned SMARTS diffractometer at LANSCE has the ability to carry out measurements on very large samples with spatial resolution of 1 mm or better. In addition, in-situ studies may be completed at loads up to 250 kN and temperatures between 77 and 2200 K, greatly expanding the existing limits to enable studies of ceramics, superalloys and cermets.   Recent developments in high-energy synchrotron x-ray production have increased the popularity of SXRD as a tool for material science and engineering. The two techniques should not be considered in competition, but rather, complementary. In the future, SXRD will continue to provide the greatest incident flux, i.e. shortest count times. Conversely, neutron diffraction will offer superior bulk averaging and handle special environments better. However, the next generation engineering diffractometer, VULCAN, will, with the expected increased in neutron flux of the SNS and extrapolated improvement in sample environment, close the gap between the two techniques and further expand the realm of material science studies with neutron diffraction.

Three-Dimensional Neutron Microscopy for Structural Dynamics Investigations*

B. C. Larson, Condensed Matter Sciences Division, ORNL

Neutrons provide unique probes of the structure and structural dynamics of materials. In addition to their lattice dynamics applications, the penetration power of neutrons provides the capability for nondestructive, spatially-resolved investigations of the structure and structural evolution in the interior of bulk materials. The relatively low brilliance of neutron sources and the difficulty focusing neutrons have limited their use for spatially-resolved measurements, however. Progress toward the development of polychromatic, 3D neutron structural microscopy with ~100 μm^3 spatial resolution will be discussed along with scientific opportunities and the experimental/detector requirements. Possible neutron microscope geometries for reactor and spallation sources will be outlined using Kirkpatrick-Baez (KB) focusing mirrors, pinhole-camera depth resolution, and step-scanning sample translations to extract white-beam "Laue" diffraction patterns from ~$(100\ \mu m)^3$ volumes. The microscopy technique is applicable to single crystals, coarse-grained polycrystalline materials, composites, functionally graded materials, and to elastically and plastically deformed materials. Non-focused, depth-resolved demonstration experiments (performed recently on the SCD beamline at the IPNS) on a deformed single-crystal Cu and a randomly oriented stack of Cu and Nb single crystals plates will be described; progress and techniques for focusing neutron beams to < 100 µm diameters with $<0.3^o$ divergence will be discussed.

*Research performed under ORNL LDRD funding in collaboration with J.Z. Tischler, G.E. Ice, J. D. Budai, S. Spooner, C.R. Hubbard (ORNL), and A.J. Schultz (ANL).

Dynamic Structure of Membranes: The Concerted Use of Bilayer Diffraction and Molecular Dynamics Simulations

Stephen H. White, Department of Physiology and Biophysics, University of California at Irvine, Irvine, CA 92697

Combined x-ray and neutron diffraction measurements on an absolute scattering-length-density scale can be used to obtain the transbilayer probability distribution functions of the principal lipid structural groups of lipid bilayers, such as the phosphate, choline, and carbonyls. But these distributions provide only a 1-D representation of bilayer structure, because they are time-averaged projections of the structural groups' trajectories onto the bilayer normal. 3-D representations of bilayer structure, on the other hand, can be obtained from molecular dynamics simulations. But these representations are tied only indirectly to experimental data. We are therefore developing methods for combining diffraction measurements with MD simulations in order to obtain experimentally validated dynamic 3-D structures of bilayer membranes. We expect that this general approach will ultimately allow MD simulations to become a significant refinement tool for determining the structure of lipid bilayer membranes and structural ensembles of peptides in lipid membranes. The concerted use of neutron diffraction and MD simulations is central part of the Cold Neutrons for Biology and Technology (CNBT) partnership underway at the NIST Center for Neutron Research. Research supported in part by the NIH (RR14812, GM68002, GM46823).

Synchrotron X-ray Studies of Liquid Surfaces

P.S. Pershan, Div. of Eng. and Applied Sciences and Dept. of Phys., Harvard Univ., Cambridge, MA 02138

The complementary properties of X-ray and neutron scattering often allow experiments that together provide much more information about liquid surfaces than would be possible with only one or the other types of scattering. In this talk we will review the special features of x-ray scattering applied to liquid surfaces. Examples will be selected from our recent results on both liquid metal surfaces and on thin wetting liquids.

Support is acknowledged from NSF-DMR-0124936 and DE-FG02-88-ER45379.In addition experiments done at NSLS acknowledge support from DE-AC02-98CH10886 and ChemMatCARS at the APS is principally supported by the NSF/DOE under grant CHE0087817 and by the Illinois Board of Higher Education.

Environments for Biological Studies

John Katsaras, National Research Council Canada, Chalk River, Canada

Of central relevance to today's seminar is the fact that unlike x-rays, neutrons are neutral particles that interact weakly with most matter. Although the study of materials under difficult environments (e.g., high magnetic fields, temperatures and pressure) is not trivial under any circumstance, it is made simpler by the fact that neutrons interact weakly (thus non-destructively) with many common materials and in particular, aluminium (Al). The incoherent and absorption neutron cross-sections for aluminium are such that Al, for the most part, is considered to be practically transparent to neutrons. Moreover, the relatively inexpensive cost of Al and its alloys, together with its useful physical characteristics make it the material of choice when it comes to constructing sample environments at neutron scattering facilities worldwide. Today's seminar will present a series of examples of biomimetic materials studied in the presence of a magnetic field, hydrostatic pressure, and a 100% relative humidity environment capable of fully hydrating, from water vapor, samples aligned on a solid support. Although similar sample environments have been developed for use with x-rays, the design and construction costs for similar neutron cells are much more favorable.

Neutron and X-Ray Studies of Surfactant Monolayers at Air-Water Interface

J. P. Majewski, Los Alamos Neutron Scattering Center (LANSCE-12), H805, Los Alamos National Laboratory, Los Alamos, NM 87545, US

In recent years, several surface-sensitive scattering techniques have been developed for probing the surface structure of materials. These include neutron and x-ray reflectometry and x-ray grazing incidence diffraction. As will be shown, these techniques can be successfully implemented for studying structures of organic, ultra-thin, molecular arrays of surfactants on water surface. In the case of monolayers composed of amphiphilic molecules standard diffraction techniques are not applicable because of the low, scattered signal rates and substantial background contribution. Nonetheless, combination of x-ray and neutron surface scattering methods allows one to deduce film morphology and structure. I will illustrate the use of the x-ray and neutron surface scattering methods to characterize the structures of a several types of amphiphilic monolayers at the air–liquid interface and their interactions with polymers present in the subphase. The properties of these soft-condensed, ultra-thin layers are of general interest to a wide scientific audience working in the field of chemistry and physical chemistry since they are relevant to such important areas as bio-mineralization, biosensors, advanced drug delivery systems, polymer-membrane interactions, and can serve as useful models for elucidating structure and function of biological membranes.

Support is acknowledged from DOE contract W7405-ENG-36 and the DOE Office of Basic Energy Sciences.

Aerosol Dynamics: What can we learn from SANS?

Barbara Wyslouzil, Worcester Polytechnic

Nanometer-sized particles form by nucleation and condensation in many environmental and industrial settings. Fine refractory fumes form in metallurgical and combustion processes, while H_2SO_4 – H_2O water nanodroplets form in exhaust stacks and volcanic eruptions. In many high speed or reactive flows it is difficult, if not impossible, to extract representative aerosol samples and in situ methods are required to follow aerosol evolution. This talk will summarize our efforts to date to develop SANS as a quantitative tool for studying aerosol formation and growth rates in situ.

Present Status of Neutron Scattering Environments at High Magnetic Fields and Low Temperatures

Michael Meissner, Hahn-Meitner-Institut Berlin, Germany

During the last 10 years the Berlin Neutron Scattering Center (BENSC) has concentrated on the development of special sample environments for neutron scattering experiments at extreme conditions. Today, BENSC offers a variety of instrumentation up to the highest vertical magnetic fields in the world available for neutron scattering: 17.5 T at temperatures 1.5 K to 80 K. For vertical fields up to 15 T and horizontal fields up to 6 T measurements can be performed in a wide temperature range 30 mK to 330K. The basic features of low temperature systems and cryomagnets most frequently used at BENSC will be discussed. In a recent proposal to the German Ministry of Research the HMI has designed a resistive 40 MW high magnetic field facility which will serve a devoted horizontal 40 T magnet instrumentation for neutron scattering. In short, the so-called N40T Facility will be presented as a future expansion of the high magnetic field activities at BENSC.

In-situ, High Temperature Study of Phase Transformations in Ceramics

Professor Waltraud M. Kriven, University of Illinois at Urbana-Champaign, Materials Science and Engineering Department, 1304 W. Green St., Urbana, IL, 61801, USA

High temperature phase transformations can be studied using a quadrupole furnace that has been developed in our laboratory. It allows in-situ, in air experiments up to approximately 2000 °C. The furnace consists of four halogen infrared reflector lamps, where the filaments are placed at one of the elliptical foci of the lamps. They are arranged in a water-cooled brass housing, forming a “hot spot”, where the other common elliptical foci of all four lamps overlap. The geometry of the furnace allows x-ray diffraction experiments to be carried out at an angular range of 0-35 degree 2ϑ for cylindrical specimens in Debye-Scherrer geometry or in a specially-adapted, specimen holder in Bragg-Brentano geometry. Temperature calibration of the system can be achieved by dip coating the specimens with a suitable reference material, where the lattice parameter development is known and which has similar absorption properties to the material under study. The lattice parameters and thermal expansion coefficients are determined from Rietveld analysis of polycrystalline powder diffraction data collected by synchrotron X-rays. The current status of known phase transformations in oxide ceramics and their potential applications in structural ceramics are presented.

New Funding Opportunities for Mid-Scale Instrumentation and International Cooperation

Thomas A. Weber, Division Director, Materials Research, National Science Foundation

During FY 2003 the Division of Materials Research (DMR) announced new funding opportunities for US scientists to establish collaborations with scientists in the Americas and Europe. In FY 2004, DMR is establishing a new program within the Instrumentation Program for mid-scale instruments such as synchrotron and neutron beam lines, which will fill the gap between the $2 million maximum allowed by the Major Research Instrumentation Program and projects funded by the Major Research Equipment Program. This talk will discuss these new opportunities.

What's New in DOE's Neutron Scattering Program

Helen Kerch, U.S. Department of Energy

The neutron scattering program in Basic Energy Sciences supports research in condensed matter and materials physics aimed at achieving a fundamental understanding of the atomic, electronic, and magnetic structure of materials, the interrelationship of those structures, and how that relates to physical properties. The recent infrastructure and instrumentation enhancements at both HFIR and LANSCE, and the planned commissioning of the SNS in 2006 afford unprecedented research opportunities for the US neutron scattering community. This talk will detail recent investments in both instrumentation and research, and will outline future research directions and opportunities in neutron scattering research.

The National Science Foundation International Materials Institutes [IMI] Advanced Neutron Scattering NetWork for Education and Research [ANSWER]: With a Focus on Mechanical Behavior of Materials

Peter K. Liaw[1], Hahn Choo[1], Raymond A. Buchanan[1], Camden R. Hubbard[2], and Xun-Li Wang[2]

1. Department of Materials Science and Engineering, 427-B Dougherty Engineering Building, The University of Tennessee, Knoxville, TN 37996-2200

2. Oak Ridge National Laboratory, Oak Ridge, TN 37830

Neutron scattering is one of the most powerful characterization techniques available for materials research, and its proven capabilities and anticipated potential justify the 1.4 billion dollar construction of the Spallation Neutron Source [SNS] at the Oak Ridge National Laboratory [ORNL] as well as a number of multi-million dollar facility upgrades at the Los Alamos Neutron Science Center [LANSCE], Intense Pulsed Neutron Source [IPNS], and High Flux Isotope Reactor [HFIR]. Moreover, the European Union [EU], Japan, China, and Australia are also currently building or designing the next-generation neutron sources. However, while the multi-billion dollar constructions are on-going all around the world, at the present time, there is no concerted effort within the US in advancing the science and education on the application of neutron scattering in materials research, specifically regarding the study of mechanical behavior of advanced materials. To be better positioned in the intense scientific competition at the international level, it is important to form a national consortium, and develop an international network with international counterparts. Therefore, a group of the most active researchers in the neutron-scattering materials research field will form an international network for research, education, and training, and provide a synergistic effort in advancing the fundamental understanding of mechanical behavior of materials at the atomic level using state-of-the-art neutron sources in the world. A five-year International Materials Institutes [IMI] Program, <u>A</u>dvanced <u>N</u>eutron <u>S</u>cattering net<u>W</u>ork for <u>E</u>ducation and <u>R</u>esearch [ANSWER], proposed by the University of Tennessee and ORNL, has been funded by the National Science Foundation to create an international neutron-scattering network for innovative, multidisciplinary materials education and research in the area of "mechanical behavior of advanced materials." The University of Tennessee, with the California Institute of Technology, Illinois Institute of Technology, Northwestern University, University of Missouri at Columbia, and University of Pennsylvania, will develop a domestic network in partnerships with the national neutron user facilities at LANSCE [Los Alamos National Laboratory], and at HFIR and SNS [Oak Ridge National Laboratory]. Furthermore, several industrial participants are committed to the strong partnership with ANSWER, which include ALCOA, Boeing Company, DANA Corporation, Federal Mogul, General Electric Company, Haynes International, and Solar Turbines, Inc. The objectives of the proposed IMI are to: [1] develop an international network of researchers and educators who are actively involved in "neutron-scattering materials research" from universities, industries, and neutron facilities worldwide; [2] facilitate the exchange of scientific information through collaborative research projects; [3] develop a world-class work force in neutron science by providing US students, scientists, and engineers opportunities to access the research programs and facilities in the US and other countries; [4] identify and lead innovative materials-research efforts; and [5] establish international Internet-based virtual institutes. We are very grateful to National Science Foundation [NSF] for the support [DMR-0231320]. Dr. Carmen Huber is the Program Director.

Enabling 21st Century Science

Zoe Bowden, ISIS, Rutherford Appleton Laboratory

At ISIS we provide a wide range of sample environments to support the experimental programme. There is a mix of standard systems providing temperatures from 2K to 2000K, pressure systems and rotation and translators stages. In addition there are more complex cryostats providing sub 1K temperatures as well as combination systems enabling, for example, pressure at low temperatures in magnetic fields. As the variety and complexity of the experiments increase, how we develop the equipment to increase the envelope of available environments is an issue for all the facilities.

In-situ X-ray Scattering Studies of Epitaxial Crystal Growth

Paul F. Miceli
Department of Physics and Astronomy, University of Missouri-Columbia, Columbia, MO 65211, USA MiceliP@missouri.edu

As an example of extreme in situ sample environment requirements, this talk will discuss our x-ray scattering studies of epitaxial crystal growth performed in UHV using synchrotron radiation. X-ray scattering provides a special advantage for film growth investigations as the technique can reveal surface morphology simultaneously with the subsurface structure. Examples to be discussed: surface diffuse scattering is used to study nucleation and growth from the submonolayer regime through thicker films where the diffuse scattering from dislocations is observed; surface roughening during film growth is quantitatively related to the effects of the kinetics at crystalline step edges; vacancies are found to incorporate at low temperature and these influence the surface morphology.
Finally, our recently completed chamber for surface x-ray scattering at the Advanced Photon Source will be described. In addition to the capabilities for film growth and analysis, the instrument permits investigations from 50K to 1500K and covers an unusually broad range in reciprocal space, which is essential for the investigation of buried defects.
Grateful acknowledgement is given to the collaborators, students and postdocs who have contributed to this work: E. H. Conrad, P. W. Stephens, C. Kim, R. Feng, C. E. Botez, W. C. Elliott, K. Li, E. Lu, A. Sahiner, and M. McManus. Research funding is acknowledged from NSF, DOE, and the U. of Missouri Research Board. Beamline funding: At the Advanced Photon Source: DOE W-31-109-Eng-38, and MUCAT through Ames Lab DOE W-7405-Eng-82. At the National Synchrotron Light Source, SUNY X3: DOE DE-FG02-86ER4523.

Accessing and Studying Metastable Materials Using Containerless Techniques

Richard Weber[1], Christopher Benmore[2], James Rix[1], Lou Santodonato[3],
Joan Siewenie[2], Jean Tangeman[1], and Kirsten Hiera[1]
[1]Containerless Research, Inc., Evanston, IL 60201
[2]Argonne National Laboratory, Argonne, IL 60439
[3]Oak Ridge National Laboratory, Oak Ridge, TN 37830

Liquids can often exist at temperatures far below the equilibrium melting point of the crystals from which they are formed. These “undercooled” liquids can display a variety of phenomena including transformation to glass, amorphous-amorphous phase changes, and crystallization to form stable and/or metastable phases. Undercooled liquids can also provide a glimpse of the metastable region between the highly ordered crystalline solid and disordered liquid states where there are many opportunities to synthesize and study new materials. This paper summarizes details of an instrument designed specifically to study deeply undercooled liquids and very hot solids in conditions that completely eliminate contact with a sample holder or mount. Samples are levitated in a stream of gas that can also be used to control the ambient $p(O_2)$, exclude oxygen, or establish reactive environments as required by the user. Samples 2-3.5 mm diameter are levitated and located in a neutron beam and heated with a laser beam to achieve temperatures limited only by the sample vapor pressure. Tests on oxide materials have demonstrated temperatures to >3000 K and undercooling by almost 1000 K. The sample temperature is monitored with an optical pyrometer and progress of the experiment is observed via a video image of the sample. We report: (i) current instrument specifications, (ii) results of studies performed at IPNS on liquid aluminate materials and solids at very high temperatures, and (iii) plans to integrate levitation instruments for studies on non-equilibrium materials at high flux beam lines such as SNS where fast measurements of structure will enable investigation of highly non-equilibrium materials under transient conditions.

References:
Novel Materials for Extreme Environments, A Vision for a Neutron Instrument for Materials Research Under Extreme Environments, C.K. Loong, Ed., IPNS 11-12 Sep., 1998.
C. Landron, L. Hennet, T.E. Jenkins, N. Greaves, J-P. Coutures, and A.K. Soper “Liquid Alumina: Detailed Atomic Coordination Determined from Neutron Diffraction Data using an Empirical Potential Surface Refinement,” Phys. Rev. Lett, 86, 4839 (2001).
J.K.R. Weber, C.J. Benmore, J.A. Tangeman, J. Siewenie and K.J. Hiera, Structure of CaO--Al_2O_3 and SrO-Al_2O_3 Liquids by Combined Levitation-Neutron Diffraction,” J. Neutron Res., in press.
Supported by DOE Grant No. DE-FG02-01ER86121, Dr. Helen Kerch, Technical Monitor

Sample Encapsulation Considerations In Designing High Temperature Neutron Diffraction Experiments

K. J. Volin, Intense Pulsed Neutron Source, Argonne National Laboratory, Argonne, IL 60439 USA

Neutron diffraction is a powerful tool for structural studies of materials in special sample environments because of the high penetrating power of neutrons compared to x-rays. This is especially true in high temperature experiments where neutrons are able to penetrate furnace heat shields, elements, and sample containers. Sample environment groups at the various neutron scattering facilities have worked hard to create ingenious schemes for heating samples to higher and higher temperatures. These have included refractory metal foil elements, halogen image furnaces, and laser heating. Sample environment furnaces, along with their control electronics, have reached high states of robustness and reliability. While the sample environment groups have had full control of the furnace design and operation, they cannot plan for every conceivable sample material or container. Furnace failures have thus become almost exclusively the result of some sort of sample container failure. These failures are typically due to sample-container incompatibility at high temperature, where chemical reactivity escalates. An overview of a well conceived approach to designing a high temperature neutron diffraction experiment, with particular attention to sample encapsulation options will be presented and discussed.

Implementing a CMMS at the IPNS for the SE Group

J.S. Fieramosca, Intense Pulsed Neutron Source, Argonne National Laboratory, Argonne, IL 60439 USA

This presentation will address the “what, why and how” of implementing a Computerized Maintenance Management System (CMMS) and a clear work management process at the IPNS for the SE group to ensure equipment readiness and reliability. Over the years the IPNS has acquired numerous sample environment laboratory equipment such as; furnaces, cryostats, closed-cycle refrigerators, electromagnets, pressure generators and cells, sample positioners and changers, water-chillers, etc.; each having been used extensively. More equipment will be commissioned soon and plans for future equipment that meet scientific and advanced SE needs are being formulated. So the daunting task for the SE group leader is to manage the present equipment while smoothly facilitating the research,

development and acquisition of newer ones all on time and on budget. The managing of present equipment lies in proper preventative maintenance resulting in increased equipment readiness and reliability. Eliminating, as much as possible, the need for emergency corrective maintenance while on the scattering instrument resulting in downtime. The implementation of a CMMS and a clear work management process are key to establishing a culture of proactivness instead of reactiveness, hopefully reducing the “fire fighting” mode. The IPNS SE group is embarking in such a direction. This talk will cover the multiple benefits from the use of a CMMS, obstacles to change, and how a comprehensive implementation plan tailored specifically for a SE group’s needs can achieve optimal overall equipment efficiency.

Do we still need Helium-Flow Cryostats?

F. THOMAS, Institut Laue-Langevin, 6, rue Jules Horowitz, BP 156, F-38042 Grenoble Cedex 9 – France

Over the last decade, a lot of progress has been made in cryocooler technologies. New machines are now cooling faster and are much more reliable. Furthermore, the use of rare-earth compounds in the second stage of the cooler allows base temperatures below 3 K and the use of pulse tube techniques reduces the vibration rate significantly. To take advantage of this progress, the ILL has built several cryogen free cryostats based on modern coolers. 4 of these cryostats will be presented. 2 are pulse tube based. The 2 others are based on a classical 10 K Gifford-McMahon cooler on which a home made Joule-Thomson stage has been added in order to reduce the base temperature below 2 K. It will be shown that these cryostats are competitive with standard helium-flow cryostats in terms of price, cooling time and/or base temperature. Finally, the conclusion will try to give an answer to the title.