

X-rays and Neutrons:
ESSENTIAL TOOLS FOR NANOSCIENCE RESEARCH

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Abstracts of Presentations

Water in Nanotubes: A New Phase of Water?

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Deep Inelastic Neutron Scattering (DINS) from Hydrogen (or other light nuclei) can be used to measure momentum distributions. The availability of sources of high energy neutrons and improvements in data fitting methods[1,2] enable us to measure the anharmonic details of the momentum distribution, and in some cases, the Born-Oppenheimer potential of the particle. Quasi-one-dimensional water encapsulated inside single-walled open-ended carbon nanotubes SWNT, here referred to as *nanotube-water*, was studied by neutron scattering at different temperatures. [3] The system is regarded as a model system for the study of the transport of water in biological pores. The earlier results revealed an anomalously soft dynamics characterized by pliable hydrogen bonds and large-amplitude motions of the hydrogen. We have used DINS to examine the momentum distribution of the protons in nanotube water. We find that the protons are delocalized to such a degree at low temperatures (4K) as to qualitatively change the nature of the hydrogen bonds, to the extent that we appear to be seeing a new phase of water, while at higher temperatures (268K) the bonds resemble that of bulk water but are much more strongly anharmonic.[4]

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Structure and Dynamics of Magnetic Nanoparticle Assemblies

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Advances in synthetic techniques have made available large quantities of monodisperse magnetic particles and molecular moieties which can be subsequently assembled into extended structures. Different aspects of the functionality of these confined magnetic entities can be addressed ranging from applications and devices, to the construction of model magnetic systems for fundamental studies. In each case, a wide range of experimental techniques must be brought to bear, including synthesis, visualization, magnetic functionality, and ultimately scattering studies of the magnetic structure and dynamics. Particularly challenging for scattering measurements is the need to determine structure on length scales which range from the unit cell, to the particle dimensions, and to the correlation scale within the assembly of nanoparticles. Similarly, studies of the dynamics must be carried out on energy scales matched to these very different length scales, spanning intraparticle spin waves, particle reorientation and precession, and the collective excitations of the assemblage. New instrumentation is required to meet these challenges.

The Grand Challenges in Nanomagnetism*

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Synchrotron x-ray facilities [1] and neutron scattering facilities [2] provide boundless opportunities to explore new nanomagnetic materials and phenomena. This talk provides an overview of the grand challenges in nanomagnetism. Examples of the use of x-rays and neutrons to explore and understand exchange coupling phenomena across interfaces of magnetic films, multilayers and laterally confined structures are provided.[3-6] The fundamental scientific problems involve the need to understand the nature of confined spin states, ultra-fast spin dynamics, and novel spin transport [7]. *This work was supported by DOE-BES, under contract W-31-109-ENG-38.

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A time lens for high resolution neutron time of flight spectrometers

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We are currently studying the imaging effects of temporal neutron lenses. Our lens is based on a traveling non-homogeneous magnetic field with parabolic shape which is analytically determined and numerically verified by appropriate calculations. The lens is intended to replace the traditional chopper system used for time of flight instrumentation to enhance intensity and/or time resolution significantly. A first estimation of the gain factor points to a value of 5.5 with a time resolution of $5\mu\text{s}$ which is hardly achieved by traditional systems. The proposed experimental setup seems to be feasible and may open new fields of applications regarding to high resolution spectrometry. The contribution starts with the basic ideas and methods applied. Some of these ideas are already published but have never been aimed to study a time lens for neutron TOF spectrometers. Further we will discuss possible design parameters and miscellaneous subjects related to time lens focusing such as aberration. We conclude with results from numerical calculations that for example provided us a view of the evolution of the phase space element during propagation through the optical system.

Atomic Imaging by Fourier Inversion of X-ray Standing Wave Data

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The need to understand the structure-property-processing relationships for various interface-based nanoscale materials systems strongly motivates the development of new in situ experimental techniques for directly observing structures at the atomic-scale. The relevant processing environments may vary from vacuum, as in the case of oxide/semiconductor MBE, to liquid, as in the case of biomolecular adsorption. While scanning probe microscopy-based techniques partially fulfill this demand by giving a top-view of a given structure, it is important to fill in the entire top-to-bottom in-depth view with quantitative, element-specific, structural information for describing the atomic arrangements, including for example, the electronic and magnetic states of atoms within the arrangement. This is a challenge for developers of x-ray scattering and spectroscopy techniques.

A crucial problem facing scattering measurements is in transforming the reciprocal space data into real-space images. This is primarily due to the “phase-problem”, which in a growing number of cases can be circumvented by using a reflection generated x-ray standing wave (XSW) as a spectroscopic probe. The XSW technique measures the amplitudes and phases for element-specific atomic distributions. As recently shown, the summation of these Fourier components produces a model-independent 3D atomic map of each XRF specified atom with a λ/Q_{\max} spatial resolution. While XSW atomic imaging has thus far have been restricted to XRF detectable atoms for element sensitivity, it should be extended to other spectroscopy channels, such as, XPS for chemical sensitivity and circular magnetic dichroism for magnetic sensitivity.

In addition to XSW measurements, other pieces of the puzzle can be filled in by applying other scattering and spectroscopy techniques, such as SEXAFS for determining bond lengths, XPS for determining the chemical bonding states, and XRR for studying molecular packing. For reliability it is important in many cases to make as many of these measurements on the same sample, in the same environment, in a reasonably short time-frame. This calls for multi-tasking experimental stations that combine x-ray scattering and spectroscopy into one integrated system.

Important areas to be studied by this combined scattering and spectroscopy approach include silicon-based molecular electronics, biomolecular attachment to functionalized surfaces, multifunctional oxide epitaxy and supported metal-oxide catalysts.

The Nanostructure Problem

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Structure is fundamental to materials properties. Revolutions in materials science and molecular biology followed rapidly from the ability to obtain atomic scale structure using crystallography. The structure of periodically arrayed nanoparticles can be solved, as in the case of protein crystallography; however, crystallography fails for discrete nanoparticles and disordered ensembles of particles.

By definition, nanoparticles are not periodically long-range ordered. As a result, sharp Bragg-peaks broaden into diffuse features that often overlap each other resulting in a loss of information in the data. On the other hand, the structure of the nanoparticles in general is complex and requires many unique atomic positions to be specified. This means that robust structural solutions of complex nanoparticles are difficult or impossible. This is the nanostructure problem.

Some progress has been made using total scattering methods such as the atomic pair distribution function (PDF) method which yields weighted distributions of inter-atomic distances. In this case, structure refinement of reasonable initial models is often possible; however, ab-initio structure solution of an unknown structure has not been demonstrated. Other techniques, such as EXAFS, NMR, TEM and other imaging techniques, are also brought to bear and provide complimentary information. However, in general, using data from any single technique, the nanostructure problem is not well conditioned. There is not a good balance between information content in the data and degrees of freedom in the model and resulting models are not unique (often far from it). A robust solution to the nanostructure problem requires that it is recast in a well conditioned way such that the data constraints are greater than the degrees of freedom in the model. This will likely require a concerted effort to combine self-consistently data from multiple complementary techniques with a software development effort to handle the diverse data and combine modeling and regression in a self-consistent optimization. Research on this topic is underway at MSU funded by a DOE-BES II grant.

What is needed is:

1. Beamlines in close proximity with a flexible access structure that allows the complementary data to be collected quickly without multiple trips and proposals
 - a PDF
 - b EXAFS
 - c NMR
 - d Anomalous scattering
 - e High intensity neutron powder diffraction
 - f Microbeam diffraction
 - g TEM/Electron diffraction
 - h Imaging techniques (Scanning probe, diffraction/nanoprobe imaging)
2. Data analysis software development in cooperation between theorists and experimentalists
3. Computing software and hardware support that is x-ray/neutron facility independent but adequately funded.

Synthesis and Characterization of Novel Polymer Architectures

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One of the greatest challenges facing chemistry, physics, and materials science today is the controlled synthesis of mesoscopic materials (i.e., materials with characteristic dimensions of 1-100 nm) with well-defined structures and properties, and the assembly of these materials into macroscopic functional devices. One approach that has generated significant interest has been the “bottom-up” approach, used by nature, which starts with single molecules of controlled size, shape, and functionality (such as amino acids) and assembles them through covalent and/or noncovalent interactions into functional supramolecular assemblies (such as enzymes). The ultimate goal is to develop reproducible synthetic methods, which controls interfacial interactions, defects, composition, and structure, to produce materials that possess the desired function or property via an understanding of the nanoscale physics and chemistry. To achieve this goal, a better understanding is needed of the thermodynamic and kinetic processes (such as self-assembly) at the nanoscale and a better understanding is needed of structure-property relationships at all length and time scales. Characterization tools are also needed to observe and characterize events at the nanoscale in real time and under a variety of environments (such as temperature, pressure, and shear). Since small angle scattering techniques using either X-rays or neutrons provide statistically averaged structural information on length scales of 1-100 nm, these tools are ideally suited to study structure and dynamics of hard and soft materials (including polymers, copolymers, biomaterials, composites, micelles, colloids, mesoporous metal oxides, magnetic materials, metallic alloys, etc.) and interfaces. The high penetrating power of neutrons also allows studies to be conducted in concentrated solutions and in the bulk. The large difference in scattering length of neutrons for hydrogen and deuterium provides a unique opportunity to manipulate the contrast without changing the structure. Thus, neutrons are ideally suited to study the molecular conformation and interactions of polymer chains in the bulk, in blends, in dilute solution, and at interfaces under a variety of conditions such as shear or deformation at controlled temperatures and pressures. Nevertheless, new developments are still needed in developing sample environments to study smaller samples at finer spatial resolution (1 nm), and to make real-time measurements. New methods are also needed to obtain 3-D images of complex nanostructured materials to truly understand structure-properties relationships at the nanoscale. This presentation will highlight some of the synthetic advances made in polymer science, the complex molecular architectures that can be made, and the opportunities to use X-rays and neutrons to provide new insight into the rule governing nanoscale chemistry and physics.

Impact of a Future Energy Recovery LINAC X-ray Source on Nanoscale Science

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The performance characteristics of modern 3rd generation synchrotron x-ray sources are approaching fundamental physical limits set by the equilibrium dynamics of particle storage rings. Even in the absence of interactions, the orbit of a single charged particle varies with the energy of the particle as it emits photons and then is re-accelerated by the RF cavities. Over many revolutions (or equivalently, in an ensemble of many particles), a finite phase space distribution that is much broader in the plane of the storage ring than perpendicular to the plane develops. This fundamental effect does not, however, apply to the electron beams in LINACs because the time-scale required to achieve the equilibrium distribution is orders of magnitude longer than the transit time. In an Energy Recovery LINAC (ERL), each particle is accelerated by the RF cavities in a LINAC, transits a brilliance preserving loop instrumented with insertion devices, and is then de-accelerated by the same LINAC. Each particle suffers only minute changes in energy. The de-acceleration process returns the particle's energy to the RF cavity with essentially zero loss. The ERL is an energy storage rather than a particle storage device. Without energy recovery, a LINAC x-ray source would be prohibitively expensive to operate.

Detailed designs developed by Cornell University's ERL prototype project demonstrate that an ERL would be a fully diffraction-limited x-ray source for photon energies less than or equal to 12.6 KeV and with a (circular) emittance of 0.008 nm-radians. These parameters imply 1-10 nm circular beam waists for focused x-ray beams but with intensities comparable to current 3rd generation synchrotron beam lines. Such an x-ray source would enable the application of essentially all existing x-ray characterization techniques to individual nanoparticles (e.g., 20-50 nm). For example, fluorescence detection and spectroscopy of individual impurity atoms would become possible. A variety of ultra-fast (sub-picosecond) and coherent x-ray imaging techniques also become possible.

* on behalf of the ERL team of I.V. Bazarov, S.Belomestnykh, D. Bilderback, J.D. Brock, E. Fontes, K.D. Finkelstein, S.M. Gruner, G. Hoffstaetter, A.Kazimirov, M. Liepe, Y.Lin, H.S. Padamsee, D.Sagan, V.Shemelin, Q. Shen, K. Smolenski, C. Sinclair, R. Talman, M. Tigner, V.Veshcherevich, Cornell University; and L. Merminga & G. Krafft of J. Lab

Relevance of Strong Electronic Correlations in Bulk and Nano Systems

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The study of Strongly Correlated Electronic (SCE) systems is among the most popular areas of investigations in condensed matter physics. The reason is that these materials have provided conceptually unexpected phenomena such as high temperature superconductivity and colossal magnetoresistance. In recent years, a large effort both in theory and experiments has shown that materials of this variety tend to have self-organization effects at the nanoscale, often crucial to microscopically understand the bulk behavior they present. Neutron and X-ray studies have been fundamental to reveal these nontrivial electronic structures, unveiling a complex behavior similar to that observed in soft matter. Transferring this knowledge to nanosystems, such as conductors involving small molecules where charging effects are important, or quantum dots with the Kondo effect, or long molecules with polaronic formation, is a frontier that it is barely explored.

I will argue that a research effort focused on the analysis of similarities bulk \leftrightarrow nano involving SCE materials is very important, particularly for the plethora of new concepts that may emerge, and the natural physical location for such efforts is at DOE laboratories where large scattering facilities are close to nanocenters.

Atomic Structure of Materials with Nano-Scale Coherence

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Nano-scale materials, such as nano-particles, are usually characterized by their size, assuming that their atomic structure is the same as in the bulk. However, for complex materials which are increasingly gaining attention as functional materials this assumption is often false. Confinement into a nano-scale can make competing phases more stable, and nano-particles can have metastable or unstable atomic structure which can produce unusual and attractive properties. Conventional crystallographic methods of structural characterization are powerless for such materials, and local methods emerge as more effective alternatives. We describe one of these methods, the atomic pair-density function (PDF) analysis of static as well as dynamic structure at a nano-scale, using pulsed neutrons and synchrotron x-rays as a probe.

Can Kinoform Hard X-ray optics produce sub 10nm beams?

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Based on our experience with, and understanding of, the refractive and diffractive properties kinoform lenses, we extrapolate beyond current practical limitations and try to understand possible limiting behavior of this type of focusing optic. Kinoform Fresnel lenses have lower absorption and larger apertures, than the equivalent refractive-limit lens. The lower absorption and larger apertures leads to optics with smaller focused spots, but this is obtained at the expense of choosing to operate these lenses at or near a fixed, design wavelength. Using micro-fabrication techniques, we have manufactured low loss kinoform Fresnel lenses for hard X-rays at energies of 11.3keV, and energies as high as 30keV. We present measurements and simulations of the effectiveness of kinoform lenses and discuss the possibility of sub 10nm focused beams. Research carried out at the National Synchrotron Light Source under DOE Contract No. DE-AC02-98CH10886.

Characterization of Soft Nanostructured Materials

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The control of self-assembly, the process by which order arises from a collection of disordered molecules or objects, to achieve functional nanoscale materials that could provide the basis of nanoscale devices (e.g., switches, gates or memory elements) remains a significant challenge and constitutes a major goal in the field of nanoscience. Unlike top-down fabrication approaches, self-assembly offers the potential of preparing nanoscale materials in an inexpensive, low energy consumption, “multiple copy” fashion. In addition, the self-assembly of molecules provides a means by which to pattern functional nanostructures with feature sizes not achievable *via* lithographic approaches and to construct patterned three dimensional structures. Although a considerable amount of effort has been directed towards the study of self-assembly, few guiding principles for the *de novo* design of functional self-assembled nanostructures have emerged. The lack of a directed/rational approach to achieving functional, self-assembled materials is the result of both the complexity of the objects and forces that control the self-assembly process and the limitations of the techniques now available for the characterization of self-assembled soft materials. Thus, improving the characterization of static, dynamic, and *in-situ* self-assembly remains a current and significant challenge in nanoscience.

Although the development of scanning probe / microscopy techniques has yielded notable advances in nanoscale characterization of materials, the study of soft materials such as complex fluids with these techniques is not possible without altering or perturbing the samples. X-ray and neutron scattering techniques therefore remain the preferred approach to study such systems, since they can probe structure on the mesoscopic (nanometer to micron) length scale and can probe bulk phase materials in a non-perturbing, non-destructive way under realistic sample environments (e.g., high levels of hydration). X-ray and neutron scattering techniques such as SAXS, GISAXS, USAXS, and reflectivity can be used to determine structure under controlled environmental conditions (e.g., under the influence of applied electric or magnetic fields) and to monitor the *in-situ* self-assembly process. Neutrons offer the additional advantage of characterizing low-Z element compositions *via* the use of isotopic substitution and contrast matching. Current approaches, however, provide low to medium resolution structure determination in reciprocal space. Improvements are needed to further increase the structural resolution and to be able to achieve this in one, two and three dimensions. A major advancement would be the development of experimental approaches to provide real-space imaging, such as those obtained by scanning probe methods. For example, 3-D tomography would yield high resolution images of soft materials and would represent a significant advance over current capabilities, bridging the gap between electron microscopy and optical microscopy. Lastly, due to the diversity of interactions among molecules, a true understanding of self-assembly will require probes that are sensitive to both local and extended structure over a wide range of spatial and temporal dimensions. Such information may only be provided by coupling scattering techniques with standard spectroscopic methods.

Nano-structured magnetic materials: novel physics and emerging technologies

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Magnetic storage has played a key role in audio, video and computer development since its invention more than 100 years ago. In 1956 IBM built the first magnetic hard disk drive featuring a total storage capacity of 5 Mbytes at a recording density of 2 kbits/in². Since then the density of bits stored on the surface of a disk has increased to 100 Gbits/in² with data rates approaching a GHz. At such densities, the bits must be positioned on the disk with nanometer resolution. In the magnetic recording industry (and similarly in the semiconductor industry) increased capacity and performance was historically achieved by scaling the critical device dimensions. While this approach has been the basis for much of the 50-million-fold increase in disk drive capacity, it appears to be reaching limits. These limits arise from difficulties in lithography and, more fundamentally, because of materials limitations. In current devices, most of the critical dimensions are now in the nanometer range where two issues become critical. The first is that surface and interface effects dominate over bulk material properties. The second is the thermodynamic properties of small structures and the increasing importance of thermal energy. The superparamagnetic limit in recording media is a prime example of the latter where the magnetic energy of nanoparticles becomes comparable with $k_B T$.

Continued growth of storage densities will require the development and characterization of new nano-structured materials and architectures that control magnetic interaction and correlations on the nanometer scale and exploit the unique physics that emerge at the nanoscale. In general much of the new physics comes from combining disparate materials (ferromagnets, antiferromagnets, paramagnets, insulators etc.) on nanometer scales. The magnet response of the composite often provides exciting new scientific questions as well as the impetus for emerging technologies. The classic example is giant magnetoresistance (GMR) originally discovered in Fe/Cr superlattices where the measured magnetoresistance was two orders of magnitude greater than that expected for Fe films. While GMR is currently used in all hard drives and has led to the general field of spintronics, it is just one example of the complex magnetic phenomena that can occur at the nanoscale. Other phenomena such as interlayer exchange coupling, exchange biasing and exchange spring magnets also provide unique functionality at the nanoscale and will be highlighted.

Neutrons and Nanoscience – Some Scientific and Instrumental Perspectives

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We estimate the length- and time scales, which are accessible with neutrons from present sources and we show what can be done and what can't be done. New perspectives will emerge in reflectometry, small angle scattering and SANS-tomography; all these are based on new or recently developed experimental methods. Finally we outline some visions, capable to open new areas for neutron science.

Probing soft nanostructured interfaces with x-rays.

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In order to understand the properties of many nanoscale materials there is a critical need for structural techniques that provide in-situ, time and depth-dependent information. We will demonstrate how Grazing Incident Small Angle X-ray Scattering (GISAXS) and X-ray Reflectivity can be used to probe (i) the structure of self-assembled systems under environmental conditions on the 1-100 nanometers length scale, and to study (ii) the effect of liquids on nano-structured surfaces.

(i) In order to control and direct self-assembly on the nanoscale, an understanding of kinetics processes and equilibrium states are required. In-situ structural studies on several self-assembled systems, including diblock copolymers and nanoparticles will be presented. For diblock copolymers, hexagonally packed cylinders are formed in a specific range of the diblock's fraction and Flory interaction parameter. These cylinders may orient either parallel or perpendicular to the substrate and the degree of crystalline order is in part controlled by the solvent concentration and film thickness. Real-time GISAXS studies, along with optical thickness measurements, provide a means of correlating the structure with the solvent concentration. In the second example, monolayers of nanoparticles, capped with organic ligands, have been investigated in the presence of ultra-thin (<10nm) liquid films. When the nanoparticle diameter is comparable to the solvent film thickness the crystallinity of the monolayer is improved, regardless of the detailed solvent properties. For poor solvents, bulk nanoparticle aggregation may be induced.

(ii) Understanding wetting phenomenon on nano-structured surfaces is crucial for enabling many emerging technologies, including nano-fluidic, advanced lithographic processes and rheological applications. Recent results on wetting of nano-patterned surfaces by simple liquids will be presented. A significant enhancement of the liquid adsorption, compared to the flat surface was detected for the filling of nanocavity with liquid. This enhancement, however, is weaker than predicted. The possible origin of this discrepancy will be discussed.

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Quantum Liquids Confined to Nanoscales Explored with Neutrons

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Neutron scattering measurements of the dynamics and structure of quantum liquids confined in nanoporous media and on surfaces has recently become possible. Porous media introduce disorder and large surface areas (2D systems) as well as nanoscale confinement. A variety of media can be investigated: Vycor, silica gels, MCM-41, nanotubes, and flat MgO surfaces but sample size and sample handling is always an issue. The neutron scattering measurements of dynamics include inelastic time of flight, triple axis, backscattering and deep inelastic scattering. The elastic scattering measurements are aimed at structure determinations on surfaces. They have been made to date at the ISIS Facility, Rutherford Appleton Laboratory, UK, and at the Institut Laue Langevin, Grenoble, largely as a result of existing collaborations and sample handling facilities. Our present goals are chiefly scientific rather than direct materials development. They are:

- a) to explore the interplay of Bose-Einstein Condensation (BEC), the elementary (phonon-roton) excitations and superfluidity of quantum systems at nanoscales. Confinement to nanoscales and disorder modifies BEC, the excitations and superfluidity differently and therefore reveals their interdependence (1). While superfluidity has been explored for many years, neutrons are unique in their ability to reveal excitations and BEC and this has only recently begun. Helium at nanoscales is a model for other disordered Bose systems (High T_c materials, Josephson Junction arrays). It is closely related to BEC and superfluidity of alkali atoms confined in magnetic traps and in optical lattices discovered in 1995 and 2002.
- b) to explore quantum liquids at negative pressures (2) and to large positive pressures (3) (metastable states in nanoconfinement).
- c) to determine the shape of the atomic momentum distribution and BEC generally in quantum solids (4) and liquids.
- d) to determine the structure of helium on nanotubes, to create 1D and 2D quantum systems and 1D-2D cross-over (5).

The unique future role of neutrons in this field and opportunities opened by the combination of improved neutron facilities and new nanostructured materials will be discussed.

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Catalysis by Metal Nanoclusters

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The electronic, structural, and chemical properties of unsupported metal and mixed-metal surfaces prepared either as single crystals or thin films have been detailed and contrasted with the corresponding properties of metal and mixed-metal nanoclusters. The latter vary in size from a few atoms to many and have been prepared on ultrathin single crystalline oxide supports of TiO_2 , Al_2O_3 , and SiO_2 . An array of surface techniques including reaction kinetics of carbon monoxide oxidation and vinyl acetate synthesis have been used to correlate catalytic function of these surfaces with their physical and electronic properties. Of special interest are the special physical and chemical properties that develop with metal cluster size reduction and/or metal-support interaction.

The active sites in catalysis

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In a broad scientific sense X-rays are an ideal probe for the study of the correlation between nanoscale structure and function. In general, the scientific questions are related to understanding the effects of finite size, local structure and confinement, and correlation of these with the unique physical/chemical properties. Diffraction and spectroscopic investigations of the atomic structure and chemical composition of nanometer-sized objects and simultaneous monitoring of chemical reactivity will lead to design of optimal materials with significant implications for the nation's energy and economic security. The ability to measure the in-situ catalytic activity of a single nanoparticle or a nanometer-sized region of a heterogeneous catalyst and correlate that with changes in the physical and electronic structure under reaction conditions of high pressure and temperature, will revolutionize the catalysis science, leading to the possibility of rational design of catalysts.

The concept of "active sites" in heterogeneous catalysis was developed around an idea of adsorbate bond activation and breaking at specific structural arrangements of surface atoms with low coordination. The identification and characterization of such active sites is paramount importance for understanding of surface chemical reaction and rate limiting steps, and for the improvement of catalysts' activity/selectivity. Using the state-of the-art instrumentation and theoretical calculations, structural defects on extended surfaces (vacancies, impurity atoms, steps, kinks, dislocations and strain etc) were identified as centers of locally increased chemical or catalytic activity. Currently, the catalysts and the active sites are described as static structural units. As demonstrated in several recent experiments the dynamic active sites are formed and dissolved on metal surfaces at relevant temperatures and adsorbate concentration. Experimental tools with very high spatial, temporal, and energy resolution will have to be developed and used in combination or simultaneously to address fundamental questions of crucial importance for catalysis science. Microsecond X-ray diffraction and absorption experiments under reaction conditions, Fast high-pressure high-energy resolution photoemission, and element specific LEEM/XPEEM with nanometer scale resolution are several examples of synchrotron-based tools that will have a great impact on future development of catalysis science.

Neutron guides with adiabatic shape transformation

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Using rectangular shaped neutron guides can lead to a change in divergence which might be very unfavorable for certain instruments, for example a horizontal reflectometer. The reason is missing exchange of horizontal and vertical divergence in such guides, because all guide surfaces are perpendicular to each other. Guide with curved surfaces may provide a solution to this problem, by making exchange between horizontal and vertical divergence possible. In my talk I will present the concept of an adiabatic shape transformation guide, which was suggested by Roland Gähler (ILL, Grenoble). After demonstrating the geometry design of that special shape I will show some Monte Carlo simulation results and talk about advantages and weak points of such a guide.

Advanced Characterization Needs for Epitaxially Grown Nano-Structures

Robert Hull

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The epitaxial growth of two dimensional (thin layers), one dimensional (wires), and zero dimensional (quantum dot) epitaxial structures is a core field of nanoscience, particularly in semiconductor heteroepitaxy. Such structures enable confinement of electronic carriers, engineering of quantum properties, and ultimately control of electronic (charge) and magnetic (spin) properties at the single electron level. Arguably, the demands on advanced characterization methods are outstripping nanofabrication capabilities in this field. Challenges include the need to measure chemistry in extremely small structures, to understand the time evolution of structures through in-situ methods, to determine the presence of electrically active impurities (dopants) and to measure surface and sub-atomic structure with atomic precision. This overview will illustrate these challenges, and pose the question of what breakthrough experiments / techniques are needed in this field.

Scientific Opportunities with Synchrotron Radiation for Nanoscience

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High brightness, tunability, timing structure and polarization available from modern synchrotron radiation facilities provide unique opportunities for nanoscience research. Synchrotron radiation provides the unique capabilities to perform *in situ* studies of materials in real environment. Various spectroscopies available with synchrotron radiation allow to study quantum confinement and electronic structure of materials with high resolution. Diffraction techniques provide the information for atomic structure. Time-resolved dynamic capabilities are provided by the pulsed nature of radiation as well as by the use of coherent scattering techniques. High brightness of synchrotron radiation allows focusing the beam down to 10's of nm dimension and thus permitting single nano particle imaging. This talk will provide information about the present state-of-the-art in synchrotron radiation instrumentation and techniques and how it is used for understanding various properties of nano structures. The talk will also discuss new scientific opportunities in nanoscience where development of new instrumentation could play an important role.

Advanced Neutron and X-ray Focusing Optics for the Study of Nanoscale Materials

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With sufficient spatial resolution, X-ray diffraction and spectroscopy can provide detailed information about the composition, crystalline structure, and chemistry of individual nanoscale particles. This information is essential to understand the materials behavior of nanoscale regions embedded in larger sample volumes or surrounded by other nanoscale regions with distinct crystallographic orientation or structure. Polychromatic or nondispersive scanning methods are particularly important as they minimize problems of sample motion that are particularly serious for nanoscale research and allow for rapid determination of the local crystal structure in a small region. Similarly, advanced neutron and x-ray diffraction and spectroscopy methods form the basis for characterizing the *average* properties of nanoscale materials. Here again the ability to study small volumes is of critical importance for understanding small amounts of material prepared by novel synthesis methods. The use of polychromatic neutron techniques is particularly important for small sample volumes; polychromatic techniques efficiently use more neutrons to maximize the signal from small, weakly-interacting samples.

Fortunately, the ability to efficiently focus polychromatic/wide bandpass neutron and x-rays beams to small sizes is undergoing a revolution. This revolution, combined with the matching 3D resolution offered by differential aperture microscopy, will greatly extend the kinds of nanoscale materials that can be studied with neutrons and x-rays from major synchrotron and neutron facilities. For example, high-performance neutron supermirrors can now focus polychromatic neutron beams to $\sim 0.01\text{-}0.001\text{ mm}^2$ with brilliance approaching the theoretical limit determined by the source. This allows for the application of advanced neutron diffraction and spectroscopy experiments to very small sample volumes with good signal-to-noise. Nondispersive Kirkpatrick-Baez x-ray mirrors can similarly now focus beams to $\sim 40\text{-}100\text{ nm}$, which allows for spectroscopy and diffraction studies of individual nanoscale regions within larger sample volumes. Even more advanced focusing methods are emerging with the potential to produce x-ray beams with spot sizes near 10 nm ($0.0001\text{ }\mu\text{m}^2$) and with the ability to produce neutron beams with sufficient intensity to characterize sample volumes less than 10^{-6} mm^3 . We discuss these developing methods and their applications to nanoscale research.

Understanding Magnetic Nanostructures with Soft X-rays

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Because of its elemental specificity and significant probing depth, X-ray spectroscopy, imaging, and scattering are unique tools for understanding buried nanostructures. With the addition of magnetic contrast from polarization control of the incident light, synchrotron-based soft X-ray characterization has already proved to be a powerful tool in understanding outstanding problems in the field of magnetism and magnetic nanostructures. Soft X-ray absorption spectroscopy (XAS) can be, and has been used; to follow the evolution of the electronic structure of small particles from a few atoms to large nanoclusters, to elucidate the effect on the electronic and magnetic structure of nanoparticle coatings and nanoparticle self-assembly, and to investigate electronic and magnetic response to the unique stresses associated with nanostructure formation. X-ray resonant scattering and imaging (with the magnetic counterparts, X-ray resonant magnetic scattering and imaging with X-ray polarization selection) have complemented the electronic probes to characterize structure and assembly. Because soft X-rays probe the appropriate nanometer lengthscale, they are ideal for determining the overall size and shape of nanostructured elements and for determining inter-element assembly and interactions. These techniques have extended our understanding in the technologically important areas of exchange bias, magnetic nanoparticle interactions, and for characterizing the new-classes nanostructured magnetic semiconductors and half-metals. The simultaneous electronic, magnetic and structural characterization of buried nanostructures is a demonstrated and unique capability of soft X-ray absorption, imaging and scattering.

With the continued improvement of high brightness, stable, polarization selectable X-ray sources, future developments in nanoscale characterization can benefit from advances in end-station capabilities and detector capacity. X-ray absorption can always be incorporated as a component of X-ray scattering or imaging to allow for simultaneous electronic and structural studies. Of particular importance are the new advances in coherent light scattering and lens-less imaging that have tremendous potential for understanding interacting nanostructures. Complimentary studies of both the average behavior of many nanostructures and the specific behavior of a single nanostructure are needed. More difficult is the incorporation of high time resolution for dynamic studies or in-situ nanoparticle synthesis and assembly to characterize these processes. Only by understanding the synthesis and formation processes can substantial control be afforded.

X-rays and Neutrons for Directed and Self-Assembly

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Self-assembly driven by thermodynamics, and directed self-assembly guided by external fields, offer two important ways to form heterogeneous nano-scale structures. When the structures have characteristic length scales on the order of 1 to 100 nanometers they are ideally examined by small-angle scattering (SAS) methods, and both x-rays and neutrons have advantages. Dynamic processes can also be probed by neutron spin echo methods. A range of examples of the power of SAS arise from the study of surfactant self-assembly into micelles, vesicles, and microemulsions. The characterization methods for these assemblies, and the ability of small-angle neutron scattering (SANS) in particular to provide unique information, are highlighted here.

Of particular utility is that polymerization reactions can be carried out in microstructured fluids such as microemulsions and vesicular solutions to yield novel polymer molecules and nanostructures. (Microemulsions are equilibrium phases that contain immiscible liquids such as oil and water stabilized by a surfactant film). We have carried out polymerization of several monomers in oil-in-water microemulsions and have produced very stable monodisperse lattices with particle sizes as small as 10 nm. Fast polymerization rates, high conversions and ultra-high molecular weight polymers are achieved with both oil-soluble and water-soluble initiators. A theory of the process will be sketched along with confirmatory novel SANS experiments. Further analysis isolates the effects of monomer water solubility, glass transition temperatures, and termination processes on the polymerization pathways. A fairly complete description of the process is in hand.

Closed spherical polymer shells can be synthesized via polymerization of monomers taken up in closed surfactant bilayers called vesicles. Typical vesicle dispersions are formed by mechanically disrupting a lamellar phase, but vesicles form spontaneously in mixtures of cationic and anionic surfactant. Proper use of surfactant mixtures avoids syntheses of specialized surfactant molecules, and indeed the electrostatic interactions of anionic and cationic surfactants makes available a rich variety of microstructures. The unilamellar vesicles that form spontaneously can be used as templates or molds for polymerization reactions, and the resulting products are characterized by Cryo-TEM and scattering experiments.

Finally, new examples of instrumentation for SAS measurements will be discussed.

Scattering techniques for the characterization of silicon based nano-materials

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X-ray and neutron scattering has become a necessary characterization technique not only for inner structural study, but also for the study of surfaces and interfaces of silicone base nano-materials. For the development of novel nano materials in Dow Corning, the microstructure study related to their physical and chemical properties is fundamental. For this, scattering techniques have been effectively used for the study of fine scale phenomena in materials. In the study of nano porous thin films for dielectric (low-k) applications, the combined methods of x-ray reflectivity and small angle neutron scattering (SANS) at NIST provided the information on film thickness, average mass density, density depth profile, wall density, porosity, average pore size, pore spacing, pore connectivity and atomic composition. The results indicated that lower dielectric constant films have larger porosities and average pore sizes but lower wall densities. Scattering techniques were also used for the characterization of silicone modified dendritic polymers. SANS measured the radius of gyration of dendrimers which is an average spatial distribution of all the units and the results indicated that the dendrimers are spherical. For the study of polymer nano-composite, such as PS-PDMS block copolymers, small angle x-ray scattering (SAXS) monitored the phase segregation behavior of two different block components in the block copolymer under various thermodynamic conditions. The well-ordered pattern in the block copolymers can be varied via the chain length, weight ratio of block components, and the processing conditions. In Dow Corning, current research fields such as carbon nano composites with silicone polymers, photo luminescent nano particles, the development of thin film barrier material and nano vesicles also require scattering techniques to understand and control the structural behavior in order to obtain desirable physical properties. Demand for establishing expertise and developing scattering techniques for advanced materials research within Dow Corning is increasing and drives our interest.

Small is Different: Emergent behavior in the nanoscale

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Investigations of physical systems of small sizes and reduced dimensionalities, exhibiting discrete quantized energy level spectra and specific structures and morphologies, open avenues for systematic explorations of the physical factors and unifying principles that underlie the transition from the atomic and molecular domain to the condensed phase regime. Such behavior, where the properties do not scale with the reduced physical size, but rather where *Small is Different* in an essential way that can not be deduced through extrapolation from knowledge of bulk behavior, is *emergent* in nature. Often, the *new and different* behavior at the nanoscale can be traced to the circumstance where one (or more) of the physical dimensions of the material aggregate approaches a length-scale characteristic to a physical phenomenon (with different phenomena being characterized by different length-scales).

Gaining insights into the nature of physical and chemical systems of highly reduced sizes, and developing experimental and theoretical methodologies aimed at probing, manipulating and controlling them on the atomic and molecular level, are among the major challenges of current basic interdisciplinary research. Computationally-based theoretical modeling and simulations play an increasingly important role in modern condensed matter physics, chemistry, materials science, and biology. In particular, such studies, that may be called “*computational microscopies*”, allow explorations of complex phenomena with refined resolution in space and time [1].

The use of *atomistic simulations as tools of discovery* will be discussed and demonstrated through studies of: liquid nanojets and lubricated nanojunctions, hydrogen welding and switching in gold nanowires, the surprising nanocatalytic activity of small gold aggregates, post-ionization counterion-assisted hole transport in DNA and the mechanism of the reaction of ionized DNA with water that may cause mutagenesis and disease.

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Biological Applications and Opportunities for Nanoscience

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I will present two examples of biological applications made possible by advances in the nanosciences. First, I will describe the ability to obtain 3-D reconstructions of whole cells in their native environment. Clearly the development of modern synchrotrons was critical to the development of this technology, but equally important was the significant progress in the development of zone plate optics. These diffractive lenses have enabled collection of biological images at better than 50 nm resolution, and continued improvements are on the horizon since these optics are now capable of achieving 15 nm resolution on test specimens. More important, we are not yet at the fundamental limits since we are using a 2.4 nm wavelength source. Continued nanofabrication developments are required to further enhance the resolution capabilities.

Second, I will describe the use of semi-conductor nanocrystals (quantum dots, or QDs) to examine cell behavior associated with the invasive potential of cancer cells. Tumor cells engulf the fluorescent nanocrystals as they crawl and leave behind a fluorescent-free trail. The relative area of this trail can be directly related to degree of tumor invasiveness. We also used QDs to examine the interactions between breast tumor cells and human mammary acini (clusters of polarized epithelial cells that closely resemble functional breast tissue). These studies of 3-D tissue cultures revealed that polarized cells ward off invasion of tumor cells by a mechanism that results in apoptosis of the tumor cells. This behavior is not observed in cells grown in typical 2D culture systems. Use of QDs enabled individual cells to be tracked over the extended time periods required for these studies.

Neutron Studies of Adsorption on Novel Nanometer Materials

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Neutron scattering techniques are particularly well suited for exploring the structure and dynamics of molecules adsorbed to or entrained within materials having a high surface-to-volume ratio. Using a combination of synthesis, thermodynamic, and elastic and inelastic neutron and computer simulation techniques we have investigated the adsorption properties of monolayer and multilayer molecular films on a variety of carbon based and metal oxide nanometer sized materials. Many of these experiments can be considered as a prelude for understanding surface mediated chemical reactions. Current efforts are aimed at understanding the structure and dynamics of nanoscale catalysts, catalytic supports and reactant-catalyst interactions using model catalysts and metal oxide supports decorated with nanometer sized metal and bimetallic clusters. Studies of translational, rotational and vibrational motion combined with structural measurements yield microscopic information about molecule-molecule and molecule-surface interactions, barriers to rotation, and reaction intermediate species. This microscopic information can be combined with electronic, chemical and thermodynamic data to develop realistic models of the potential energy surface and reaction mechanisms. In-situ studies under reactive and non-reactive conditions are needed to determine the dynamics of reactions and transformation of adsorbed species on heterogeneous catalysts and to provide fundamental insights into catalytic mechanisms, reaction intermediates, and catalyst deactivation/poisoning processes. Instrumentation that enables these in-situ studies to be performed and is compatible with the neutron scattering environment must also be developed. Many of these studies have been performed in Europe (ISIS and the ILL) because current US neutron fluxes are limited and instrumentation is not yet available. VISION, a neutron vibrational spectrometer with simultaneous diffraction capabilities is currently being developed for the Spallation Neutron Source and will provide significant opportunities to investigate a wide range of nanometer scale materials including sorbents, catalysts and catalytic supports.

Neutron scattering probes of phase separation and interface magnetism in magnetic oxides and metallic heterostructures

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I will provide a couple of examples of the use of current polarized neutron reflectometry (PNR) and small-angle neutron scattering (SANS) techniques in the study of important problems in nanomagnetism. Using PNR we have probed the depth dependent chemical and magnetic scattering density profile at antiferromagnet / ferromagnet interfaces to elucidate the physics behind the exchange bias phenomenon. This exchange bias effect is of fundamental interest in addition to being used in technological applications in the magnetic recording industry. Surprisingly, we observe a temperature dependent *magnetic* interface location, which is ascribed to a strongly temperature dependent competition between antiferromagnetic and ferromagnetic interactions in the interdiffused region near the interface. This example highlights the use of PNR to examine the general issue of the relationship between chemical and magnetic interface morphologies.

In the second example we have used SANS to probe the magnetoelectronic phase separation that occurs in doped perovskite cobaltites. This phase separation is intrinsic (i.e. it occurs in the absence of chemical segregation), is common to numerous transition metal oxides, and plays a key role in their most intriguing properties, such as colossal magnetoresistance and high temperature superconductivity. SANS reveals the formation of close packed nanoscopic ferromagnetic clusters in a non-ferromagnetic matrix, allowing for the observation of giant magnetoresistance-type phenomena in a system that has no chemical interfaces. This opens up the possibility of using this phase separation to fabricate “interface-less” nanostructures with useful properties.

Finally I will discuss some areas, such as inelastic scattering from thin films and nanostructures and critical improvements in sample environment, which, if realized, would allow for great advances in many nanomagnetism problems. In particular I will emphasize the potential benefits that would be provided by the ability to routinely probe spin wave excitations in thin film heterostructures, and magnetic Bragg reflections in relatively thin films.

Nanomagnetism: Relevance, Issues and Tools

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Nanostructured magnetic materials form the basis of a variety of functional devices that support National strategic missions. These include ultra-strong, light-weight magnetic components, highly efficient electrical transformers and vibrational energy harvesting devices to foster energy independence, new medical protocols for advanced health care, and new spintronic technologies that streamline communications and stimulate the economy. The phenomenon of magnetism itself is determined by the local electronic bonding environment and is hence inherently a nanoscale issue. Very subtle aspects of the local structure (1-, 2- or 3-D defects, antisite occupancies, variable compositional and roughness profiles and strain) can alter the magnetic exchange length and create a dramatic change in the macroscopic technical magnetic properties such as remanence and coercivity. Such information is largely missing from most nanostructured magnetic materials studies.

Harnessing the potential of nanostructured magnetic materials requires atomic-level, element-specific probes that simultaneously monitor the magnetic response and the lattice structure with variable temperature capability in applied magnetic, electric and strain fields. In specific, correlation between the magnetism and the structure at an interphase interface is of particular interest, especially for the so-called “buried interfaces” inherent to nanostructured magnetic systems. It is essential to capture the magnetism and structure of interfaces located far from the free surfaces. While these interfaces are well-defined in thin-film multilayer geometries, the more challenging interfacial regions of granular films and nanocrystalline bulk materials also warrant attention.

As the key element to this challenge is the ability to non-destructively probe matter at specified depths, there are opportunities for both synchrotron and neutron radiation. All techniques may be applied, including scattering, imaging and spectroscopy. The synchrotron radiation feature of element specificity is very important. Improvements in depth resolution and beam size are desirable, as are features such as *in-situ* application of temperature and fields (magnetic, electric and strain). Real-time coupled structural and magnetic imaging of interfaces under *in-situ* annealing conditions would be an exciting way to elucidate subtle structure-magnetism connections in magnetic nanomaterials.

Small Angle X-ray Scattering/Diffraction of Supramolecular Assemblies

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One of the areas in which x-ray and neutron techniques can make a big impact is in studying the structure and interaction of supramolecular assemblies of macromolecules (e.g. polymers, proteins and nucleic acids) as well as nanoparticles self-assembled in solution or on surfaces. The formation of these hierarchical structures with length scales spanning a few nanometers to thousands of nanometers is driven by a variety of interactions, including specific (e.g. chemical bonding) or non-specific (electrostatic and hydration forces, etc.) ones. Understanding the nanoscale structure and interaction, and ultimately achieving control of the self-assembly process, has far-reaching scientific and technological implications. Nature provides us with ample examples of self-assembly in work; for example, a large number of sub-cellular structures (e.g. cytoskeleton) are formed and controlled through this process. The multitude of structural morphologies exhibited in nature has lent much inspiration to producing man-made nanostructured materials for a broad range of applications including drug and gene delivery agents, nanoporous networks, miniaturized bio-sensors, etc.

Applying x-ray and neutron techniques to studying self-assembly presents some unique challenges. One common characteristic of the self-assembled structures is the low degree of ordering in the material, which weakens the scattering signal. Consequently the data quality improves tremendously by using high brilliance sources. In addition, the low scattering angles associated with the large lengthscale (1 nm – 100 nm) of the structure require special data collection as well as analysis methods. Small angle x-ray scattering (SAXS) and small angle x-ray diffraction (SAXD) are the most common tools used for investigating the structures of supramolecular assemblies.

To elucidate the complex structures of self-assembled materials generally requires the application of multiple structural tools. We have used SAXS and SAXD, in conjunction with real space imaging techniques (optical and electron microscopy) to solve a number of unique structures of the lipid-biopolymer (DNA, RNA, actin, microtubules) complexes. The complementary information provided by imaging proved crucial to the construction of the structural model which would fit the scattering data. It would be extremely useful to build instruments which perform two or more types of measurements simultaneously on the same sample. For example, by combining an epi-fluorescence or confocal optical microscope with a cutting edge SAXS instrument it would enable the investigation of structural features at the nano- and micro-scale at the same time, which would dramatically enhance the capability of each technique by itself.

Another area in which a concerted effort could be directed is to develop software tools that can perform sophisticated analysis and modeling of scattering data from non-crystalline samples with an easy-to-use interface. The huge success of protein crystallography can be partially attributed to the development of such packages. However currently there is no generalized software tools available to model partially ordered (1D and 2D) or completely disordered systems, which characterize most self-assembled nanostructures. This limits the ability of non-experts to derive maximum information from their data. NNI can play an important role in initiating and supporting these efforts.

Nanomaterials Science at the Advanced Photon Source

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X-ray tools for nanomaterials science enable the quantitative analysis of compositional, structural, chemical, magnetic and dynamic properties at the interatomic, atomic and molecular level, over a wide range of time scales, and including *in-situ* capabilities. The Center for Nanoscale Materials (CNM), currently under construction in partnership with the APS at sector 26, will be dedicated to the development and characterization of novel materials and devices at the nano-scale.

The CNM Hard X-ray Nanoprobe Facility will provide unique hard x-ray microscopy capabilities dedicated to the study of nanoscale materials and devices. The Nanoprobe will provide analytical capabilities at a spatial resolution of 30 nm, and provide fluorescence spectroscopy, diffraction imaging and microdiffraction and high-resolution transmission imaging.

In addition to the Nanoprobe instrument, the APS provides numerous complementary experimental facilities that serve the community of nanoscience researchers in the areas of interfacial structures, nano-systems, confinement, and self-assembly of hard materials, soft materials, and biomaterials, or nanofluidic phenomena. These facilities include several nanoprobes around the APS ring:

- x-ray-excited optical luminescence (XEOL) sensitive to structures < 10 nm
- soft-x-ray photoelectron emission spectroscopy (PEEM)
- (6 – 13 keV) scanning fluorescence microscopy and micro/nano-diffraction (70 nm probe)
- (1 keV – 4 keV) scanning transmission microscopy, scanning fluorescence microscopy, and coherent scattering (50 nm probe)

In addition to these imaging techniques, x-ray scattering provides another important approach to the study of nanoscale materials. Capabilities at the APS include:

- Characterization of magnetic interfaces using magnetic reflectivity
- Small-angle X-ray scattering and grazing incidence small-angle x-ray scattering for following, in situ and in real time, the formation of nanocrystal monolayers, nanoparticle arrays and the associated kinetics
- Coherent diffraction, currently under development at the APS and sometimes called “lensless imaging,” for imaging structures at the nanoscale
- Diffraction under *in-situ* growth, including MBE, MOCVD, and PLD
- X-ray photon correlated spectroscopy (XPCS)

Characterization of Functionalized Biomimetic Membranes

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A new neutron spectrometer – the *Advanced Neutron Reflectometer and Diffractometer AND/R* – has been recently commissioned by the *Cold Neutrons in Biology and Technology* (CNBT) research consortium, a U.S. university/government lab research partnership located at the NIST Center for Neutron Research. This new instrument is optimized to investigate specifically problems in membrane biology and biotechnology. These are mostly concerned with dynamic, structurally disordered systems. Thus, MD simulations, solution scattering and NMR are an integral part of the CNBT mission. Investigations in the first year of AND/R operation include studies of surface-grafted DNA, chain interdiffusion within polyelectrolyte multilayers, and high-resolution structures of hydrated lipid membranes. This contribution will focus on studies of surface-tethered membrane structures for the development of robust sensing applications.

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Superlattice Crystals for Forefront Neutron Science

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The outstanding challenge of 21st Century science that can be addressed by neutron scattering is the need to understand self-organizing behavior that first emerges on the nanoscale in chemically complex systems. Electronically highly correlated and structurally similar complex oxides epitomize this challenge, by exhibiting a rich variety of electrically conducting and insulating behavior, high-temperature superconductivity, magnetism, colossal magnetoresistance, and ferroelectricity. In the words of Birgenau and Kastner, these phenomena “present us with profound new problems that ...represent deep and formidable challenges ...”, for which “...neutron scattering is an absolutely indispensable tool ...” [1] However, the most revealing neutron scattering studies require single-crystal samples of significant size. Moreover, conventional crystal growth offers limited capacity to design novel samples for interesting experiments.

In contrast, new understanding and methods for pulsed laser deposition (PLD) recently enabled us to grow one micrometer-thick oxide heterostructures with atomic-layer control and complete reproducibility of interfacial and surface quality over thousands of unit cells. These advances were demonstrated by carrying out the first experimental verification of theoretically predicted polarization enhancements in artificial PLD ferroelectric “superlattice crystals” grown from periodically repeated SrTiO₃, BaTiO₃, and CaTiO₃ building blocks. [2] Subsequent simulations and experience-based estimates show that PLD growth of crystalline heterostructures with sufficient mass for the full range of bulk-like neutron scattering measurements — reflectometry, diffraction, and inelastic scattering — now is feasible.

Consequently, there now is an outstanding opportunity and need to build a first-of-its-kind “superlattice crystal” growth facility to provide *thick* (up to 100 μm) epitaxial oxide heterostructures for forefront neutron scattering studies at the Spallation Neutron Source (SNS). This growth facility will permit neutron scattering to move beyond the limits currently imposed by conventional crystal growth. Novel properties are known often to result from the competition between the physical dimensions of nanostructures and the length scale(s) relevant to magnetism and other collective phenomena. Consequently, the *ability to control the dimensions of individual components within multi-layered samples at the nanoscale is essential*. Others also have pointed out recently that the *design* of novel samples should be *an integral part of the planning process* for a major new neutron scattering facility. [3] The benefits of developing a PLD-based epitaxial heterostructure growth facility will be to fully engage the international scientific community — theorists and experimentalists — in designing materials to create new phenomena, and to greatly enrich the data stream resulting from the national investment at the SNS.

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3D Coherent Diffraction Microscopy and Its Applications in Nanoscience

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Visualizing the arrangement of atoms has played a crucial role in the rapid progress of nanoscience and technology. There are already a few ways of imaging atomic structures, but each has its limitations. Scanning probe microscopes are limited to imaging atomic structures at the surface. Transmission electron microscopes can resolve atoms but only for samples thinner than ~ 30 nm. Crystallography can reveal the globally averaged 3D atomic structures based on the diffraction phenomenon, but requires crystals. These limitations can in principle be overcome by coherent diffraction microscopy based upon coherent X-ray scattering in combination with the direct phase recovery method called oversampling. Coherent diffraction microscopy has been successfully applied to 2D and 3D imaging of nanoscale materials and biological systems. A highest spatial resolution of 7 nm has been achieved, while the ultimate resolution is only limited by the X-ray wavelengths. By using the 3rd generation synchrotron radiation sources, better-designed instruments and more robust image reconstruction algorithms, we expect to improve the spatial resolution to the 1 nm level within the next few years.

Meanwhile, we will also pursue its applications in materials and nano science. We will focus on 3D imaging of porous silica particles, GaN semiconductors, quantum dots and mineralized bone. The ability to image the internal pore structures in three dimensions, 3D morphology of GaN and its alloys in nanocrystal form, and 3D internal structures of quantum dots, coupled with computational methods such as molecular dynamics and *ab initio* calculations, will profoundly expand our understanding of the critical structural and morphological features required to make superior catalysts, adsorbents, electrodes or semiconductors. Understanding the mineral component of bone such as the size, shape and arrangement of the calcium apatite crystals in a collagen matrix will be of fundamental importance in biology and medicine.

In the long run, with the prospects of brighter X-ray sources such as the Linac Coherent Light Source and pixel array detectors of higher quantum efficiency and a higher dynamic range, coherent diffraction microscopy can potentially be used to determine the 3D structures of single particles down to the atomic level.

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Improvements to Existing Facilities for the Study of Nanoscale Materials

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Quantum size effects in nanoscale materials with reduced dimensions can have a profound influence on the properties of a material and on the way materials assemble and grow. A more significant effect, however, is the role played by surfaces, interfaces and internal defects, which can dominate the energetics of these small systems. Therefore, in order to ultimately harness potential applications of nanoscale materials, we will need to understand their structure. Surface x-ray scattering has a truly unique role to play here because of its ability to obtain both surface and subsurface structure over a range of length scales. Our studies of metals have amply demonstrated the importance of looking at subsurface structure in relation to the surface.

The small bright beams provided by 3rd generation synchrotron radiation sources are well suited for these studies. Two significant improvements would better utilize what we already have.

- As x-ray sources and optics have developed, a similar development of detectors has not kept pace. There is a need for faster multi-element detectors for real time measurements, detectors with significantly larger dynamic range of count rate, and for multi-element energy sensitive detectors.
- There is a need to expand the available beam time for in situ studies, which are essential for understanding nanoscale phenomena. In situ studies in general and in situ surface science studies in particular are time consuming measurements. This fact needs to be recognized when designing beam lines and allocating time. A second important facet of this problem concerns student training. Since surface scattering experiments require a “sophisticated user”, student training needs to allow the time for students to make some mistakes and work independently. It is a crucial consideration for producing the next generation of scientists who will develop future infrastructure.

Regarding neutron sources, existing smaller satellite reactors can play a significant supporting role for national facilities. Unlike laboratory x-ray sources which are many orders of magnitude weaker than synchrotron sources, satellite reactors are only a factor of 2 to 10 weaker than national facilities. Thus, satellite reactors can provide much needed training, concept and instrument development, as well as supporting research for the national facilities. This model has been successfully employed in Europe for decades.

Magnetic Excitations in Dimensionally Restricted Systems

Stephen Nagler

Oak Ridge National Laboratory

With the advent of next generation neutron sources inelastic scattering measurements will be possible on smaller sample masses with higher resolution than has been achievable to date. Characterization of the excitations in systems with nano-scale dimensional restriction should become possible. Examples include thin films and multilayers, arrays of magnetic nano-wires, assemblies of magnetic nano-particles or quantum dots, magnetic molecules, and magnetically dilute systems.

Marrying Reaction Chemistry to Surfaces

Colin Nuckolls

Department of Chemistry and The Nanoscience Center, Columbia University

This presentation will explore methods to direct the assembly of molecular wires on metal and metal oxide surfaces and to measure their electrical properties in nanoscale test structures. Attachment of aromatic molecules to oxide surfaces using multivalent interactions provides high coverage monolayers. Synchrotron reflectivity on these monolayers reveals that they are as tightly packed as molecules in bulk crystalline state. These tightly packed monolayers spontaneously assemble to form monolayer field effect transistors.

A parallel effort is developing the chemistry to grow wires from metal surfaces. The presentation will explore new monolayers that form on some metal surfaces reacted with carbenes. These monolayers are exceedingly stable and are able to initiate olefin metathesis. This interfacial chemistry provides a method to both structurally and electrically contact molecules on metal surfaces.

Patterned Nanoscale Materials

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This talk will focus on how patterning and synthesis at the nanometer-scale enable unique ways to study a broad range of scientific queries. Our research uses developments in nanopatterning to address important problems in chemistry, materials, and photonics. This combination has significant promise for the discovery of new fundamental principles in physical chemistry and the advancement of sensitive and powerful analytical techniques. Using a variety of unconventional patterning methods, we have generated nanostructured surfaces with three-dimensional features at nm-length scales and over cm²-areas in a single step; moreover, these patterns exhibit hierarchical order and selective chemical functionality from which to build or assemble new types of mesoscale (100-1000 nm) structures. One outstanding issue is the characterization of nanostructures on surfaces. X-ray scattering at small angles can help solve the crystalline structure, the size distribution and orientation, and the interface between the surface and the nanoscale materials. One way that the current resources can be applied to nanoscience problems is to construct a simple but informative site that lists common challenges of nanoscale characterization—and then how x-ray/neutron techniques can solve them. Individual PIs could then contact the appropriate scientists to set-up collaborations and analyses.

**Functional Bio-nano molecular Materials:
Is there Something to Learn from Mother Nature?**

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Nature synthesizes functional materials in an integrated and highly controlled manner. The study of this integrated system is the realm of the developing field of SYSTEMS BIOLOGY. Biology celebrates diversity but at the molecular/cellular levels, a few motifs are seen repeatedly. We provide some examples in the context of functional materials. These include hierarchical assembly, motor driven synthesis, programmed assembly, functional materials. A challenge for the Neutron/ X- Ray communities is to provide tools for the characterization of hierarchical systems at both the static and slow dynamical levels.

Challenges for X-Ray Characterization of Nanoscale Soft Matter Assemblies

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The soft matter systems that are being studied at the nanoscale are often hybrid assemblies, for example, nanoparticles in a polymer matrix or inorganic nanocrystals decorating organic fibrils. Such hybrid systems incorporate ordering from nanometer to micrometer length scales with the local atomic structure and associated functionality being as important as the long-range correlations. The systems are often radiation and environmentally sensitive, when fabricated into devices contain a range of different types of buried interfaces, are usually solution-processed, and their assembly is often directed by mechanical constraints, chemical templates, or external fields. The x-ray characterization of the evolving structure of these hybrid systems, under processing conditions, presents considerable challenges for current synchrotron x-ray facilities.

The current trend at synchrotron facilities is to instrument and staff beamlines optimized for a specific x-ray technique. This specialization offers advantages in terms of rapid access to standardized set-ups as well as access to instrumentation that is fine-tuned for specific techniques. These optimized beamlines, especially x-ray nanoprobe and SAXS-GISAXS beamlines with in-situ processing chambers and complementary probes, will address most of the characterization needs for soft matter nanoscience assemblies. Nonetheless, we propose that there will continue to be a need for 'general-purpose' beamlines to provide the flexibility for researchers to develop new approaches to sample characterization. In this presentation, we will describe two efforts to develop new approaches for soft matter characterization utilizing 'general-purpose' undulator beamlines. One project involved the study of 100 μm x 100 μm patterned alignment areas of layered liquid crystals created using AFM-scribed polymer alignment coatings while the second project involved an analysis of crystal truncation rods to determine model-free electron density maps of semiconducting organic thin film interfaces. Future refinements of these techniques will be described along with the features of the 'general-purpose' diffraction beamlines which made their initial implementation feasible.

Advanced Instrumentation for Neutron Scattering Studies of Nanoscale Materials

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Fundamental properties of the neutron's interaction with matter make neutron scattering a tool that is especially well suited to characterizing the structure and low-energy dynamics of both hydrogen-containing and magnetic materials. The technique has already contributed significantly to research on nanoscale materials of these types, such as complex fluids and magnetic multilayers, and I will present some illustrative examples to show the range of information that has been obtained. To make neutron scattering even more useful for the characterization of nanoscale materials, several more or less serious impediments will need to be overcome through the development of a new generation of neutron scattering instrumentation and through the coupling of neutron instrumentation and high performance computer simulation. I will discuss in some detail one of the more serious impediments – the fact that neutron scattering is a signal-limited technique. One consequence of this is that it is difficult to study small or dilute samples, features that are quite common for nanoscale materials produced either by self-assembly or by deposition techniques. A second consequence arises because nanoscale materials display order on length scales that are typically between one and three orders of magnitude larger than the wavelengths of cold neutrons. Resolving such length scales with neutron diffraction generally demands tight collimation of the neutron beam, reducing the neutron count rates and limiting the statistical precision of the measured neutron intensity. I will discuss these limitations and suggest ways in which they might be overcome with a new generation of neutron scattering instrumentation.

Reinforcing Polymer Composites with Protein Functionalized Nanotubes

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We have developed fully integrated nanotube composite materials through the functionalization of multiwall carbon nanotubes (MWCNTs) by covalently attaching ferritin protein molecules onto the surface of MWCNTs. The investigation of the thermo-mechanical behavior was performed by dynamic mechanical thermal analysis (DMTA). The results demonstrate a dramatic enhancement in the mechanical properties of PVA, for example a 100-110% increase in the modulus with the addition of 1.5 wt% of ferritin functionalized MWCNTs. Samples containing functionalized nanotubes show a stronger influence on glass transition temperature than composites containing the same amount of non-functionalized nanotubes.

High-resolution neutron scattering studies with back-scattering and/or spin-echo techniques as the glass transition is approached from above would provide a microscopic picture of the macroscopic dynamics probed by the mechanical property measurements. In addition, inelastic neutron scattering could be used to confirm the covalent bonding between amino and carboxylic- functionalities on the surface of ferritin functionalized nanotubes and the PVA matrix.

Probing nanomagnetism with neutrons and advanced simulations

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In recent years there has been wide interest in the study of highly anisotropic magnetic nanoparticles. Besides basic science interests, there are important applications of these systems for future data storage and information processing technologies as well as permanent magnets. Significant progress has been made in the synthesis of mono-disperse assemblies of complex nanoparticles, such as FePt, CoPt, and various transition metal oxides. However, the understanding of their magnetic properties has been lagging behind, largely due to lack of adequate experimental probes. One of the urgent issues seems to be the understanding and control of the magnetic switching in nanoparticles. It appears that these processes are more complex than expected from simple models. But the involved length and time scales are outside the reach of conventional magnetization measurement techniques.

Here is where a systematically coordinated effort of polarized neutron scattering experiments and advanced theory and modeling can make significant contributions. Neutron scattering experiments are capable of probing magnetic structure as well as magnetic excitations at intra and inter particle length scales. With advanced simulation techniques it is now possible to model from first principles the magnetic structure of nanoparticles and to reproduce the measured dynamic correlations functions of spin models. With a combined neutron characterization and modeling effort, it should be possible to fine tune spin models of nanoparticles. Parameters of these models can be gauged by comparison of the magnetic structure and excitations with first principles calculations and inelastic neutron scattering spectra, respectively. These models can subsequently be used to study magnetic switching and other properties, in order to help understand and guide more conventional magnetic characterization techniques.

The theoretical methods and software tools as well as the neutron scattering techniques that are being developed in this effort are not limited to magnetic nanoparticles. The coordinated effort of developing joint neutron scattering methods and simulation tools for magnetic nano-structures will therefore generate solutions to other characterization problems in nanoscience.

Neutron Refractive Optics and Peripheral Techniques and Their Applications

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The small-angle neutron scattering (SANS) is a general tool to investigate the static structure in the mesoscopic region of about 1 nm to 100 nm. The neutron sensitivity to light elements enables us to observe the structure of soft-matters including polymers and proteins. Radiation damage s to samples can be suppressed since the neutron does not have the electric charge and its kinetic energy is sufficiently low to avoid direct destruction of chemical bandings, which is due to the neutron mass comparable with atomic mass.

The focusing of cold neutron beam using refractive optics has been applied to extend the minimum value of accessible q -region (q_{\min}) of spectrometers for small-angle neutron scattering at JAERI. The refractive optical devices were sextupole magnetic lenses and compound refractive lenses. The q_{\min} was decreased from 10^{-2} nm^{-1} to 10^{-3} nm^{-1} by employing a scintillator-based neutron-imaging detector on the focal plane together with the focusing lens. If we extend the q_{\min} by increasing neutron source intensity without the focusing lens, we need much more intense neutron source by two orders of magnitude. In other words, the “neutron utilization efficacy” was enhanced by two orders of magnitude.

The improved sensitivity introduces the SANS capability to observe long-term change in dilute soft-matter samples. The magnetic lens delivers a spin-polarized neutron beam, which enables to study magnetisms of nano-particles. It also introduces possibilities to decompose the coherent and incoherent scattering components from hydrogen nuclei. A spin contrast variation would be possible by developing techniques to dynamically polarize protons in samples.

The polarizing power of magnetic lenses is calculable from the geometry of device arrangement. The polarization loss is expected to be negligible since there is no material on the beam path. Thus, we can expect an extremely high purity of neutron spin polarization together with well-defined beam distribution both in space and beam divergence. It can be applied to improve the performance of neutron spin echo techniques both in its maximum value of the Fourier time and q_{\min} . It will contribute to observation of slow dynamics at nano-scale in soft materials.

Some possible challenges for modeling soft x-ray spectra on the nanoscale

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Soft x-ray spectroscopic techniques can help characterize many properties relevant on the nanoscale. These properties include: chemical element identification, structure (in the sense of local atomic geometry), vibration properties, oxidation states of transition-metal ions, effects of ligands, and so forth. As an example, one may consider the detailed Fe 2p absorption spectrum of an iron ion surrounded by a cage of ligand oxygens and embedded in a large molecule. The local atomic geometric and iron oxidation state can be inferred in part from the multiplet structure of the Fe 2p edge. Charge-transfer excitations and other satellite features in the spectrum might provide further insight into the interactions between the iron ion, its nearest neighbors, and perhaps other surroundings. Ultimately, if theoretical treatments can describe spectra with great detail, high energy resolution could be increasingly valuable.

To perform modeling to support and interpret experiments, it is necessary to amplify current methods that are commonly used. There are several areas for potential improvement. Often, nanoscale phenomena occur at certain sites in artificial structures or large molecules. The “relevant volume” for some nanoscale phenomena of a physical system can be small compared to the total system, and yet large compared to typical unit cells of many crystalline solids. This entails large-scale computation to model physical systems, but hopefully with attention focused mostly on the relevant volume. In order to address this, one needs to consider how to efficiently yet accurately “embed” the relevant volume in a larger system, allowing for “communication” with the surroundings along the lines of dielectric screening, electron transfer, etc, subject to sufficiently realistic “boundary conditions.” In particular, the ability to compute polarization and screening effects of electron-electron interactions in real space would be helpful, if the one-electron density matrix allows this.

In the previous example, there is also still room for improving the describing of even just the iron atoms and its ligands. It is difficult to solve the essentially configuration-interaction problem necessary to obtain a complete spectrum, and it is also difficult to know how to “truncate” one’s physical system in real space, in order to account for the placement of the iron ion and its ligand within a larger system, without worsening results through artificially imposed boundary conditions or attempting to describe the quantum mechanics of an unnecessarily large physical system.

Synergism of Neutron Scattering and Molecular Simulations in Understanding Dynamics and Structure of Polymer-Based Nanomaterials

Grant Smith

University of Utah

Neutron scattering is one of the most important probes of structural and dynamical properties of polymers on the nanometer length scale. The power and utility of neutron scattering as applied to polymers is greatly enhanced when utilized in coordination with molecular dynamics simulation studies of the same material. The synergism between neutron scattering and molecular simulations is potentially even greater for polymer-based nanocomposites and self-associating nanomaterials. The potential for coordinated neutron scattering and molecular simulation studies to provide unique insight into the nanoscale structural and dynamical behavior of polymer-based nanomaterials as well as examples of synergism between neutron scattering and molecular simulations in studies of polymers and polymer nanocomposites will be presented.

Structural studies of confined soft matter- surfaces and interfaces

Gregory S. Smith

Oak Ridge National Laboratory

This area is wide ranging in both the materials studied and the methods of confinement. The materials include biomaterials (e.g. lipids and proteins), biomimetic materials, polymers, surfactants, liquid crystals and small molecule liquids. Specifically, we are searching for novel materials properties and structures as macromolecules are confined in 2-dimensional space. 2-D confinement can be achieved in Langmuir monolayers (liquid-air interface), in solid-supported membranes or thin films (solid-liquid or solid-air interface), or by compressing thin films between parallel solid surfaces (solid-solid interface). In addition, controlled synthesis of nanomaterials and nanofabrication strategies provide an interface to biosystems on a nanoscale and allows one to probe, modify, or mimic live cell, cell components, or molecular structures, forming an emergent field of nano-enabled biology or simply nanobio. Creation of such nanoscale architectures is going to enable new strategies for probing biosystems with neutrons.

At the molecular level, our recent neutron reflectivity experiments have been very successful in providing detailed density distribution profiles of adsorbed diblock polymers confined between two substrates under good solvent conditions as a function of confinement. We are continuing to investigate polyelectrolyte brushes and extend these studies to include the ability to shear the polymer brushes. Combined with state-of-the-art techniques in polymerization and selective deuteration, we are building controlled model architectures to elucidate the structure of these complex systems at interfaces under confinement and applied shear stress. Further development of neutron and x-ray scattering techniques such as grazing incidence diffraction may yield even more in-plane detailed structural information.

One way we can increase the chances of success of neutron and x-ray studies of these materials will be to develop the sample environment equipment needed in the labs and for neutron scattering experiments specific to these special materials. For example, we will continue the development of the confinement/shear cell and extend it to the study of lipid membranes. With the advent of the SNS we can use smaller samples and get the smaller confinement gaps needed to study these systems which have intermembrane separations of approximately 5 nanometers. Similarly, continued development of new ways to modify the carbon nanofibers with various specificity will enable us to control the interactions between substrates and supported membranes.

Mesoscale inhomogeneity in colossal magnetoresistive manganites

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Manganites display a complex phase diagram, with various types of spin and charge ordering as its ground state. More interestingly, the various phases of the manganites can coexist in the same sample, leading to an inhomogeneity in the physical properties ranging from the mesoscale down to the nanoscale. This inhomogeneity is considered to be important in explaining the large colossal magnetoresistance exhibited in this class of materials. Focused x-rays are ideal for addressing the inhomogeneous phases in manganites. I will discuss how the inhomogeneity in the magnetic ordering temperature and wavelength of charge density waves can be addressed using x-ray microdiffraction, and the insight that we gain about the origin of the inhomogeneity.

Heterogeneous Catalysis: Nanoscale Meets the Atomic Scale

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There is abundant evidence that the activity and selectivity of heterogeneous catalysts are controlled by a combination of properties at both the nanoscale and the atomic scale. It is now within the realm of possibility to synthesize catalytic nanoscale particles, supported on a solid surface, with control of both their composition and topology at the atomic level. The characterization of such materials, particularly the measurements required to prove the successful synthesis of a specific composition and structure, will require x-ray and neutron measurements that can penetrate both the gas or liquid environment of the catalyst and the catalyst material itself. These measurements will not only need to provide average composition and structural information, but provide cluster-by-cluster information. During my 5-minute presentation, I will provide some specific examples from my own interests.

In Situ X-ray Studies of Nanostructure Creation

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Some of the greatest challenges in nanostructure creation are associated with understanding and optimization of synthesis and patterning processes for novel materials (e.g. complex oxides, bio-inorganic composites, etc.). Many of these processes are difficult to probe at the atomic scale by traditional electron imaging or spectroscopy, including vapor-phase processes such as chemical vapor deposition, atomic layer deposition, and reactive ion etching, as well as aqueous solution growth and etching. X-ray scattering, spectroscopy, and imaging are ideally suited to provide the first *in situ* observations of the atomic-scale mechanisms of these processes.

This is a very exciting time for progress in x-ray capabilities. Now that extremely high brilliance sources are available or under construction, large payoffs can be anticipated from developments in optics, detectors, and specialized instruments such as in-situ analysis facilities. In particular, breakthroughs in x-ray focusing optics indicate paths to achieve sub-10-nm resolution, which will provide powerful new imaging capabilities for nanostructures.

X-Ray Studies of the Nanoworld – Motivation, Concepts and Applications

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In an era where many powerful techniques can provide images of the nanoworld, I will start my talk by addressing the unique capabilities of synchrotron radiation and synchrotron radiation techniques for the study of nanomaterials. The *short wavelength* allows resolving, seeing and studying individual nano-objects. The *tunable photon energy* provides access to resonance effects that are elemental and chemical state specific and exhibit large cross sections. The latter in combination with the *high photon beam brightness* provides sensitivity to a small number of atoms. Finally, the x-ray *polarization* allows the separation of charge and spin phenomena. The interaction of selected nano-objects with the incident x-ray beam can be studied by a variety of experimental techniques that give electronic, magnetic and structural information. I will present several examples where x-rays have provided unique information on nanostructures that cannot be obtained by any other technique. Finally, I will address the important correlation between space and time phenomena – the smaller the faster. I will emphasize that technological advances require exploration of both the ultra-small and ultra-fast. This exploration is the real strength of x-rays and the basis for revolutionary developments in the future.

Probing Magnetic Nanostructures by Polarized Neutron Scattering

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Surfaces and interfaces greatly impact the characteristics of nanoscale materials. Therefore, in order to understand the physical properties, detailed characterization is essential. In the case of (magnetic) thin films and heterostructures with nanoscale thicknesses, (polarized) neutron reflectometry (PNR) has for many years been an excellent tool for probing (magnetic) interface structure. PNR provides the depth dependent magnitude as well as the orientation of the magnetization, along with the structural parameters of a thin film.

PNR studies of interface phenomena such as exchange bias and exchange coupling have led to a greater understanding in these areas. Materials nanostructured in three dimensions can be probed by small angle neutron scattering (SANS) and by grazing incident scattering, where the ensemble average of the properties is obtained. Again the depth sensitivity of surface scattering methods provides access to buried interfaces or layers, that is hardly obtainable with other techniques. I will present various examples, illustrating the effectiveness of PNR, scattering at grazing incidence, and SANS in providing important information in magnetic nanoscale materials.

Neutrons, Pressure and Nano Materials: Emerging Possibilities

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A brief overview will be given of the extreme sample environment capabilities planned for the SNS. This will particularly include design and performance features of the Spallation Neutrons And Pressure diffractometer planned for completion in early 2008. In addition, examples of our recent experience with neutron micro-focusing prototype devices will be discussed as it relates to augmenting high pressure neutron diffraction instrumentation and techniques. More generally this emerging focusing technology may help alleviate the challenges of large scale synthesis traditionally associated with neutron diffraction. Sample volumes in the sub100x100x100 micron range are now becoming feasible for neutron scattering studies. Also, future possibilities of in situ characterization during Extreme Environment Nano-Material Synthesis (XENoMS) will be briefly highlighted by showing our recent neutron studies of high pressure magnetic transitions in nano-sized particles of hematite (Fe_2O_3).

Structure and dynamics of nanostructured materials

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Neutron and synchrotron x-ray are great tools for probing the bulk behavior of a material, and nanostructured materials are no exception. With the advent of powerful sources and the scattering instrumentation, in-situ, time-resolved experiments are becoming a reality. These experiments will play a crucial role in elucidating the structure and dynamics at nanoscale.

Our research in nano science encompasses three areas: (1) Deformation mechanisms in nanostructured materials, (2) Phase transformation at nanoscale, (3) Structure and thermal stability of self-assembled nanoclusters in metallic matrices. Highlights from recent experiments will be presented.

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Applications of electron microscopy for discovery, understanding and property measurements of oxide nanostructures

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Nanowire and nanotube based materials have been demonstrated as building blocks for nanocircuits, nanosystems and nano-optoelectronics. Quasi-one-dimensional nanostructures (so called nanobelts or nanoribbons) have been successfully synthesized for semiconducting oxides of zinc, tin, indium, cadmium and gallium, by simply evaporating the desired commercial metal oxide powders at high temperatures [1]. The belt-like morphology appears to be a unique and common structural characteristic for the family of semiconducting oxides with cations of different valence states and materials of distinct crystallographic structures. Using the technique demonstrated for measuring the mechanical properties of carbon nanotubes based on in-situ transmission electron microscopy [2,3], the bending modulus of the oxide nanobelts, the workfunction at the tip have been measured. Field effect transistors [4] and ultra-sensitive nano-size gas sensors [5], nanoresonators and nanocantilevers [6] have also been fabricated based on individual nanobelts. Thermal conductivity of a nanobelt has also been measured. Very recently, nanobelts, nanorings and nanosprings that exhibit piezoelectric properties have been synthesized, which are potential candidates for nano-scale traducers, actuators and sensors [7, 8, 9, 10]. This presentation will focus on our recent progress in the controlled growth, nano-scale property measurements and nano-size device fabrication using oxide nanostructures that are semiconducting and piezoelectric.

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Characterization of the Surfaces of Nanostructures using Positron Annihilation Induced Auger and Gamma Spectroscopies

Alex Weiss¹

1. The University of Texas at Arlington

Experiments performed at the University of Texas at Arlington have demonstrated that Positron annihilation induced Auger spectroscopy (PAES) is capable of characterizing the elemental content and structure of the topmost atomic layer with single atomic layer resolution and its ability to eliminate the secondary electron background. Here we present experimental and theoretical results that indicate that positrons will trap with high efficiency in surface states localized on top of surface nano-structures. We propose to exploit this effect by developing and applying new site-specific spectroscopic tools for characterizing the elemental content and electronic structure of surface nano-structures. The elemental content of the top most layer of the nano-structure will be determined from the PAES spectra resulting from the annihilation of a positron in this surface state. In addition, information regarding the electronic structure at the surface of the nano-structure will be obtained from the energy spectra of Doppler broadened annihilation gamma rays. The localization of positrons on top of the nano structures will allow us to selectively probe the nano-structures without interference from signals from the substrate material.

X-ray Imaging at Nanometer Scale: Opportunities and Challenges

W. Yun

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X-ray imaging offers important and desirable visualization and characterization capabilities for nanoscience and nanotechnology. For example, its short wavelength permits nanometer resolution imaging without the limitation of wavelength. Its high penetration power allows nondestructive imaging of internal structures of an object. It has many contrast mechanisms that can be employed beyond simple structural imaging, such as chemical state imaging or elemental specific imaging. To realize the potential of x-ray imaging with nanometer scale resolution, there is significant challenges. In my presentation, I will present a theoretical framework why x-ray imaging with nanometer scale resolution is achievable and discuss the technical challenges that need to be overcome.