II. BREAKOUT GROUP SUMMARIES

IIi. Hard Condensed Matter: Chemistry, Materials, and Small Molecules

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Science Opportunities:

General topics of interest center around investigation of the structure, dynamics (vibrational and diffusive), and composition (speciation) of molecules at interfaces and in other confining geometries (layers and porous media). Length and time scales of interest are typically probed by neutron scattering techniques such as quasielastic neutron scattering, inelastic neutron scattering, SANS, and reflectometry. Advances in neutron scattering capabilities will impact multiple areas of strategic and technological interest such as energy (hydrogen storage, transportation, production, and conversion), synthesis and characterization of nanostructured materials, catalysis and surface science, and biological systems. Specific focus areas and representative examples include:

1) Organic/Inorganic interfaces – organic species at surfaces, porous media (bulk and nanoparticle)
    a. Ethane/Ethylene binary diffusion in Na-Y zeolites to elucidate the relationship of structure and transport of non-ideal mixtures for industrial separations.
    b. Anisotropic diffusion of xylene isomers in oriented nanoporous thin films of silica or zeolites for chemical sensing or separation applications.
2) Hybrid Materials – nanoparticle/polymer, molecules attached to nanoparticle surfaces, nanoporous layered materials
    a. Proton transport and fuel crossover in hybrid polyelectrolyte/layered silicate membranes.
3) Water at surfaces and in confinement – hydration shells, separation membranes, fuel cells, reverse micelles ("nanobeakers"), clathrates
    a. Hydration water dynamics as it influences the behavior of globular proteins and DNA.
    b. The dynamics of surface water on oxide nanoparticles.
4) Hydrogen storage materials – chemical hydrides, physisorption/chemisorption, metal hydrides
    a. Measurement of hydrogen and methane adsorption site potentials at unsaturated metal centers in metal-organic framework materials.
    b. Dynamics of alkali borohydrides in confined geometries.

Information/Technique:

1) Diffusion – self-diffusion, collective transport on length scales < 10 nm, anisotropic transport, measured using quasielastic neutron scattering, NSE
2) Quantitative assessment of hydrogen – vibrational spectroscopy, prompt γ/imaging
3) Structure – 3-d: SANS to USANS – (length scales to 10 μm), nanoparticles, hybrid materials, 1-d: Reflectometry – (length scales from single molecule layer (< 1nm) to 1 μm)
4) Composition and speciation of molecules at buried surfaces– accessed via vibrational and other low-energy modes, energies < 15 meV to 30 meV

Sample Environment:

Emphasis on *in situ* measurements – environments where real operating conditions can be realized.

1) *in situ* multi-probe characterization – light scattering, optical spectroscopies, heat capacity, thermal conductivity
2) *in situ* hydrothermal synthesis
    a. Up to 67 bar (1000 psi) hydrostatic pressure
    b. Up to 500 °C
    c. Sample cell materials compatible with chemistry that can range from $2 < pH < 11$
    d. Capable of flowing materials
    e. Capable of mixing
3) *in situ* gas loading
    a. Control T and P – isotherm measurement, stable sample conditions
    b. $0.05\ K < T < 400\ K$
    c. P up to 100 bar
    d. Species – oxygen, nitrogen, carbon monoxide, hydrogen, methane, argon, ethylene, water, short alcohols, mixtures of above

New Instruments/Techniques:

1) High count rates enable
    a. kinetic measurements with SANS
    b. surface sensitive measurements
    c. polarized neutron studies
2) *in situ* prompt γ as a quantitative probe of hydrogen content – coupled with microfocused beams (Kirkpatrick-Baez mirrors can provide ≈ 100 μm spot size or less) would enable an innovative imaging technique (further development could incorporate a fast chopper and provide depth sensitivity)
3) *in situ* spin labeling – use NMR type manipulation of spins to highlight specific atoms – requires use of polarized neutrons and difference measurements

4) Polarized neutrons can be used to separate out incoherent from coherent scattering
    a. Differentiates self from collective diffusion

b. Differentiates coherent elastic substrate scattering from incoherent adsorbate signal
c. Separates phonons from incoherent quasielastic signal

5) Routine access to selective deuteration of small molecules

Suggestions:

Instruments could be optimized for high counting statistics, even at the cost of more modest instrument resolutions. In many cases, differentiation between molecular behavior at an interface vs. bulk relies on high statistical measurements. The trend in materials chemistry is towards more complex, multi-component, and multi-phase systems. Many of the topical areas above are in this category. An order of magnitude improvement in counting rate would significantly expand the range of problems investigated. This includes both new and existing instrumentation.

Exploration of additional means to differentiate molecular species could also be investigated. Selective deuteration of small molecules could be routinely accessible. More speculative techniques include the use of spin labeling via NMR inspired manipulation of nuclear spins. This application requires the use of polarized neutrons and instruments capable of high counting rates enabling difference measurements.

It is absolutely essential that a broad range of sample environment capabilities with *in situ* characterization be available. Many of the technologically interesting materials involve processing that produces significant changes in the sample that could be probed *in situ*. Samples will be produced, transformed, or even destroyed during the course of measurement. Routinely available *in situ* characterization could include optical spectroscopies (IR, Raman, UV), static and dynamic light scattering, heat capacity and calorimetry.

IIii. Hard Condensed Matter: Dynamics

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Since its inception, inelastic neutron scattering has played a crucial role in advancing our understanding of condensed matter and our ability to tailor materials properties for application. Though it is a mature technique, new instrumentation concepts continue to emerge with the potential to produce even more incisive data. The NIST expansion initiative is an opportunity to provide new capabilities to a broad range of users for maximal impact in science and technology.

The Hard Condensed Matter Dynamics Breakout Group identified a wide range of research areas where cold neutron spectroscopy is poised to make major contributions in the next decade. Transition metal oxides continue to challenge our understanding of correlated many body systems and provide new materials to fuel technological developments. Properties of current interest include high-T_c superconductivity, multiferroic behavior, and thermoelectricity. A particular challenge is associated with understanding interactions between charge, spin, lattice, and orbital degrees of freedom. Systems with novel ground states caused by geometrical frustration or by the formation of cooperative spin singlets provide unique laboratories for the exploration of the fundamental physics of many-body systems, and this exploration cannot proceed without the comprehensive information about dynamic spin, charge, and lattice correlations. Many of the systems described above, as well as exotic metals such as the heavy-fermion compounds, are thought to display quantum critical phenomena with low-energy dynamics that can be explored at the relevant time and length scale through cold neutron inelastic scattering. Probing the low-energy spectrum of the recently-discovered supersolid phase of helium has potential to shed light on a new phase of matter, and studies of single molecule magnets promise new insights into the fundamental physics of macroscopic quantum tunneling. Further, our understanding of a broad range of materials of technological importance, including thermoelectrics, relaxor ferroelectrics, and magnetic thin films can be greatly advanced through cold neutron spectroscopy. Hard condensed matter is a field that is driven by the continued discovery of new materials. So while it is precarious to predict even which classes of materials will be important in the coming decades, it is entirely reasonable to anticipate that cold neutron inelastic scattering will continue to play a central role. If the past is to serve as a guide, it is also sensible to anticipate that the introduction of new instrumentation capabilities to a larger user community will lead to advances both in fundamental and applied materials science.

We strongly endorse the proposal to develop world-class, high flux polarized neutron capabilities at the NCNR. All of the research areas enumerated above offer unique challenges and opportunities that can be exploited by advances in polarized neutron technology. Two types of polarized instruments appear particularly promising. First, a spin-polarized triple axis spectrometer equipped with the CRYOPAD technology would provide definitive information about complex spin correlations and excitation eigenvectors via spherical neutron polarimetry. Such an instrument could also be used for conventional polarization analysis without the CRYOPAD, for example, in cases where magnetic fields need to be applied to the sample.

Furthermore, such an instrument could be used to uniquely determine the magnetic structures of today's materials, which have become increasingly more complicated. Such a facility is currently not available in North America. A complementary approach is to develop polarization analysis capabilities on a fully optimized modern TAS using emerging large solid angle detection technologies. It would also be highly desirable to develop a cold triple axis spin echo spectrometer. Such an instrument can provide μeV energy resolution at ≈ 1 meV to10 meV energy transfer with good Q-resolution and Q-space flexibility, and can uniquely enable probes of quasiparticle lifetimes and interactions in solids. Other possibilities to be considered, include a super focusing spectrometer optimized to deliver high flux to small samples and thin films, and a dedicated high field spectrometer for fields in excess of 20 Tesla. This latter option probably cannot be developed on the relatively short timescale of the proposed upgrade initiative, but the possibility of implementing it in the future could be built into facility designs. It is strongly suggested that a sample-alignment station be included in the upgrade. The ability to align crystals without using precious beam time on a front-line instrument can greatly enhance the NCNR's scientific output.

Desired advances in sample environment capabilities were also considered. Specific needs were identified for extreme sample environments including high magnetic fields, pressure, and uniaxial stress, as well as improvements in experimental access for non-neutron probes of samples during scattering measurements, and the need for easier access to a wide temperature range. With the continued growth in complexity and expense of sample environment systems, particularly large cryomagnets, we strongly suggest that the NCNR move fully into a mode where adequately trained NCNR personnel operate the sample environment equipment and user involvement occurs through computerized access to the relevant thermodynamic variables. Investment in sufficient staffing for this mode is essential to prevent waste of beam time and to operate the advanced sample environment systems that are needed for cutting edge research.

IIiii. Hard Condensed Matter: Structure

Participants: Julie Borchers (NIST scribe), Tom Clinton, Cindi Dennis, David Lederman, Sara Majetich (chair), Paul Miceli, Jim Rhyne, Mohana Yithiraj

Nanoscale condensed matter materials will be at the forefront of important science in the next decade, motivated in part by technological applications. Magnetic, chemical and structural characterization on the nanoscale of nanostructures will be significant. For these systems, there is thus a need for nondestructive, non-perturbative characterization techniques, and neutron scattering is an ideal probe due to its sensitivity to buried interfaces, magnetism and light elements. In contrast to local probes such as microscopy neutron scattering provides ensemble averages of nanoscale systems. Neutrons are also one of the few direct probes of elementary excitations.

Of particular significance is the application of magnetic nanostructures for magnetic storage and sensor applications. The data storage density of hard disks has been on a continuous growth curve that even exceeds Moore's law (40 %), making it arguably the fastest moving technology in the high-tech industry. However, the rate of growth has begun to slow in recent years as magnetic recording is up against a fundamental problem known as the superparamagnetic limit. This occurs when bits of digital data become so small that ambient heat demagnetizes them, leading to loss of the stored data. To overcome this data integrity issue, new media materials are being developed with increased anisotropy for greater thermal stability. Traditionally, such media requires larger write fields, but, to circumvent the rising magnetic field requirements, the field required for recording data can be dynamically reduced using the novel approach of heat-assisted magnetic recording (HAMR), also known as "thermally-assisted" or "hybrid" recording. In this approach, optical energy provides a small, precise spot of heat at the disk during the writing of data, reducing the required field to a level accessible by the recording head. In the development of this technology, this novel approach to recording requires new materials for the media, recording head, disk-head interface, and other integrated components, as well as better understanding of the physics governing the recording process in the very different thermal environment from traditional recording techniques.

There are a number of other materials and phenomena of current interest. Multi-functional materials, such as multiferroics, have potential applications as sensors and actuators and could underlie an alternative storage technology. Spintronic materials, including magnetic semiconductors and half-metallic ferromagnets, are dependent upon the exact conditions of their interface for operation. Understanding of these interfaces is crucial for the design of effective devices, as well as for comprehension of the fundamental science and interactions between layers. Furthermore, these materials have interesting and complex interactions with electric, magnetic and strain fields that can be probed with neutrons. In addition, neutron scattering can most comprehensively examine the flux lattice phase diagram and possible excitations in superconductors. Hydride materials such as clathrates are of interest for hydrogen storage, and neutrons are uniquely suited for examining their structure and dynamics.

Patterned and self-assembled structures, including nanoparticles, nanowires, nanocomposites, etc., are used as structural materials and catalysts, among other applications.

There are two fundamental length scales of interest: structural and magnetic. In core-shell structures and multilayer materials, there are issues of interface structural and magnetic couplings. The separate intra- and inter-particle correlation lengths that exist in these materials may also exist in the inorganic-biomaterial hybrids, which have potential applications in drug delivery, cancer treatment, and catalysts.

For nanostructured materials, the primary issues are nanoscale characterization

1. to probe length scales from 0.5 nm to 1000 nm in all three dimensions,
2. to determine 3-D magnetic correlation lengths and domains with ensemble averaging,
3. to examine quantum size, finite size, confinement, and interface effects,
4. to explore the thermal stability and relaxation at moderate temperatures using time-sliced or pump-probe techniques
5. to study elementary excitations and lifetime effects in nanostructured systems.

In particular, the fifth item would potentially have a huge impact because theoretically these materials could have fundamentally different dynamics than bulk materials. We want to focus on low-Q experiments that probe the interparticle interactions as well as higher-Q experiments that would also examine the intraparticle interactions.

Other issues include structural, magnetic, and superconducting phase transitions; magnetic coupling at interfaces; magnetic versus structural boundaries; wetting, self-organization and thin film growth phenomena; and the question of dimensionality cross-over to two dimensional behavior will be critical to the search for a minimum usable particle size.

In designing appropriate neutron instrumentation, flexibility in sample geometry and instrumental configuration is required. The primary objectives are separation of magnetism from nuclear structure in nanomaterials, energy dispersion measurements with depth resolution for the nanoscale (low Q – High E), and separation of underlayer and interface signals from bulk. Most experiments will require highly efficient polarized beams (> 98 %), enhanced flux (via focusing neutron optics, monochromator array, or Larmor labeling), small beam size for small sampling size (0.3 mm × 2 mm or less), low background, and inelastic scattering capability for nanoscale materials and small sample sizes. To perform the experiments of interest, a variety of sample environments are of interest, such as:

1. Sample chamber with pulse/probe capabilities (optical, magnetic, electronic) for dynamics
2. Multi-directional magnetic fields
3. High T (1000 K) as well as low, with fast heating and cooling
4. Ability to make *in situ* magnetic, electrical measurements
5. Uniaxial and hydrostatic pressure
6. Concurrent electric and magnetic fields

Suggestions for new instrument development include three items, all with polarized beam capability. First, a hybrid reflectometer/SANS is proposed to probe length scales in all three dimensions in nanostructured materials. A possible design to consider is the sSANS (subsurface Small Angle Neutron Scattering), which uses neutron spin echo techniques to resolve nanometer length scales in the third dimension. Second, a grazing angle with 3-D resolution (GISANS)

with small spot size is proposed for laminar materials. Capabilities of this instrument could include a broad-band incident beam with a focused 0.3 mm beam and angular divergence that integrates over the reflectivity rod to enhance the intensity in a grazing incident geometry. Diffraction could be measured in the sample plane and for this an in-plane beam size on the order of 2 mm would be necessary. Larmor labeling could be explored as a possible way to deal with the angular divergence generated in creating the small 2mm in-plane beam size. Third, a triple-axis instrument optimized for inelastic scattering in thin films and nanoparticles is proposed. Flexibility in the triple-axis instrument design could allow for a variety of detection and analysis schemes, such as wide-angle detection and supermirror analyzer array.

IIiv. Instrumentation: Imaging and Detectors

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Cold Neutron Imaging

The NCNR expansion presents an exciting opportunity for cold neutron imaging, and therefore a cold neutron imaging facility will offer unique measurement capabilities for both basic science and new technology development which will greatly augment the existing thermal neutron imaging facility in its support of American industry. Water and its spatial distribution plays a central role in biological systems such as membranes and microfluidic devices, as well as for many physical systems of great practical importance such as hydrogen fuel cells. Cold neutron imaging coupled with new imaging detectors will enable us to improve both the measurement sensitivity and spatial resolution of water distribution by orders of magnitude in both biological and physical systems, and for example, this will open the opportunity to study the fundamental water transport mechanisms in geological samples, biological systems, and hydrogen fuel cells. Additionally, with increased penetration and enhanced phase sensitivity, cold neutron imaging would enable high turnover non-destructive evaluation (crack and defect detection, residual stress) of mechanical components to improve manufacturing techniques.

The reasons for this lie in the combination of the physical properties of cold neutrons, their interaction with matter, new developments in imaging and counting detectors and the emergence of new imaging modalities such as phase contrast imaging which make use of the wave properties of neutrons.

- The total scattering cross-section for water increases by a factor of about 3 for cold versus 0.1 nm neutrons, while that of Al, and C and other materials remains nearly constant. Further, since cold neutrons are above the Bragg cutoff for many metals for industrial use, cold neutrons will have greater penetration into metallic matrices. This results in a 2 order of magnitude increased sensitivity to water.
- Phase effects are proportional to the neutron wavelength, yielding an increase of about 3 going from thermal to cold, and so there is about an order of magnitude reduction in image acquisition time.
- The proposed cold source's predicted fluence rate of 4×10^9 cm^{-2} s^{-1} coupled with high its high brightness and low divergence is well suited to the needs of neutron imaging.
- Current detector systems will have a near doubling of detection efficiency, which will result in improved spatial resolution of current scintillator technology.
- Differential Radiography, employing a velocity selector in the beam path will provide the ability to scan above and below Bragg edge to gain compositional sensitivity, both in distinguishing water from ice as well different metals.
- With increased sensitivity to water as well as a quieter radiation environment reducing the radiation damage, cold neutron imaging will have find more biological applications looking at membranes, cells with antibodies tagged with high contrast agents and calcification.
- In order to maximize signal to noise in high resolution (0.01 mm pixel pitch) neutron images

will require high detection efficiency (near 100 %) to maximize neutrons/pixel, which, with large numbers of pixels will result in high global counting rates (10 MHz to 100 MHz). Further, cold neutrons will allow the use of thinner detection medium reducing the gamma background which serves to improve signal to noise.

- To employ and develop new imaging modalities (phase imaging with gratings, coded source imaging, differential radiography) a new neutron imaging facility will require ease of access to the aperture system, a flexible and long (at least 10 m) flight path downstream of the aperture. In order to accommodate large objects, a 4 m to 6 m width about the beam center-line would be required. Floor space will required for ancillary equipment, such as a fuel cell stand. High resolution images will require massive data storage (10 TB to 100 TB per year) and user access to data analysis tools.
- With current resolution capabilities, the improved sensitivity will enable evaluation of casting defects in primary metals, investigation of precipitates in alloy formation, rapid measurement of residual stress, crack detection, investigation of crack and pinhole formation in hydrogen fuel cell, detection of de-lamination in aerospace composites, measurement of polymer film density, two phase flow in heat pipes, and measuring the lubricant flow and fuel spray in aluminum and steel internal combustion engines.
- Improved detector resolution (0.01 mm) coupled with measurement sensitivity will enable detailed study of hydrogenous transport in porous media (geology, fuel cells, microfluidics), biological systems (bone, calcification, tagging antibodies with contrast agents), magnetic domains (via phase imaging).

IIv. Instrumentation: Instrument Concepts

Participants: R Gähler, M. Rowe (chair), L. Robertson, J. Copley, L. Passell, G. Ehlers, D. Pierce, D. Mildner (NIST scribe), I. Zaliznyak

Suggestions for technologies to be developed, with priorities;

The construction of the 2nd guide hall at NIST would start in FY 2007. This does not leave time to develop new technologies or to develop existing technologies further, except for those where NIST already has strong competences. We propose as actions:

1. A majority of instruments for the new guide hall will likely use polarized neutrons. Polarizing guides might be a good choice in this case. European experience shows that the technique still needs R&D on the polarizing mirrors. As NIST has no strong competence in this area, we consider these guides as risky at present.
2. At present, polarisation techniques for cold neutrons rely to a large extent on polarizing benders. Those can be purchased on the market; in-house developments in near future are not suggested. For the longer term, NIST and the other US facilities could pursue R&D of some of the basic issues involved in super-mirrors and polarising mirrors.
3. The neutron centers could promote the development of m = 2 supermirrors of higher reflectivity. The use of m > 2 super mirrors / guides could be considered with great care.
4. The SEOP (Spin Exchange Optical Pumping in order to polarize neutrons) * method for beam polarization, where NIST has a pioneering role, could be followed with high priority. We expect that SEOP will develop towards a standard method for neutron beam polarization, except for special cases such as very high polarization (> 98 %), narrow beams, and hot neutrons.
5. A number of applications exist in which high beam polarization is desirable (> 99 %). Such beams open new opportunities for instrument design. For example, high polarization beams can use spin manipulation to condition the incoming beam (time structure).
6. In case NIST decides to build a NRSE spectrometer, we suggest that NIST joins the envisaged collaboration (SNS, UNC, ANL, ILL) on new high performance spin flippers.
7. NIST could develop together with ILL-Grenoble a new design of neutron guides, implementing state-of-the-art technologies, alignment methods and control systems. Both institutes plan to install several hundred meters of new guides in the next years.
8. Cold neutrons are amenable to a variety of focussing techniques using both crystal and super-mirror reflection. Focussing techniques could be carefully optimized for the application in mind.
9. We are concerned that the choice of velocity selectors may become more limited in future.

Suggestions for instruments for 2nd guide hall;

In general:

1. The instruments for the new guide hall could be optimized for the new H_2-cold source of very small size (∅ ~ 8 cm). This favours instruments using beams with smaller cross section and narrow divergence. Instruments using large area focussing monochromators or large area beams with significant divergence could be considered for the reconfigured cold source in the existing guide hall.
2. In case instruments from the present guide hall may be moved to the new guide hall, beam positions with large guide heights will become available, which broadens the spectrum of possible new instruments.
3. The existence of two guide halls will make it possible to separate high field magnets from instruments sensitive to stray fields.

The following instruments (no particular order implied) look favourable for the new guide hall:

1. A long SANS instrument with polarization option and perhaps further options (VSANS, GISANS, SANS tomography).
2. A reflectometer: most likely with vertical sample geometry (perhaps with SERGIS option). It may focus on a large dynamic range (low background) and complement SNS instruments.
3. A station for fundamental physics will profit from a full beam end position, a large experimental area, and low background.
4. A low-energy polarized beam inelastic spectrometer, which could also be used in 2 axis mode (diffractometer for diffuse scattering). This instrument could be of D7 type (time-of-flight)) or use large area crystal analysers.
5. A zero field spin echo with wide angle analysis and/or conventional wide angle spin echo (SPAN type).
6. A LADI-type diffractometer for cold neutrons, equipped with scintillator-based detectors. It will benefit from recent advances in scintillator technology at SNS.
7. A polarized powder diffractometer for magnetic materials, working in the range of 3 Å to 4 Å.
8. Two versatile test stations for neutron optics, crystal testing/aligning and n-detector development. In contrast to all other proposed instruments, these stations do not need end positions.

*SEOP lends itself to continuous optical pumping to maintain the ^{3}He polarization at a constant value. This makes it possible to use ^{3}He cells as beam polarizers and beam polarization conditioners. It also makes it much more convenient to employ ^{3}He cells as wide-angle polarization analyzers. And of equal (or perhaps greater) importance, SEOP is much less expensive to implement than MEOP.

IIvi. Instrumentation: Polarized Beam Methods and Optical Devices

Participants: John Barker, Mike Fitzsimmons (chair), Wangchun Chen, Jeremy Cook (NIST scribe), Tom Gentile, Thomas Krist, Seung-Hun Lee, Christian Schanzer, and Hirohiko Shimizu

Suggestions

- There are many of attractive options to polarize cold neutron beams, but the best option depends upon which spectrometer(s) will use the guide. Optimization of the facility portfolio requires integration of source-guide-instrument designs.
- NCNR could consider assuming responsibility for providing several instruments with access to polarized neutron beams by investing in polarizing guides.
- The ILL (and others) has pioneered a variety of interesting technologies that are boot-strapped onto existing instrumentation. NCNR has an opportunity to exceed the ILL by optimizing spectrometers to the most interesting technologies.
- Make effective use of the space between the guide and sample to focus the neutron beam, etc. Investment made in this space varies with linear foot, rather than with volume in the space between the sample and detector, and thus is well leveraged.
- Invest in devices that better optimize $d\lambda/\lambda$ to $d\theta/\theta$ (or $\cot\theta\, d\theta$), e.g., synthetic monochromators. Instrument performance will scale linearly with investment here.
- Invest in beam splitting options to maximize end-guide positions. More end-guide positions enable optimization of $d\lambda/\lambda$ to $d\theta/\theta$ using velocity selectors to instruments other than SANS machines.
- If NCNR invests in polarizing guides, then NCNR could specify non-magnetic supermirror coatings for any unpolarizing guide surfaces from which reflection of a polarized neutron beam may occur.

Polarization of the incident neutron beam.

There exist several options to polarize the incident neutron beam, including polarizing guides, cavities, and beam benders and beam splitters (the latter retain both spin states). Obtaining a beam polarization $P \approx 95$ % is straightforward for a 4 cm wide by 6 cm guide (typical size for the new hall and one that is not particularly large). Guides and beam benders could achieve 95 % beam polarization for beams having a divergence equivalent to $m = 2.5$ in the horizontal plane. The divergence of the beams in the vertical plane will likely be of order $m = 2.5$ achieved with Ni-Mo/Ti unpolarizing supermirror coatings on the horizontal guide surfaces. N.b. it is essential that a non-magnetic alternative to Ni/Ti unpolarizing supermirror coatings, e.g., $Ni_{93}Mo_7$/Ti, be used in polarizing beam guides so as to avoid depolarization of the neutron beam through reflection from unpolarizing surfaces. Alternatively, a polarization cavity using a 5 m to 8 m long $m = 4$ polarizing supermirror can provide *spin up and spin down* polarized beams with divergence of $m = 2$ in the horizontal plane. Ideally, these beams would travel down separate polarizing guides and become even better polarized as they separate with distance from the cavity. Finally, schemes exist that can produce polarized neutron beams of very large cross-section having the divergence of $m = 1$ when viewing an unpolarized guide (of similar m-value).

Guide coatings containing Co may pose a radiation hazard. A coating of an FeCoV alloy can be particularly soft requiring only fields of 20 Oe or so to maintain their magnetization.

Polarization analysis

Polarization analysis involves measurement of the component of the polarization vector along the axis of the magnetization of the analyzer. In the simplest case, only non-spin-flip and spin-flip cross-sections are measured for a beam with polarization parallel to the field applied to the sample. Such measurements distinguish between coherent and incoherent scattering (particularly important in materials possessing hydrogen), or to distinguish between nuclear and magnetic scattering. In Europe, the constraint to have the polarization of the beam parallel to the applied field is relaxed allowing measurement of the complete three-dimensional spin dependent scattering (generalized polarimetry).

Polarization analysis with supermirrors

Since polarizing supermirrors function in a limited range of wavevector transfer, the ranges of angle and wavelength of the neutron beam incident on the mirror must be limited; thus, effective polarization is only possible if the neutrons originate from a point or line source. There are, however, many options that can be tailored to specific needs. Supermirror analyzers are not susceptible to stray magnetic fields.

1. One or multiple (radial) cavities

A cavity can analyze in one dimension a divergence of $\pm 0.1° \lambda$ (Å) to $0.15° \lambda$ (Å). In the other dimension the walls are either non-reflecting, as in multiple cavities, or can be coated for a cavity with a single or V-shaped line of wafers. If the divergence is larger, then the excess divergence is absorbed in the analyzer and does not deteriorate the polarization.

The system must be chosen according to the divergence distribution in the incoming beam:

For large divergence in both directions cavities are arranged radially to cover an angular range up to 5° or more. In this case the cavity walls must be absorbing and thus reduce the transmitted intensity by a factor of two. Also the shadows of the glass walls are in the direction under consideration. The shadows only reduce the intensity of the scattered beam but do not affect its polarization.

If the incoming beam has in one dimension a divergence in the order of $\pm 0.1° \lambda$ (Å) a multiple cavity with parallel reflecting walls can be used which has a higher transmission and no shadows in the direction under consideration.

Various designs are available according to the experimental requirements.

Most advantageous arrangement is with the supermirror wafers parallel to the direction under consideration (no unique information in the shadow area).
References (**ILL**: ADAM, SERGIS, **IPNS**: POSY II)

2. Solid state devices

Radial bender, 5 cm to 7 cm long, large 20° divergence in one of two dimensions and 10° or more in the other dimension, 2x cost of cavity design, deflects the beam, slight intensity fluctuations in the radial direction.

Bender+collimator, large 20° divergence in one of two dimensions and m=3 in the other dimension, can use the non-deflected beam.

References: first system **HMI**: prototype tested, second system **JRM3**: SANS

3. Arrays of supermirrors

Wide-angle analysis presently exists for instruments at the ILL and consists of very large arrays of supermirrors, e.g., 30 cm by 30 cm, etc.

References: **ILL**: IN15, D7

Polarization analysis with ^{3}He spin filters

New instruments will need polarization analyzers that can accept highly divergent beams. Spin filters based on the large spin dependence of the cross section for absorption of neutrons by ^{3}He gas can address this issue and have additional features. The current maximum ^{3}He polarization is presently 75 % for practical spin filters, which yields 90 % neutron polarization with 28 % absolute transmission of neutrons. The key technical issues in incorporating ^{3}He spin filters include the requirement of sufficient space after the sample (about 30 cm), and a uniform magnetic field environment in this space. The key scientific issues include the development of experiments and experience that will make use of this technology. ^{3}He spin filters require magnetic field gradients of not more than 10^{-4} Oe cm^{-1} to 10^{-3} Oe cm^{-1}, which is in direct conflict with experiments that require high field superconducting magnets, hence the technology to shield such fields needs to be developed for each situation. Further development is required to actually make the large variety of spin filters that will be required. The spin-exchange-optically-pumped (SEOP) method is compact and can be operated continuously on a beam line, if necessary, but instruments must be designed and/or adapted to allow for this option by including additional space next to the device. An alternative approach is a "filling station" approach, which decouples the optical pumping requirements from the spin filter requirements.

Elliptic guides

In order to account for the demands of neutron optic components providing higher flux, an elliptic guide is proposed, which enables i) extraction of a large phase space out of the source, ii) reduction in the loss of neutrons and iii) focused neutron beams. Simulations have demonstrated their performance is superior to conventional neutron guides[1]. The elliptic guide provides benefits of:

- gain in intensity (focused on small spots, e.g., of the size of the sample)
- homogeneity of the delivered phase space
- spot of highest intensity is beyond the end of the guide – conveniently accessible for further handling, e.g., choppers can be placed at the focus
- cold source can be kept small
- optimized variation of supermirror coating enables to confine the costs

Issues regarding manufacturing precision and high m-value supermirrors (necessary only for rather short sections) are solved, e.g., it is now possible to manufacture supermirrors with m = 5 with excellent reflectivity over large areas. The concept of an elliptic guide has been experimentally realized using a miniaturized prototype[2].

An application of elliptic neutron guides is anticipated for:

- a virtual source, e.g., for triple axis spectrometer
- an alternative for linearly tapered ballistic guides
- transform spatially compact beams between choppers in ToF instrumentation
- combination of elliptic and curved guides

In order to test the anticipated scenarios, careful simulations have to be conducted (as is true for any guide system), in particular to combine the benefits of elliptic and traditional guides.

[1] C. Schanzer, P. Böni, U. Filges, T. Hils, "Advanced geometries for ballistic neutron guides", Nucl. Instr. and Meth. A **529** (2004) 63–68

[2] S. Mühlbauer, M. Stadlbauer, P. Böni, C. Schanzer, J. Stahn, and U. Filges, "Performance of an Elliptically Tapered Neutron Guide", Physica Bin press (2006).

Magnetic lenses—opportunities for polarizing or focusing neutron beams

Magnetic lens offer opportunities to polarize or focus neutron beams without introducing material into the neutron beam that might otherwise enhance the instrumental background through nuclear scattering. These devices consist of permanent magnet multipoles and thus require no maintenance. The cost is about $100 k per meter. Variable permanent multipoles are under development.

A quadrupole magnet is suitable for filtering neutron spins according to their spin. A 10m long quadrupole with a 3 cm diameter aperture is capable of polarizing 4 Å wavelength neutron beam to in excess of 99.99 %. It also functions as a loss-less neutron guide of m = 0.5. The polarization increases with neutron wavelength. The length of device inversely proportional to neutron wavelength and aperture diameter. The stray field at a distance of 0.1m from the end of the magnet axis or 0.4m radially from its axis is equivalent to the Earth's magnetic field.

A sextupole magnet is suitable for point-to-point focusing of a polarized neutron beam. A 0.6 m long sextupole focuses 6 Å wavelength neutron beam with a focal length of 10 m. Neutron polarization of 99 % or larger is necessary to suppress the background level below 10^{-6} of the direct beam. The background consists of neutrons with spin opposite to the majority spin and widely spread about the forward scattering direction.

References

H.M.Shimizu et al., Physica B (in press as a proc. of ICNS2005)

T.Oku et al, Physica B (in press as a proc. of ICNS2005)

Focusing using Elliptically Shaped Mirrors

Advantages:

- Achromatic: focal length independent of wavelength.
- Can greatly reduce the wavelength dependent distortion of the beam by gravity by reflecting in the vertical direction.
- Existing SANS instrument KSW-3 at Julich can be used as reference of capabilities (Kentzinger E., Dohman L., Alefeld B., Rucker U., Stellbrink J., Ioffee A., Richter D. and Bruckel T. (2004) Physica B **350** e779-e784.

Disadvantages:

- Expensive: > $100 K. For best performance, requires extremely smooth substrate and Cu or Ni single coating.
- To allow 1 cm sample size with 6 Å neutron wavelength requires mirror length of approximately 1 m. Divergence accepted limited by the critical angle of coating.
- Nonspecular scattering can produce significant background even from smoothest mirrors having the best figure control from existing technology.

Opportunity afforded by highly intense polarized neutron beams

For enhancing the capability of neutron scattering, we wish to note that methods for polarizing proton spins are at hand that can be probed with intense polarized neutron beams. For example, site-specific spin contrast variation out of thermal-equilibrium using dynamic nuclear polarization or microwave-induced optical nuclear polarization, increasing the neutron utilization efficacy by realizing superparallel branch together with compromised q-resolution focusing SANS, point-like neutron source with a very strong bending optics to deliver more neutrons into a tiny aperture.

IIvii. Instrumentation: Sample Environments

Participants: Andrew Allen, Shenda Baker (chair), Dan Dender, Joe Dura, Evan Fitzgerald, John Katsaras, Alamgir Karim, Juscelino Leao (NIST scribe), Matthew Liberatore, Sarah McKenney

Overview

Sample environments are a crucial aspect of any experimental setup. *In-situ* processing and/or real-time analysis of sample physical properties are keys to a successful neutron scattering experiment. Our premise is that the study of materials by neutrons can be enhanced by the use of complementary techniques that can give corroborating data with regards to sample properties and that NIST will have a unique presence in the neutron scattering community if it can provide these tools simultaneously with the neutron experiments in the beam. Our assumptions in making our suggestions are 1) extremes in environments will not be addressed here but may be addressed in field specific sections 2) each instrument scientist and user will have to assess the trade offs in intensity, resolution and background given the addition of sample environments and 3) innovation could be supported by NIST, but the biggest impact in sample environments will be in establishing capabilities that attract large numbers of users for reliable, complementary and plug-in capabilities.

The critical capability lacking in the *in situ* sample environments available at neutron scattering facilities is the ability to easily achieve combinations of sample environments. While this desired capability includes control over multiple thermodynamic variables such as temperature and magnetic field, the scientific need is broader by also including additional measurement opportunities, either *in situ* or in an on-site laboratory. Spanning all disciplines from biology to hard condensed matter, from structure determination to dynamics, is the desire to control not one but multiple relevant thermodynamic variables (temperature, pressure, humidity, electric field, magnetic field, stress/strain) and to combine them with complementary measurements (UV/optical/Raman spectroscopy, thermal conductivity, calorimetry). Instead of concentrating on extreme environments (e.g., 25 T to 40 T magnet, 100 GPa pressure cells, etc.), we think that there exists a much more basic need in the community. At present, anything more than basic sample environment setups is difficult to accomplish at NCNR. By developing the capability and reputation for combination environments, NCNR will attract users. Currently, users try to mimic the thermodynamic conditions from their laboratory measurements with the samples they bring to NCNR. Reproducing these measurements is difficult due to different measuring devices, hysteresis in the sample, or the need to include additional tools and techniques not available at NCNR.

We have considered many physical measurements and have described most of these suggestions in the remainder of this report. Of these ideas, we believe that the greatest scientific impact can be achieved by concentrating on providing a core foundation in the following six areas:

1. Highly uniform temperature control across the entire sample, real time control and measurement
2. Expanded offerings for pressure on the sample, both hydrostatic high pressure and gas-loading into the sample, real time control and measurement

3. Humidity control
4. Calorimetry
5. Spectroscopic techniques via fiber optics (UV/Vis, FTIR, interferometry, DLS, raman, etc.)
6. Optical examination of the samples via mini-CCD camera.

It must also be noted that sample environments do not "operate in a vacuum." Extensive interplay and tradeoffs exist between the sample environments and other objects such as the instruments, sample preparation and characterization facilities. Sample environments could keep these considerations in mind when being designed.

Effective partnering of the facility and users can leverage the capabilities of both. While some general use sample environments could be developed and supported solely by the facility, other more specialized environments could be developed in partnerships with users, or via facility support in the form of either technical or financial assistance. Supporting innovation through collaborations with users has been a trademark of NIST and could be fostered. Although many of the most extreme regimes of high T, high P and high magnetic field are not specifically addressed here, NIST could maintain well supported staff and instrumentation for access to routine sample environments addressed below.

Many of the suggestions enumerated in this report are neither complicated or new, however they are often not realized due to the lack of resources such as staffing. This breakout groups recognizes that the advances in throughput and in new capabilities suggested here will not be realized without significant additional support for personnel, equipment and floor space.

Rheology

The combination of SANS and shear flow has advanced the fields of soft matter and rheology over the last decade. These measurements need to interrogate the length scales important in the complex fluids of interest ranging from 1 nm (about the radius of a wormlike micelle) to 1 μm (the size of concentration fluctuations in polymer solutions). Current, available technology allows simultaneous rheological measurement (with a commercial rheometer) and SANS in a Couette cell in the flow-vorticity (1-3) and gradient-vorticity (2-3) planes of the flow. Rheo-SANS facilities need to be expanded to include cells to probe the flow-gradient (1-2) plane of flow with gap resolution, oscillatory shear flow, and elongational flows. Scattering with light and x-rays in these flow environments is common and thus, a need to be probe samples with neutrons expands understanding to smaller length scales. The ability to resolve alignment and structure with SANS in all three planes of shear flow allows three-dimensional representations of the microstructure to be formulated. The cells would need to be easy to load, clean, and control temperature with 0.5 ^{o}C sensitivity over a temperature range of 0 ^{o}C to 100 ^{o}C. Gap resolution investigates phenomena including shear banding and slip at the wall and necessitates a high flux to keep measurement times reasonable. Planar shear cells can work in NR geometry and can address very high shear rates > 10^5 /s but only with 1-D information.

Microfluidics Platform with Controlled Environment

The combination of Neutron Reflection (NR) and Small Angle Neutron Scattering (SANS) Neutron can be a powerful combination of characterization tools for interrogating the self-assembly, hierarchical structure and molecular level information within the microfluidic and millifluidic environments, both under flow and under static conditions. NR will provide the average information of fluid composition as a function of distance from the walls in the 10 nm to 100 nm range of the confined flowing microfluidic solution. This is expected to experience a varying shear field as a function of distance from the walls, so a variation of components distribution can be expected. This information can be complemented by SANS that will provide the in-plane distribution of composition also with (1 to 100) nm spatial dimensions. Together, NR and SANS will provide 3-D information of fluids in micro and milli-fluidic environments. The ability to couple effects of pressure and temperature to these on-line devices will be very valuable for a variety of biological and soft matter problems. On-line spectroscopic characterization tools such as UV-VIS and raman and optical microscopy will be especially useful.

Liquids

For horizontal geometry, neutron reflectivity (NR), a Langmuir trough, preferably small so equilibration times can be accelerated, is essential. The addition of the deposition mechanism (Langmuir-Blodgett) allows for the series examination of the film on the liquid and on the solid surfaces. For all cells, interchangeable fluid management (pH, solvent, rinsing, introduction of adsorbing material, etc) is needed in real time. To a lesser extent, control of vapor pressure above the surface is desirable.

Biology

The study of biologically relevant materials under difficult environmental conditions (such as high magnetic fields, high pressures, shear, and 100 % relative humidity) is by no means straight-forward and requires specialized equipment. Absolute control of humidity is imperative. Cell could be capable of automated humidity change, and provide fine control changes in RH (0-100 % RH). Hydrostatic cells to 5 kbar are needed. These could be of two types: (1) copper/beryllium or titanium/zirconium cells for membrane studies and (2) sapphire window cells for SANS studies of protein and other solutions. Horizontal and vertical field magnetic fields, up to ≈ 10 T, and suitable for sample environment temperatures between ≈ 0 ^{o}C and 100 ^{o}C are needed. Impedence measurements for use with neutron reflectometry (NR) are currently being worked on by the Lösche group. A rheometer for studying molecular structure under shear flow, which is induced between two concentric quartz cylinders with outer one rotating around the inner stationary one (Couette flow) or planar geometry for NR (Pouiseuille flow)

Solid Samples

Solids can be examined in bulk or as substrates for small molecules. Given the breadth of sample environment needs, a few priorities have emerged. Sample temperatures need to be controlled to sub degree accuracy and uniformity with rapid heating and cooling and coupled with the ability

to trigger time sequences of data acquisition. Gas handling capabilities could include control of partial pressures of various species including water and D_2O vapor from 0 % to 100 % relative humidity. *In-situ* analytical techniques such as FTIR, Raman and UV spectroscopy, calorimetry, thermal and electrical conductivity measurements could be made available as well as the ability to perform *in-situ* sample modifications and/or preparations. MBE may be a very useful tool to have in a NR beam which combines exquisite control of surface preparation with angstrom precision.

Routine access to a variety of pressure cells, temperature control and magnetic fields applicable to hard condensed matter experiments needs to be developed and maintained. Easy access to fields in the range of 7 T to 10 T could be provided, and the ability to attain fields up to 17 T is now essential for a world class program in magnetism. The ability to orient magnetic fields is highly desirable. Pressures of up to 10 kbar hydrostatic and up to 30 kbar with clamp cells could be made available and easily accessed with temperature ranges down to 0.3 K. Ready access to temperatures as low as 0.03 K is also important. Cryo-furnace from 1 K to 800 K and uniaxial stress to 10 kbar could also be made available. This level of user support and performance can only be achieved by appropriate levels of staffing.

Instrumentation

The facility's infrastructure is presumed to have ample sample preparatory laboratory space, adequate facility chilled and hot water, gas, and power. Moreover, these could all have a standardized interface. Some important requirements for individual instruments are discussed below.

The facility power supply could be available in diverse "flavors" providing consistent current capability and stability including European and North American standard. Ease of access to the facility's infrastructure including nitrogen, helium, and compressed air, as well as electric and chilled water by the user at each of the instruments could be standardized.

Built in permanent vibration isolation is imperative for a horizontal reflectometer and needs to be incorporated from the slab up. A sample mount table, designed and tailored to each instrument, will provide a more uniform mounting procedure as well as appropriate free space to accommodate a sample along with its sample environment equipment. Standard sample holders could be developed for samples either between neutron scattering instruments or neutron and other analytical techniques (x-rays!).

The process of sample alignment could be done easily and rapidly. Incorporating laser sample alignment in the instrument provides a quick and simple visual approximation. Rapid turn-around, alignment and equilibration of samples will enable faster processing. The ability to scan the sample in 2-D across a very thin beam with knowledge of beam placement is important.

Remote viewing and access of certain sample environment parameters will allow for safe neutron experiment monitoring. The advent of remote viewing and control of parameters such as sample temperature, concentration, and flow among others provide for monitoring of the experiment during difficult hours of operation, which works in conjunction with A.L.A.R.A.

The availability to a clean instrument sample containment area with appropriate ventilation of solvents and gases will increase the quality of results. Many of molecular samples and thin films require a clean room environment with HEPA filter ventilation.

Associated Laboratories

Appropriately equipped, staffed and located laboratories are essential to support the neutron scattering experiments. Currently, insufficient space exists for the number of users at NCNR. New space could be as close to the guide hall as possible to assure easy access and facilitate transfer of sensitive samples to and from the beam lines. New labs could support chemistry and molecular biology and provide clean room space (class 10,000). These labs could include a comprehensive suite of instruments that may include UV/Vis spectroscopy, FTIR, SAXS, AFM, DSC, dynamic light scattering, static light scattering, optical and polarized microscopes with temperature control stages. More general lab equipment could include reasonable numbers of minicentrifuges, glove boxes, spin coaters, fume hoods, balances, CO_2 cleaner and oven capable of vacuum and multiple gas atmospheres. NCNR could supply substrates for NR that have the reference layer for both polarized and unpolarized beams. Samples as prepared, shipped or taken from the beamline need to have appropriate storage for short or longer term. These environments include controlled temperature and/or humidity and appropriately address safety issues.

NCNR staff must be available in appropriate numbers to see an experiment through from start to finish. The technical expertise behind an instrument makes it accessible to an outside user and useful and accessible laboratories facilitate the preparation of high quality, reproducible samples.

Computing Interface

For all instruments, both instrument and environment parameters need to be monitored. An active log of the instrument parameters must be maintained and include sample environment parameters (*i.e.*, temperature, partial pressure of gases, pH, flow rate, etc) that are date and time stamped. This logging could be done in a modular format so that particular data can be selected without interruption of the other data streaming and can be presented to an external user by placing an image on a webpage (common practice at APS and ISIS).

A modular capability to log parameters allows for feedback to the instrument of sample environment controls and requires a uniform interface for all types of instruments. Wireless addressing of the sample equipment will minimize lines into the beamlines and provide remote access. Assessment of neutron data in real time could be available in order to make adjustments to environmental parameters. For example, in monitoring pressure or temperature for a phase transition, a data profile change may be used to induce a reduction in temperature/pressure increase so that more data can be collected around the phase transition point. Feedback algorithms will need to be written by NCNR staff or experienced users, but the system needs to be designed to allow modular control to be set up with minimal lead times.

All sample environment parameters need to be controllable in real time remotely. Debate still exists as to the extent to what level of *instrument* optimization during the experiment from off-site should be or could be allowed.

IIviii. Neutron Physics

Participants: David Bowman, Timothy Chupp, David Cory, Thomas Dombeck, Brian Fisher, Geoff Greene, Paul Huffman, Pieter Mumm, Jeff Nico (NIST scribe), Dmitry Pushin, Mike Snow, Fred Weitfeldt (chair)

Scientific opportunities in neutron physics using cold neutrons at the NCNR over the next decade:

Precision measurements of basic neutron properties allow us to search for violations of fundamental symmetries and to make critical tests of the validity of the Standard Model of particle interactions. This includes measurements of neutron beta decay parameters (lifetime, angular coefficients), studies of the weak interaction in the hadronic sector (capture asymmetry, spin rotation), and searches for the neutron electric dipole moment. Neutron interferometery allows for precise determination of the phase and coherence of neutron waves. This enables precision measurements of basic nuclear interactions and basic investigations of quantum behavior and information.

- Neutron decay is a relatively simple system, so the angular correlations between the momenta of neutron decay particles (electron, proton, and antineutrino) and/or the neutron spin can be related to basic parameters in the underlying theory such as the weak axial vector coupling constant g_A and the quark mixing parameter V_{ud}. Furthermore they test the self-consistency of the Standard Model of particle physics and could find evidence of new physical forces. It is very important to improve the precision of these correlations to the 0.1 % level in next-generation experiments. One correlations (the D coefficient) violates time-reversal symmetry and may help explain the matter-antimatter asymmetry of the universe.

- The neutron decay lifetime helps test the Standard Model and is a key input parameter for theoretical calculations of abundances of light elements produced by the Big Bang. There is currently a six standard deviation disagreement between the most recent ultracold neutron lifetime experiments performed at the ILL. Two NCNR experiments, one using the cold beam method and the other using magnetically trapped ultracold neutrons, will help resolve this discrepancy and improve the precision in the neutron lifetime.

- The neutron has a rare decay mode in which a gamma ray is emitted along with the electron, proton, and antineutrino. This process, known as radiative neutron decay, was recently observed for the first time at the NCNR. New experiments are being devised to investigate this decay in more detail and measure the gamma ray energy spectrum and polarization.

- The weak nuclear force between neutrons and protons is very fundamental but poorly understood. In order to observe its effects in the presence of the much stronger nuclear interaction, one must study extremely small parity violating observables such as spin rotation and gamma ray asymmetry. There has been much recent progress in the theoretical analysis of these interactions using chiral perturbation theory, but this progress

is constrained by a lack of experimental input. New high precision experiments are badly needed.

- Neutron interferometry is a unique method for directly observing the phase and coherence of neutron matter waves. This allows for precision measurement of nuclear scattering lengths that are needed for understanding nuclear potentials and structure. It also enables novel and important studies of quantum information and coherence.

- The neutron is electrically neutral but it is composed of charged quarks so it can have an internal charge distribution. In particular it may have an electric dipole moment (EDM), but it must be very small because it violates time-reversal symmetry. Ultracold neutron experiments at the ILL have established experimental limits on the neutron EDM at the 10^{-26} e ·cm level, but there is strong theoretical justification for a nonzero neutron EDM in the range of 10^{-29} to 10^{-26} e ·cm. A nonzero measurement would be a tremendous discovery. A new experiment that uses multiple Bragg reflection in a perfect crystal that could achieve this sensitivity has been proposed for the NCNR.

- An ultracold (UCN) neutron instrument that produces UCN via the superthermal method in liquid helium from a 0.89 nm beam has been operating at the NCNR for the past eight years. It led to the first demonstration of magnetic trapping of UCN (published in Nature in 2000). A current experiment to improve the neutron decay lifetime to 0.1 s precision is in progress. This technology can also be used to produce high UCN densities (perhaps > 100 cm^{-3}) that can be delivered to a variety of new experiments.

This is an exciting time to be working in fundamental neutron physics. The field is growing and there is a large number of scientific opportunities. A coordinated national program is developing that will make the best possible use of new, technically complementary facilities such as the NCNR Expansion and SNS.

Experimental needs at the NCNR:

In order to accomplishment the experimental program outlined in Scientific Opportunities, the fundamental neutron physics community would need the following facilities.

- **Neutron Physics beamline in new guide hall**

  – There could be a brightness-optimized end position for the experiments that currently do not utilize the neutrons with a large divergence. Experiments such as the proton trap neutron lifetime and radiative decay employee highly collimated beams and therefore require a high brightness beam to achieve the necessary counting statistics. Because brightness is important, the guide should be of minimal length.

  – There could be guide breaks for monochromatic beam positions. Specific opportunities have been identified: a neutron interferometer for studies of

quantum information, a station for an experimental effort into measuring the neutron magnetic and electric dipole moments, and a beam for the development of precision neutron polarimetry.

– Given the large footprint required by typical experiments and the number of monochromatic beams, a side guide is needed to provide sufficient lateral floor space for the experiments. A staging area is necessary to facilitate the assembly of these experiments offline. Some experiments have a significant vertical dimension that extends downward; a pit is needed to accommodate those experiments. The pit would be normally covered.

- **Neutron Physics end position in existing guide hall**

  – The fundamental limitation for the majority of neutron physics experiments is counting statistics. Hence, a beam line optimized for high flux is essential for making advances in those experiments. They accept large beams (6 cm to 8 cm diameter) and high divergence (30 mrad to 50 mrad). The flux can be significantly increased through the use of high-m guides, a ballistic guide, and possibly an optical filter.

  - There needs to be a capability for providing polarized beams through both supermirror bender polarizers and He-3 spin filters.

  - A velocity selector could be available on the beam line. There is a class of experiments where energy information is needed, for example, those requiring precision polarimetry or neutron energy information.

- **Enhanced UCN capabilities:**

  – The existing 0.89 nm beam line and optics for the UCN lifetime experiment are not optimized for filling a trap. The beam line could be located at a position where one can optimize the collection of 0.89 nm neutrons. It could reside in either guide hall.
  – This technology could also be applied to make an ultracold neutron production source on a dedicated end position that would enable a wide variety of new UCN experiments.
  – A UCN facility requires a large footprint.

IIix. Soft Condensed Matter : Biology

Participants: Frank Heinrich, Kalina Hristova, Susan Krueger, C. Ted Lee, Mathias Lösche, Tracy Nixon, Hirsh Nanda (NIST scribe), Adrian Parsegian, David Worcester, Ting Xu, Stephen White (Chair)

Scientific Opportunities

Determination of function in the context of structure forms the foundation of modern biomedical research. The past twenty years have witnessed a revolution in protein crystallography that has provided thousands of high-resolution 3-D protein structures, which are providing unprecedented insights into cellular function at the molecular level. Nevertheless, it has become clear that crystal structures, frozen in a particular conformation, cannot alone provide a complete picture of biological function. Protein structure must also be studied in native-like environments: complex aqueous environments for soluble proteins and their complexes, and lipid bilayers for membrane proteins. In these environments, proteins can no longer form the crystalline arrays necessary for crystallographic studies. Neutron scattering methods are ideally suited for studies of these partially ordered systems because of selective deuteration and the ability of neutrons to distinguish deuterons from protons. SANS studies of proteins in solution and neutron reflectivity/diffraction studies of membrane proteins in lipid bilayers will consequently play increasingly important roles in structural biology. Computational biology will be central to these efforts, because it allows reconstruction of dynamic protein structures from the low-resolution data inherent in partially ordered systems.

Increased Neutron Capacity

As the NCNR biology user community grows, the capacity of the current SANS and reflectivity/diffraction instruments, already oversubscribed, will become exhausted. New instruments are essential if this growth is to be accommodated. Biological samples, particularly deuterated ones, are expensive and are demanding to produce. It is therefore desirable that these instruments be optimized for small samples, e.g., 10 microliter volumes for SANS and a few square millimeters for reflectivity/diffraction (R/D). But this will require higher effective neutron fluxes on the sample, which means improved neutron optics for SANS. For R/D, important gains may be achieved using so-called white-beam technology that uses multiple wavelengths to enhance data collection by a factor of 10. Another important capability, not presently available with R/D instruments, is grazing incidence (in plane) diffraction for studying the lateral organization of membrane proteins in lipid bilayers. R/D instruments show promise for low-resolution neutron crystallographic studies of membrane proteins. Such studies can reveal the organization of disordered water and lipids in membrane protein crystals and thereby provide important information not accessible to x-ray crystallography. Because of the long data collection times required, it is desirable to have an optimized diffractometer dedicated to this purpose.

Detector Technology

Reflectometry, diffractometry, and high-resolution SANS applications will require two-dimensional detectors with millimeter or sub-millimeter resolution in both x and y directions. This resolution is about a factor of two better than currently available in detectors of 20 cm to 30 cm size. Separation of specular from off-specular scattering, as well as data collection for

resolving large in-plane features are best achieved with such resolution. In diffractometry, resolution of diffraction peaks from large unit cells is optimized by this resolution, since crystal sizes are typically sub-millimeter.

A reliable source of millimeter or sub-millimeter-resolution detectors must be established. In addition to serving new instruments, such detectors will also benefit existing instruments, for example the NG1 and NG7 reflectometers. The detectors need to have reliable position encoding with minimal oscillations across readout nodes, or some convenient means to correct for such oscillations during the experiments and not afterward. Convenience of use and visualization of scattering results during experiments is essential.

Sample Environments

Significant improvements in data collection are likely to be achieved by more effective use of polarized beams for both SANS and reflectometry/diffractometry. For reflectometry, a magnetic under-layer in combination with a polarized neutron beam allows for additional scattering contrast without changing the sample properties. This adds an additional constraint for the development of structural models. The use of a polarized beam in combination with polarization analysis on the detector side is expected to reduce background from isotropic scattering of the sample and, thus, expand the applicable Q-range of SANS experiments.

Convenient and efficient sample environments are essential for both SANS and R/D. For the latter method, a new generation of sample holders is essential for effective use of neutron beam-time. The sample environment could allow rapid equilibration at the desired humidity, temperature, and pressure. Sample cassettes could be designed to allow rapid insertion and alignment of pre-equilibrated samples in the beam. There is in addition a need to develop microfluidic sample holders that will make possible in-beam preparation/modification of very small biological samples. This design will require beams with very small cross-sections on the order of a few square millimeters.

Computational Capability

High-performance computing is important in four areas: Molecular dynamics and Monte Carlo simulations of molecular interactions and motions restrained by neutron data; construction of models for protein shape reconstruction from SANS data; visualization of molecules and molecular complexes; and new software designed to make these computational techniques more accessible to general users.

Molecular dynamics calculations are already an integral part of structural refinement for proteins in techniques such as x-ray crystallography and NMR. They can also complement neutron reflectivity and diffraction methods of membrane systems by providing detailed in-plane structure of these systems. In addition to structure, molecular dynamics can also complement inelastic scattering methods to map observed relaxation behavior onto specific molecular groups. An important advance in computational approaches is the use of “coarse grain” models that allow the simulation of much larger systems. This technique may in particular complement reflectivity and SANS measurements by providing a level of molecular detail consistent with experimental resolution. Recent developments have revamped interest in reconstructing low-resolution structures from small- and wide-angle scattering data. These include robust determination of the p(R) or d(R) distribution functions from scattering data, and structure

reconstruction via simulated annealing of coarse-grained models. For all of these purposes, ample computing capability, e.g., a 1024-processor PC cluster, is essential.

Biochemical Infrastructure

Recent efforts have been made to improve the biochemistry lab facilities at the NCNR. These could continue in order to make possible on-site purification of protonated or deuterated proteins. A deuteration facility, based on perdeuteration of biological material by the original method of photosynthetic algae cultures with CO_2 as the sole carbon source, could be implemented at NCNR. By assisting in the preparation of deuterated proteins, the NCNR will assure that users have optimal access to the contrast-variation method. The wet lab could thus provide for bacterial fermentations, cell harvesting, cell lysis, and preparative centrifugation. In addition, chromatographic instruments, such as HPLC, are essential for purification and analysis of proteins and lipids. To assure aggregate-free protein solutions, the chromatography systems could be integrated with modern dynamic light scattering – this will provide information about protein concentration and independent measures of particle homogeneity, mass and size.

Physical Characterization Infrastructure

In order to use the neutron beam time effectively, it is necessary for users to have access to other equipment on-site that will assure sample quality before the experiment, analyze data during the experiment, and plan subsequent experiments in a timely and efficient way. Necessary equipment includes small-angle x-ray diffraction (SAXS), static and dynamic light scattering, x-ray reflectivity, ellipsometry, and atomic-force microscopy (AFM).

SAXS and light scattering are especially important for characterizing biological samples in solution in support of SANS experiments. Neutron reflectivity experiments require the samples are flat and homogenous over a large area. To assure this, x-ray reflectivity and ellipsometry are essential for characterizing substrates before neutron reflectivity measurements are performed. For many experiments, particularly studies of nanostructures in thin films, it is not sufficient to characterize just the substrate homogeneity. Atomic force microscopy is necessary for examination the surface topography of complex samples.

Expanded Scientific Expertise

Because of the new opportunities for biological research and biotechnology that have emerged at the NCNR in the last few years, additional scientific personnel in several key areas are essential. These areas include computational biology, structural biology, membrane biophysics, and biology-related surface chemistry. This in-house scientific expertise will drive neutron-technology development and to ensure full utilization of the neutron facilities. Furthermore, these personnel help provide to the broader biological community access to neutron methods and facilities.

IIx. Soft Condensed Matter : Complex Fluids

Participants: Bill Hamilton, Yamali Hernandez (NIST scribe), Andrew Jackson, Eric Kaler (chair), Yun Liu, Danilo Pozzo, Greg Smith, Cherie Stancik, Lynn Walker, Barbara Wyslouzil

There are numerous scientific challenges in the area of complex fluids that can be addressed by neutron scattering methods. Examples include dense colloidal suspensions, polyelectrolytes, surfactant solutions, and a range of biologically related or biomimetic self-assembling systems. This science is also connected to a broad range of industrially significant technology challenges related to formulation stability and processing pathways in applications ranging from cosmetics to oil recovery. These materials are of interest at rest as well as under conditions of flow and during chemical reactions.

These fluids display a range of length and time scales suitable for study by neutron techniques. Length scales range from molecular dimensions (1 nm) to microns, and time scales from 10 μs to minutes. These fluids are also routinely studied by static and quasielastic light scattering and x-ray scattering methods, and a key requirement is that both static and dynamic neutron methods overlap the q and time ranges probed by light scattering.

The principal tools used are SANS, USANS, Spin Echo and Reflectivity. Both the sample environment and the robustness of the instruments currently limit the range of experiments that can be carried out. Particular comments are directed to each technique in turn, with the overarching need that instruments be robust and easily tuned to user needs. The availability of polarized neutron beams can be important for probing complex fluids, including for example the study of magnetic colloids. Another great need that must be address with all these techniques is the need of smaller sample size.

SANS: Users require a combination of high resolution, high flux, low background and a broad q-range for experiments. However, users do not typically require all of those features for any given experiment, so different spectrometers can be optimized for different requirements, as for example to achieve high resolution at moderate q-values with low background. The low-q limit is an important, and in many cases the dominant, consideration, and it must reach and substantially overlap the q-range available in laboratory light scattering instruments, i.e., a minimum q-value of 0.0005 $Å^{-1}$ with at least ten reliable data points below 0.0015 $Å^{-1}$. Q resolution also needs to be improved, which will reflect a narrower wavelength (velocity) distribution at moderate q and improved detector resolution so as to narrow $\Delta\theta/\theta$ at low q. The particular resolution needed will depend on the sample of interest. Finally, because samples may age or change dynamically, it is critical in many cases to measure the entire relevant q-range of 0.0005 $Å^{-1}$ to 0.5 $Å^{-1}$ simultaneously.

Low background levels are essential for measurements of samples that scatter weakly, and additional efforts could be made to reduce parasitic scattering, electronic noise, and fast and stray neutron fluxes. Location of a low-noise spectrometer in the new guidehall may provide an opportunity to address these issues, and development of a dectector with at least some level of energy resolution will also be helpful in this regard.

USANS: USANS can be an important technique for the study of larger (micron) scale phenomena in complex fluids (aggregation, emulsions, etc.) for which light scattering is limited because of turbidity, or for which H/D contrast variation is useful. The primary need is for more rapid experiments, which necessitates a substantial increase in flux. Secondary improvements would be development of a new camera to replace the Bonse-Hart optics which would i) enable simultaneous measurement of the spectra at all q values, ii) eliminate slit-smearing, and iii) enable measurements of more weakly scattering samples. Some of these requirements could be mitigated by a broader q-range in SANS.

Spin Echo: The main need is for overlap in q-space and time resolution with the method of quasielastic light scattering in order explore a full range of time and length scales. This would require *routine* measurements at q of 0.001 $Å^{-1}$ and correlation (Fourier) times of up to 100 μs. Examples would include the dynamics of mixed colloidal and surfactant solutions.

Reflectivity: Reflectivity is useful to probe the properties of complex fluids at surfaces, and to examine processes such as the structure of adsorbed layers, the flow of structured fluids near interfaces, and the kinetics of competitive adsorption. The principal need is enhanced fluxes and focusing to enable the use of smaller spot sizes for samples with in-plane variations or for high-value samples. Focused beams are more easily realized with reactor sources. Spin labeling can be used to optimize the use of divergent beams and enable measurements of in-plane structures on smaller volumes approaching a single monolayer. Figures of merit would be *routine* measurement of reflectivity of 10^{-8} for q ranges from 0.005 $Å^{-1}$ to 0.5 $Å^{-1}$.

Sample Environment and Instrumentation: There is a ***DRAMATIC*** need to decrease the time spent on sample environment changeovers and instrument configuration (e.g., alignment), and on changes in the sample conditions (e.g., temperature changes). Given the flux of existing instruments more than half of the measurement time can be devoted to changing sample conditions rather than measuring data, and this will only get worse with increased flux. Sample environments need to be enhanced with rapid electronic heating and cooling, control hardware needs to be upgraded to allow efficient and accurate changes in the configuration of the instruments, and there needs to be a robust 'plug and play' configuration for users to mechanically and electronically interface sample environments with the instruments.

The facility needs to recognize and support more sophisticated user needs for laboratory services at the instrument (plumbing and electrical) and enhanced air handling capabilities to safely handle fugitive emissions (elephant trunk ventilation). In house light and small and wide angle x-ray scattering facilities could be provided, and full laboratories for sample preparation could be provided immediately adjacent to the appropriate instrument.

There is a substantial need for specialized sample environments that would allow for stop-flow and T or P (or other field) jump experiments and for the easy synchronization of the time structure of those experiments with the detector (see below). Effort could be spent to enable and/ or enhance users' abilities to simultaneously measure neutron scattering with, for example, static and dynamic light scattering (Simultaneous Neutron and Photon Scattering (SNAPS)), FTIR, and rheology. Video observation of the sample could be provided. There is a need to control both

sample temperature and humidity (reflectivity). Finally, adequate floor space could be provided near the sample environment for users to load and prepare samples.

A plug and play interface could be provided to enable time-slicing data acquisition. For complex fluids experiments the characteristic times are not faster than 10 μs, but may range to minutes or hours.

Detectors: Detectors could be developed and made available to enable the simultaneous measurement of the full q range and could be robust enough to enable the full use of the available flux. Transmissions could be measured *in situ* and automatically.

IIxi. Soft Condensed Matter : Dynamics

Participants: Dobrin Bossev, Joseph Curtis (NIST scribe), Robert Leheny, Janna Maranas, Maikel Rheinstädter, Vicky Garcia Sakai, Alexei Sokolov (chair), Amos Tsai

Soft Condensed Matter: Dynamics

The study of soft condensed matter requires an extremely broad energy and momentum transfer range. These materials have no well-defined structure and are studied in amorphous and liquid phases. The spectra are very broad and have multi-exponential relaxations without a sharp Q-dependence.

Our suggestions are to develop instrumentation, sample preparation abilities, and software to provide *quantitative and routine* dynamical measurements. Considering the capabilities of world-class dynamics instruments, we feel that the NCNR could develop a first-class spin-echo instrument with ≈ 10 to 100 times higher improvement in the signal than the current instrument. We also envision that the instrumentation be adapted with capabilities to perform in-situ structural and spectroscopic measurements that will allow for qualitatively different dynamical measurements that will dramatically expand the number and types of experiments that can be done. Additionally, we call for the development of dynamical scattering instrumentation that covers a contiguous range of time and length scales. We feel the both the dynamical and structural soft-matter fields would greatly benefit from the addition of sample deuteration facilities at the NCNR which would concurrently dramatically extended both the type of science that can be done and the breadth of biological researches that would be willing to perform dynamical measurements.

Scientific Opportunities

There are many important scientific opportunities that will be positively impacted by the proposed dynamical instrumentation. Herein we are highlight two important areas, biological and polymer dynamics, where neutron science can have a major impact.

Biomolecular dynamics in complex condensed phase environments is going to continue be a very important area of scientific growth in the coming decade. It is becoming increasingly apparent that many proteins and other biomolecules have functional properties that are much more complex than simply catalyzing a biochemical reaction. Many proteins exist in solution as partially disordered structures, with their functional role dictated by the dynamics of the particular structural state that exists at a given time. Developing methods to study these proteins and to elucidate the specific sub-molecular events that lead to their function is an important research challenge over the next decade. Proteins and nucleic acids also are often associated with biological membranes. The understanding of the dynamical events that modulate activity at and through the membrane is a major research objective using a variety of techniques by scores of researchers. The dynamical events that dictate the preservation of protein structure in carbohydrate glasses is an area where neutron spectroscopy will continue to have an un-rivaled

impact. This is important to determine the fundamental microscopic factors that lead to drug development, delivery and bio-preservation.

In the past ten years, the study of polymer dynamics by neutron scattering has shown that classical models of chain dynamics only provide a qualitative description and traditional methods, such as rhology fail to provide microscopic insight. Improvements of neutron-spin echo instrumentation will allow for ground-breaking studies of thin polymer films, nano-composite materials, polymeric fuel cell membranes, electrolytic polymers. Understanding the influence of spatial confinement on the local and collective dynamics as a function of length scale is crucial to understand these complex systems.

Why Neutrons?

The ability of neutrons to probe the structure and dynamics of materials is well documented. Using the present examples described above, the ability of neutrons to measure atomic motions over time and length scales relevant to biological systems in heterogeneous environments is unparalleled, although, the capabilities have not been exploited to the extent that is theoretically possible due to limitations of robust instrumentation and sample preparation capabilities.

Neutron Instrumentation

For soft-matter condensed phase systems the important time range is from the sub-picosecond to microsecond (and eventually millisecond) and momentum transfer range from 0.001 $Å^{-1}$ to 3 $Å^{-1}$. Considering the capabilities of pulsed neutron sources at the SNS, we have decided to focus on suggesting the development of a world-class neutron-spin echo instrument. We strongly suggest a time-scale from 100 ps to 1 μs and a momentum transfer range from 0.001 $Å^{-1}$ to 1 $Å^{-1}$. We also strongly suggest that the signal to noise of the new instrument be ≈ 10 to 100 times better than the current instrumentation at the NCNR. To achieve these goals, it was decided that one could develop a multi-angle spectrometer with a contiguous range of time and length scale to overlap with other dynamics instruments at the NCNR.

One of the major suggestions for the new instrumentation is that it could provide quantitative results (reproducible signal from the same sample) and that it could be built with the appropriate hardware and software so that it can be routinely used. Our hope is that the level of ease of use approach or surpass that of the SANS instrumentation at the NCNR. Additionally, we feel that the instrument utilize a smaller neutron beam size without a loss of neutron flux. This will allow users to use samples with a smaller volume.

Sample Environment

There are two important and novel sample environment capabilities that would make the neutron spin-echo instrument at the NCNR stand apart from other neutron facilities and provide users with unique and versatile capabilities.

The first suggestion is to add optical access to the sample chamber. This will allow for the concurrent observation of the sample by various means (visual, spectroscopic) and for the perturbation of the sample (photo-initiation, pump-probe experiments).

The second suggestion is that the NCNR develop a deuteration facility to help with the preparation of samples that are unique to the neutron scattering measurements. This of course would benefit many other neutron measurements at the NCNR (SANS, reflectivity, back scattering, time-of-flight, etc.). This facility could be adequately equipped and staffed to collaborate with researchers to develop both small molecule and large-molecule deuterated samples. We do not see either a technical hurdle to develop this technology at NIST, nor a reason to depend on other deuteration facilities such as the capabilities being developed at the SNS. For example, nearly every NMR lab that performs dynamical measurements has internalized the technology to develop per-deuterated proteins. The benefits of having both the technical capabilities and the personnel to carry out these preparations would be tremendous and would lead both to the development of new science and it would improve the types of experiments that have traditionally been done at the NCNR. The latter point would evolve from the communication of biochemical details with neutron users that may not have the biochemical expertise to perform new and important physics measurements on such samples.

We suggest the development of a pressure cell (up to ≈ 10 kbar) that can be used inside the cryo-furnace. We also suggest that a controlled humidity cell be developed to allow for the study of hydration dependent systems.

Additionally, we envision that adequate computational chemistry resources (hardware, software, and personnel) be allocated to allow users to develop atomistic models of their neutron measurements. The exact enumeration of such resources could be scaled based upon the number of users that require these capabilities.

And finally, we strongly believe that a guesthouse (on-site lodging facilities) be built at NIST to allow users to visit the NCNR for extended times at a reasonable cost. Clearly, these facilities could be dormitories with limited facilities in order to maintain a low cost both to the user community and NIST. It is also suggested that users be able to check the status of dynamical instrumentation remotely via a web-based interface.

IIxii. Soft Condensed Matter: Polymers

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Introduction:

Understanding the structure and transformations of polymer-based advanced materials is a key to American competitiveness. Neutron scattering has played a crucial role in the understanding the bulk and thin film behavior of polymeric materials and will be central in realizing revolutionary advances required to maintain American economic competitiveness and leadership in future. Advanced, multifunctional materials integral to future progress include hybrid or composite materials exhibiting multi-length scale (tens of nanometers to many microns) ordering that form hierarchically ordered structures that will play a key role in future applications from the emerging fields of nanoscience, biotechnology, and energy.

Current neutron facilities, in particular small-angle neutron scattering (SANS) and neutron reflectivity (NR) instruments, cannot meet the challenges posed by these opportunities. Oversubscription, limited wavelength resolution and neutron flux represent barriers in realizing the full potential of these emergent fields. The advances in the proposed NCNR upgrade, as part of the National Initiative on Competitiveness, will overcome these impediments by significantly expanding the capabilities of the current facility.

Five new capabilities needed to maintain competitiveness:

- **higher flux**
  - 10× over current required to revolutionize science
  - Larmor spin labeling SANS, NR for higher flux (divergent beam, limited collimation)
  - faster turn-around, more samples, kinetics of structure evolution possible
- **lower wavelegth spread/better energy resolution for both SANS and NR**
  - spread ($\Delta\lambda/\lambda$ = 0.01 to 0.02)
  - dual beam guide (SANS) for both high flux and low wavelength spread
  - Larmor labeling SANS, NR for high resolution experiments, lower q_{min}
- **ultrasmall beam (SANS)**
  - < 100 μm (cross-cutting with optics-focusing)
  - smaller sample size
  - interfacial structure, microfluidics flow structure)
- **measurement of dynamics and 3-D structure near interfaces**
  - spin-echo NR
- **more beamtime available**
  - multiple converging collimation for new VSANS (V = very) on 30 m
  - short beamline (10 m) for measurement of small objects requiring only higher q
  - additional SANS, additional NR (> 2× oversubscription of current facilities)
  - higher flux for faster experiments, smaller sample sizes,
  - experimental optimization (e.g., faster instrument configuration for SANS/NR)

Advanced Materials for Nanoscience, Biotechnology or Energy in Need of New SANS/NR capabilities

Smart biomedical materials through self-assembly

Exciting advancements in the treatment of diseased/damaged human tissue, ranging from scarred heart infarct tissue to tissue voids created after tumor tissue removal (resection) , are strongly dependent on the successful development of new, physical hydrogel materials. The ability to create biocompatible hydrogel materials via physical association , i.e., self-assembly, of organic/polymer molecules in solution is critical for in vivo therapies in that hydrogels containing drugs and/or cells can be delivered via simple injection into the damaged tissue/tissue void. While the hydrogels must obviously be chemically compatible with mammalian cells/the human body for the success of these therapies, the materials must have the correct morphology. Local hydrogel scaffold structure (1 nm to 10 nm) must be porous for salt and protein diffusion but yet have a discrete nanostructure for interaction with cell membranes. The local structure must interconnect on larger length scales (10 nm to 100 nm) to provide for desired network rigidity but yet display porosity on the micron/cell level length scale. While laser scanning confocal microscopy and cryogenic transmission electron microscopy provide critical local structure information, SANS is vital for global hydrogel structure determination. However, there is currently no neutron method available to characterize this hierarchy of structure, critical for the biological success of the materials, in one, *in situ*, experiment.

With an increase in neutron flux (10× great than current) and lower wavelength spread ($\Delta\lambda/\lambda$ = 0.01 to 0.02) in SANS, scattering can be resolved down to much lower q ranges that current available in SANS (($q \geq 0.0003$ Å^{-1}). In addition, lower wavelength spread would provide the capability for multiple order Bragg scattering on the nano- through micro length scale in order to probe periodicity locally or globally within the self-assembled network. Increased flux and resolution also provides the tantalizing capability to monitor the self-assembly of the local structure in real time. This ability to monitor the kinetics of structure evolution, combined with the ability to characterize the entire hierarchy of structure, provides an important feedback look between advanced biomaterial properties and polymer design for self-assembly. Consequently, SANS upgrades at the NCNR fulfills a key need in nanotechnology and advanced materials for medicine that fulfills the mission of NIST to support potential biotechnology economic development.

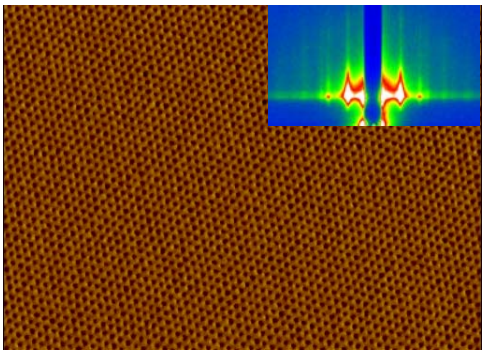
Advanced Nanocomposite Materials

The ability to increase polymer material properties via the addition of nanoscopic fillers is well-established. New advances in these composite materials are dependent on new hybrid materials to not only enhance mechanical properties of the polymer matrix through desired distribution/suspension of the filler phase within the polymer matrix, but also to add functionality to these hybrids (e.g., electrical conductivity through a percolated, conductive reinforcement phase). It is mandatory to be able to tailor the spatial distribution of these nanoscopic fillers in the polymer matrix and have the means to characterize the structure of the composites quantitatively. For example, the performance of carbon nanotube (CNT) reinforced thermoplastic polymers is strongly dependent on the CNT distribution throughout the polymer from the nanometer to micron length scales. This CNT distribution is depends strongly on the processing conditions and the interactions between the CNT and the polymer. To quantitatively characterize the structure of the composite or thin films within 1 μm from the interface, a lower

wavelength spread, $\Delta\lambda/\lambda = 0.01$ to 0.02, is required for thick film, high resolution NR. Low q, high resolution SANS measurements of CNT distribution from both local (nm) length scales up to very low q ($q \geq 0.0003$ Å^{-1}), large length scale (1 μm) is needed, but not currently possible, via a SANS measurement. Only with an easy to characterize the CNT distribution over multi-length scales can structure-property and structure-processing relationship be formulated for advanced hybrid materials.

Thin Film Metrology

The fabrication of nanostructured materials holds the promise of revolutionizing storage media, display technology, and advanced sensor technologies. This can be achieved by producing addressable media comprised of 2-D or 3-D periodic, highly ordered arrays of nanoscopic elements with long-range lateral order. This order, however, must be a close to perfection as possible and manipulating the self-assembly processes that lead to this ordering is key. However, there is a critical need to characterize these emerging nanostructured materials. We are faced with the daunting challenges of quantitatively characterizing materials on the nanoscopic level over areas that are cm^2 in size. At present, there is no method or technique that can meet this challenge. As an example, consider the scanning force microscopy image shown of an array of nanoscopic domains in a thin block copolymer film where each domain is ≈ 15 nm and this array extends of many square microns. Scanning force and electron microscopies, while exquisite on the μm^2 scale, fail miserably with larger area. Quite simply, there is no way to correlate multiple images with each other on a nanometer level. X-ray scattering (shown in the inset), while comparable in providing identical structural information as neutrons, can cause damage or degradation of the soft materials being probed. The planned NCNR upgrade offers a unique opportunity in the metrology of thin films.



In particular, with a nearly an order of magnitude increase in neutron flux on a sample with a wavelength resolution of $\Delta\lambda/\lambda \approx 0.01$ it becomes possible to obtain SANS on thin film arrays of nanostructured organic and inorganic materials. With typical neutron beam sizes of cm^2, structural information on the nanoscopic level is obtained over large areas. With improved wavelength and angular resolution, $\Delta q/q$ better than 0.01 can be achieved, allowing structural information to be obtained over multiple length scales. At lowest q, macroscopic information on the size of grains is afforded, while higher order reflections characterize the extent of lateral order. From the analysis of the SANS, not only can one obtain information on defects (spatial and angular) in the structures via standard crystallographic methods, but higher order correlations can be assessed that will be essential in assessing order. Consequently a new metrological tool is enabled with the NCNR upgrade that fulfills a key need in nanotechnology, offers a new metrological tool, and fulfills the mission of NIST.

Medical Device Characterization

Medical implant devices typically comprise a variety of materials that include a structural substrate and biocompatible coating that may serve multiple functions. The substrate is often a metallic alloy or a ceramic material that has suitable biological properties. In some cases, the

structural substrate may be a polymer material. The biocompatible coating typically comprises a polymer layer possessing a thickness of order several microns, and this may also be comprised of more than one polymer material. Further the polymer layer is often impregnated with a small-molecule drug or a biologic agent that serves to effect or enhance an improved therapeutic benefit. Such devices are often called "smart" because they are able to perform multiple functions over time that can be remotely activated, or are activated in response to physiological conditions.

Because such a medical device must perform several functions simultaneously, its composition includes polymer materials and interfaces between the polymer layer with the biological environment and the substrate. The structure and dynamics of polymer chains at these interfaces determines how well the device will function in vivo. These interfacial interactions affect the adhesion of the coating to the substrate, and the various polymer layers, interaction of the biocompatible coating with the patient's body, and release profile of the impregnated therapeutic agent. Neutron scattering offers unique capability to probe and understand the nature of the polymer layer and its interactions with the various components of the device and the patient. Current capability in neutron reflectivity is a limiting factor that could be addressed by the expansion. These include: 1) increased Q range and Q-resolution will make possible the study of these "thick" films; 2) reduced beam size will provide missing capability to query samples that are directly relevant to application, avoiding the need to develop model samples; 3) Ability to probe 3-d structure; 4) Ability to probe polymer chain dynamics at the interfacial regions; and, 5) Ability to probe time-dependence (kinetics) of polymer chain structural evolution.

Hierarchical, multiple length-scale, 3-D structure in thin films

In many hybrid materials essential in nanotechnology, biotechnology and energy applications, thin films are present and a substantial fraction of the total volume lies adjacent to an interface. Key advances in our understanding of the 1-D variation in structure across interfaces and in thin films have been made using SANS and NR. Now, however, it is critical to understand the structure in the direction parallel to the interface as well. Off-specular scattering experiments or grazing incidence small angle scattering (GISANS) experiments will be central in this effort. These measurements present competing demands. Greater flux is needed to distinguish the weak off-specular scattering from background. The maximum value of q_y could allow the study of structures on the 10 nm length scale. At the same time, GISANS measurements demand higher resolution in q_y than do conventional NR measurements. Scattering features characteristic of micron-sized grain structures lie close to the specular beam where intensity is high, but high q_y resolution is demanded. A proper combination of improvement in source brilliance, incorporation of 2-dimensional position-sensitive detection with improved spatial resolution, and spin-labeling to allow the use of divergent beams could put such measurements within reach.

Interfacial characterization of polymers dynamics

The interfacial characterization of solid-solid and solid-liquid interfaces involves several research areas in emerging and established technologies where polymers play a critical role. Examples include the characterization of tissue engineering scaffold or drug-delivery materials, .surface swelling of advanced photoresists, microfluidic and microarrays used to manipulate DNA, and confined nanostructures. Metrologies to characterize the macromolecular dynamics

on the time scale of 10 ns to 100 ns by inelastic scattering by interface-sensitive methods are an immediate metrology challenge which can be met by a combination of improved flux and applying neutron spin-echo spectroscopy concepts with reflectivity (NSE-R).

These measurements will improve advanced materials design by providing how the material properties (interfacial diffusion and distribution) are affected by the interfacial confinement. Such information is unavailable for direct measurements by other complimentary methods.

Important points for new users/unforeseen new scientific developments

Several points need to be emphasized that would impact all of the scientific advancements mentioned above. First, NCNR could continue to focus on the education of and outreach to the scientific public to increase a savvy user base to take advantage of the improved/expanded facilities this initiative will make available. Importantly, stable or increased staffing at the NCNR is critical to the success of this outreach. Second, the sample platforms of both the existing and proposed SANS and NR instrumentation need to be open. In other words, NCNR has been successful in the past in the incorporation of novel sample environments (e.g., sample cell compatible with a rheometer, high pressure sample cells) with both SANS and NR. New experiments and emerging areas of science (e.g., biosciences with materials under *in vitro* conditions) will demand unique new sample environments/experiments coupled with NR and SANS. NCNR could design new instruments that accommodate and invite new sample environment platforms. Third, development of higher resolution and higher reliability 2-D position sensitive detectors will be a key to both the development of GISANS and off-specular measurements with the reflectometer and the development of low q, high resolution SANS experiments. Third, an issue that extends well beyond NIST is the need for uniformity in user interfaces and data analysis software across laboratories. Fourth, running experiments would be greatly facilitated if users faced the same user interface at every SANS instrument within NIST and also at other U.S. national labs. Development of new software for instrument control and data analysis could be concurrent with the development of new instruments. User-friendly software for control and analysis could ideally be in place when new instrumentation/capabilities come online. This will facilitate the rapid production of scientific publications from the new instrumentation that will be critical for justifying continuing investments in the improvement of NCNR capabilities. Finally, rapid production of scientific impact will also be aided by providing staffing at the new instruments that will facilitate efficient use of beam time in the face of an influx of new users expected as a result of the dramatic new capabilities this initiative will bring. Improvements in the user interface will encourage new academic users and new industrial users as well.