Neutron Measurements for Materials Design & Characterization Workshop
August 21-22, 2014

Executive Summary

The success of America's industrial and academic institutions and America's role as an economic leader in the world is built upon innovation and entrepreneurship in fields of science and engineering. Maintaining this leadership requires pushing the boundaries of discovery and the constant creation of new knowledge to provide a pathway for the development of new technology. Neutron scattering is a key measurement technique for elucidating a broad range of physical phenomena ranging from the atomic structure of new materials, the complex interplay of electronic and magnetic behavior, the interactions and flow of colloids and macromolecules and the dynamics and superstructure of complex biomolecule assemblies. In the era of big data and combinatorial materials synthesis, neutron scattering is a leading tool for guiding the design, discovery and characterization new materials. The NIST Center for Neutron Research (NCNR) has been at the forefront of neutron science for the past 20+ years and with the construction of a new instrument guide hall, the installation of five new neutron guides , a new fundamental physics station, improvements in current instruments, novel detector technologies, two innovative new instruments (VSANS, CANDoR, and an imaging station with a neutron microscope) under development, and planning for a new D_2 cold source, the NCNR is poised to remain a leader in neutron research in the U.S. and the world for many years to come.

With the new guide hall there are additional opportunities to strengthen the science in both hard and soft matter.

Soft Matter

Neutron scattering has provided fundamental insight into the structure and dynamics of soft matter for more than 40 years through deuterium labeling and by exploiting the large incoherent cross-section of hydrogen. With advances in optics and instrumentation there are a wealth of opportunities for new understanding in colloid and polymer science and in biology using neutron scattering. Recent advances in synthesis of designer colloid systems allow sophisticated control of particle size, shape and surface chemistry that provide a large design space for self-assembled structures. Many of the most important applications of polymers involve multicomponent systems with end-use properties that are optimized by kinetically trapping the nanoscale structure. Neutron scattering can provide both an understanding of the structure and information on the kinetics of structural evolution and local dynamics within the morphology. In contrast to bulk systems, polymer surfaces and interfaces hold the key to our understanding of some physical processes. For example the location of just a few layers of water molecules can be tracked using neutron reflectometry during the initiation of corrosion or the operation of proton exchange membrane fuel cells.

The wealth of information on biomolecular structure provided by X-ray diffraction, NMR and cryo-TEM over the past 50 years has helped build an understanding of biological function on the molecular level. It is now apparent that there are many supramolecular assemblies of proteins with DNA, RNA, lipids and other biomolecules that play a critical role in cellular function. Neutron scattering can provide powerful insights into the structure and local dynamics of these assemblies through the use of selective deuteration to highlight (or contrast match) parts of the system. By leveraging the NIST/IBBR Biomolecular Labeling Laboratory (BL^2) specific labeling of proteins can be done to examine the dynamics of disordered proteins, protein aggregation and elastic properties of lipid arrays.

The penetrating power of neutrons makes many types of *in-situ* experiments feasible that are much more difficult using other techniques such as X-ray scattering. For example many of the most interesting soft and biological materials are strongly influenced by external fields (electric, shear, pressure) and neutron scattering can provide fundamental insight to the behavior of soft materials under these conditions. Similarly neutron scattering could provide real time information on the local diffusion and mixing process occurring during additive manufacturing through interfacing a 3D printing system on a small angle scattering beamline.

Hard Matter
Neutron scattering has had tremendous impact on our understanding of hard matter ranging from fundamental condensed matter physics to nanomaterials to materials for energy conversion and storage. The ability to probe structure and dynamics over a broad range of length and time scales is indispensable in areas that include hard and soft magnets, quantum correlated materials, high-T_c superconductivity, multiferroic, ferroelectric, and thermoelectric materials. Because their nuclear and magnetic interactions with materials are of similar strength and can be distinguished in polarized beam experiments, neutrons are essential to disentangle the complex interplay of charge, spin, lattice and orbital degrees of freedom that can produce unique physical properties. It is also important that neutron scattering can be applied to bulk and nanostructured solids under a wide range of thermodynamic conditions that mimic applications or expose novel properties.

While quantum coherence beyond the atomic scale is best known from superconductivity, it can also occur in magnetic materials when competing interactions frustrate the development of conventional magnetic ordering. One of the largest focus topics at the annual APS meeting, the field of frustrated magnetism is uncovering fundamentally new forms of quantum correlated matter with potential impacts in quantum information processing using neutrons as the preeminent experimental tool.

Neutron scattering is also essential to understand and control materials in the technologically important area of spintronics where functionality is associated with the spin of the electron rather than its charge. Thin film neutron diffraction was thus cited by the 2007 recipients of the Nobel Prize in Physics as critical to understanding the phenomenon of Giant Magneto Resistance, which is the key technology in more than 5 billion read heads for gigabyte hard disk drives shipped since 2007.

Materials Chemistry
Technological breakthroughs are often derived from discoveries of new materials. It is through rational design and discovery of new materials that the US can maintain technological leadership in the world. This includes high throughput synthesis and screening of materials such as metal oxide framework and zeolites, piezoelectrics, superconductors, ionic conductors, *etc*. as outlined in the Materials Genome Initiative. These materials often combine many different atoms in large unit cells with subtle distortions of atomic positions and bond lengths that give rise to interesting and useful properties.

With increased interest in materials for energy conversion and storage there is a major opportunity for neutron scattering to provide non-destructive characterization of complex systems such as Li-ion batteries, proton exchange fuel cells and carbon sequestration materials *in-operando*. Neutron scattering can play a complimentary role to electron microscopy and X-ray scattering in charactering thin film organic and heterojunction photovoltaics, supercapacitors and solid electrolyte batteries.

Nanostructured materials such as quantum dots, nano- rods and particles, topological insulators, and catalysts for use in next generation magnetic storage media, quantum computers, nano-medicine and advanced chemical synthesis represent an wealth of scientific opportunities where neutron scattering will play an important role. Many of these systems will require the state of the art neutron instrumentation associated with the new NCNR guide hall and future D_2 cold source to provide the range of brightness, Q-range, and energy transfer to characterize effectively.

Engineering

Neutron diffraction measurements of residual stress in solids has proven to be a very valuable technique for examining bulk materials and manufactured objects both prior to service and post-service for failure analysis. Due to their penetrating power, neutrons are particularly powerful for analyzing residual stress in dense materials and for measurements on large specimens at high diffraction angles. Additionally, innovative sample environments for realistic, multi-axial materials deformation will enable the measurement of mechanical properties of engineering materials in ways not available to other methods. This combined with the ability to exploit neutron cross-sections to examine structures associated with light elements (hydrogen) and differentiate elements such as Co and Fe makes neutron diffraction a unique tool for understanding engineering materials.

Instrumentation and Facilities

With the development of VSANS and CANDoR, the promise of a new D_2 cold source, improvements in many other instruments and new detector technologies, the NCNR will remain one of a small number of leading neutron scattering facilities in the world. There are still open end positions available (one served with thermal neutrons and one with cold neutrons). These positions are quite valuable and new instruments should be built at each after a careful evaluation of proposed instrumentation is carried out. Some proposed instruments include:

> *High data rate materials diffractometer* – this is particularly relevant given the emphasis on materials discovery and high throughput characterization techniques and the Materials Genome Initiative. Such an instrument could play a role across many fields of science and technology, for example when a new material is identified with unexpected properties (such as a new high Tc superconductor), for screening large numbers of newly synthesized compounds or for following the kinetics of temperature or field driven phase transformations or synthesis of novel materials in real time.
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> *High wave-vector resolution multiplexing spectrometer* – A new concept for high efficiency neutron spectroscopy at a reactor-based source is proposed where the detection system *simultaneously* covers a wide range of scattering angles and a full spectrum of neutron

energies. Such an instrument would retain the high momentum resolution of a conventional cold triple axis spectrometer such as the SPINS instrument while gaining at least two orders of magnitude in data rate. The proposed instrument would constitute a powerful new tool for the elucidation of collective phenomena at the mesoscale in hard condensed-matter physics.

Biological SANS instrument – there has been a resurgence of interest in small angle scattering in the biological research community over the past 10 years with a dramatic increase in small angle X-ray publications and a similar increase (but at a lower level) in SANS publications. The current NCNR SANS instruments are oversubscribed and are likely to remain so for the foreseeable future. A biological SANS instrument should be optimized for beam brightness at the sample position to allow for smaller sample volumes and lower concentrations (≤100 µl or less with ≤1% sample concentration).

Upgraded NSE instrument – neutron spin echo which is essential for elucidating details of the motions of materials provides the highest energy resolution available with neutron scattering. The recent upgrades to this instrument have improved the performance by a factor of 3. Even larger gains could be realized by improvements in the magnetic field correction elements, replacing the resistive main coils with superconducting coils and by providing better focusing at the sample position through the use of Wolter optics or other focusing schemes.

There should be a concerted effort to develop new neutron optics (focusing optics and Wolter optics) to provide high flux and high brightness neutron beams for all instruments. In many cases increasing the scientific impact and user base for a technique is limited by the flux and brightness of the incident beam. For example, neutron spectroscopy of soft materials could make many substantial new scientific contributions with improvements in optics to allow for sample volumes on the order of 100 µl. The implementation of brighter neutron beams will allow for *in-situ* experiments, irreversible time-resolved kinetic measurements, smaller and more compact sample environments and the opportunity to examine a broad range of novel devices (batteries, microfluidics, chromatography systems, *etc*.) while in operation in the neutron beam.

NIST has played a leading role in the development of neutron polarization techniques through the use of spin filters based on polarized ^{3}He. This is having a major scientific impact in hard condensed matter experiments. Thus these efforts should be expanded to more instruments. Moreover NIST should continue to work at improving all aspects of the performance of these devices with a particular goal of reaching at least 90% polarization of the ^{3}He nuclei in the filters.

The large penetration depth of neutrons allows for a range of sample environments including shear, pressure, electric and magnetic fields. Although these areas have been explored, there is a need to expand the availability of such sample environments to all instruments and to engage the scientific community in identifying and developing new systems that extend the range of the applied fields.

In addition to the capabilities at the neutron spectrometer in terms of sample volumes, sample environments, *etc*., there needs to be a concomitant level of support for the users in terms of local lab facilities, software and scientific expertise at the facility. What often differentiates one user facility from another is the quality of the support infrastructure in terms of lab space for sample preparation, ancillary characterization tools, high quality data collection, visualization, and analysis software and staff expertise in the relevant science, modeling, and analysis. Having data collection and visualization software that can be learned in the time frame of 1-2 hours benefits both external users and instrument scientists. Too often improvements in data collection, visualization, and analysis software take the lowest priority and results in frustration among users and reduced scientific productivity.

Access to neutron scattering beamtime is a valuable national resource and throughout the world most neutron facilities report that the majority of their instruments are oversubscribed by a factor of 1.5-3x. With the expansion of detector arrays, data sets have become increasingly large and complex, particularly when combined with time resolved measurements as a function of temperature and/or external field. This makes it difficult for the original experimentalists to extract and publish all scientific results supported by the data in a timely fashion. NIST already makes all raw neutron data available to the general scientific community. Thought should be placed on improving both the searchability of these datasets and the provision of relevant supplementary information to fully define the experiment. In this way, NIST could play a key role in facilitating expedient analysis of neutron scattering data by engaging a broader array of scientists.

Through the implementation of the on-going and planned upgrades to the facility and new instrumentation and by following the science outlined by the participants at this workshop, the NCNR will remain a world leader in neutron science for the next decade and beyond.