Soft- and Bio-Materials: Scientific Strategic Planning White Paper — *31 March 2008*

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1. Introduction to Scientific Theme

In soft matter and biomolecular materials, the structure-function relationships are critically important, for basic knowledge and for application to medicine and industry. Mobility in liquids and soft condensed matter, even in homogeneous systems, leads to rich phase behavior: glass transitions, liquid crystalline order, and boundary structures such as surface layering are all examples of basic discoveries which led to new directions in applied materials science. Increased complexity arising from mixtures and from asymmetric or polar molecules leads to phase separation, to ordered mesostructures such as lamellae in diblock copolymers or microemulsions, and to useful properties such as ferroelectricity. Biological functionality can tune a material further by creating highly specific interactions. Addressable structures can be built around nucleic acid hybridization. Hierarchically structured assemblies achieve otherwise unattainable functions, ultimately exemplified in true biological systems.

These extremely broad classes of materials have in common the fundamental importance of structure to their function. Synchrotron based measurements in these systems are necessary for successes to continue in this field. Although a huge number of very different systems fall under the “soft and bio” rubric, classifying them by the nature of their molecular assembly makes it clear which beamlines are of perennial importance:

Composite and hierarchical materials such as polymers, inorganic-organic composites, and biomaterials require scattering from large and small q ranges to capture their multi-component structures — *SAXS/WAXS* capabilities. Probing individual substructures requires *micro/nanobeam* capabilities.

Confinement leads to new physical phenomena in nano- and micro-fluidic systems, soft materials in contact with patterned substrates, and molecular scaffolds. *High q-resolution scattering* and SAXS/WAXS are apt probes. Surface scattering is essential, especially the *grazing-incidence* geometry. Liquid surface capabilities are also important.

Molecular self assembly underlies soft and bio materials. Basic information about molecular conformation and assembly is obtained from *solution SAXS* for proteins and can be applied to synthetic protein- and DNA-based systems. Biomimetic systems are also frequently arranged in thin film geometries, especially for studies of membranes. *Liquid interface scattering* is a crucial technique for study of molecular self assembly.

To completely specify the structure, methods sensitive to *chemical composition* and *bond orientation* are needed. Spectroscopic methods such as *x-ray absorption spectroscopy*, *x-ray photoemission spectroscopy*, and *resonant x-ray scattering* must be used. In soft- and bio-materials, light elements predominate. Therefore, the science demands scattering and spectroscopy developments in the *0.2 to 5 keV x-ray range*.

Knowing that these techniques are available, the challenge we now confront is to take the very best advantage of them. New chemical synthetic approaches and bio-inspired models are driving soft- and biomaterials development. The degrees of positional and orientational order that give rise to the unique materials properties are accessible at levels of detail which are unprecedented, using cutting-edge synchrotron experiments. Here we highlight three areas of very high impact:

The brightness and stability of NSLS-II will transform microbeam SAXS/WAXS, a frontier at which dramatic discoveries are made every time the spatial resolution is improved. Biomaterials especially tend to be hierarchically structured, and microscopies (electron and optical) reveal structures from molecular to nanometer to micron and larger length scales. Scattering properly quantifies the structural correlations. For soft matter, microbeam scattering must be applied in SAXS/WAXS transmission geometries as well as grazing-incidence (GISAXS/GIXD) geometries for surfaces. Scattering from these structures is often weak and has continuously varying features. Many orders of wave-vector transfer q must be accessible under conditions of extremely low background scattering. This must be accomplished for samples being studied under hydration, applied fields, stress/strain measurements, processing apparatus, and during chemical reactions. The brightness of NSLS-II enables scattering at very small angles, stable focus to tiny spot size, and the prospect of illuminating very small surfaces at the critical angle to reduce background scattering from the bulk or substrate.

NSLS-II is an ideal source for the next generation of liquid interface scattering. Liquid interface measurements access fundamental aspects of self assembly, since surface energetics can be measured directly with no imposed epitaxial order from a substrate. Systems of interest include metallic, polar, and non-polar liquid interfaces, supporting interface phases and films of all kinds. Scientific impact has always been high, since unique information is obtained. Now, the keenest scientific interest lies in areas of greater complexity, including more realistic mimics of interfacial biological processes, and the description of dynamic interfacial assembly and reactions. Water-oil interfaces are a model for the interaction of water with a hydrophobic molecular environment, important for protein folding and the formation of structure in complex fluids. Phase transfer catalysis, pharmaceutical drug delivery, many electrochemical processes, and numerous chemical reactions take place at the interface between two immiscible liquids. Industrial applications include surfactants in domestic products, ion selective electrodes, tertiary oil recovery, and removal of unwanted ions and radionuclides from the environment. Understanding these processes requires techniques that probe absorption, ion and charge transfer, and molecular ordering at the liquid interface. NSLS-II will deliver bright beams over the wide x-ray energy range that these experiments demand.

Low energy x-rays in the 0.2 to 5 keV range at NSLS-II represent new opportunities for soft-matter science. In non-crystalline materials, molecular orientational order creates technologically important properties, by coupling electronic, optical, and mechanical response. The study of orientational order requires using the superb control over the polarization state of the x-rays which NSLS-II will provide. Resonant scattering techniques and related methods can elucidate molecular orientational ordering as well as orbital and spin ordering, and provide contrast between materials with otherwise similar electron densities. Frequently, the contrast limitation is overcome by combining selective deuteration with neutron scattering. Resonant scattering near C, N, and O edges is potentially as sensitive as neutron techniques without the need for deuteration. Improved x-ray microscopy methods with compositional sensitivity (NEXAFS microscopy) will provide complementary real-space information for many soft-matter materials and organic-inorganic composites. The x-ray energy range from 0.2 to 5 keV is well served by NSLS-II and covers important edges for both soft and hard condensed matter physics and materials. Furthermore, the high brightness and flux will provide an opportunity to develop a new XPS three dimensional chemical microscope (currently under development by NIST). Development of resonant scattering, spectroscopy and microscopy in this lower x-ray energy range is a natural fit to the mission and capabilities of NSLS-II.

A community of several hundred people perform synchrotron experiments in soft- and bio-materials every year. At NSLS-I, seven x-ray scattering beamlines have soft matter as their major scientific focus. Soft- and bio-materials are also routinely studied at the hard and soft x-ray microspectroscopy beamlines, at XAS beamlines (three soft x-ray NEXAFS and XPS beamlines), and of course share an overlap with the Life Sciences community. Surface and interface scattering are useful for solid state systems and for study of catalysis. Significant synergy with other communities is available, if the soft- and bio-materials communities design and maintain their beamlines of primary importance.

Participation in synchrotron radiation research by industry is long-standing. ExxonMobil, Dow Chemical (with NIST), and IBM maintain scattering and spectroscopy beamlines at NSLS-I. Dow Chemical is an active user of microscopy and resonant soft x-ray scattering facilities at the ALS, programs which would be attracted to NSLS-II by unique capabilities such as polarization control. Synchrotron research results are very well received at conferences where industrial researchers are present, but the barriers for new companies to conduct experiments can be high. Significant improvements in scientific software support, beamline-specific data visualization, and analysis tools are required to make a real difference.

2. The Growth, Expansion, and Transition of NSLS Scientific Programs

NSLS programs in small-angle x-ray scattering

SAXS is one of the most active and heavily subscribed NSLS x-ray scattering techniques. At the same time, there is a significant gap between SAXS capabilities at NSLS and those at third generation sources. Most SAXS at NSLS takes place at bending magnet stations, except for Beamline X21, a shared insertion device beamline operating for only half of each cycle.

Beamline X27C, dedicated in large part to in-situ processing of polymers, composites, and biomaterials, has gathered a unique set of sample chambers including dual-cell temperature jump apparatus, tensile stretching apparatus, continuous fiber draw apparatus, fiber gel/melt spinning apparatus, high pressure cell with supercritical fluid, parallel-plate shear apparatus, dynamic rheometer, and stress-control rheometer. The community will clearly benefit from continuing these programs at NSLS-II with a brighter source and new detectors. New detectors will increase data rates by two to three *orders of magnitude*. It will become obvious that data management, visualization, and analysis create the bottleneck for the entire experimental process. Existing data analysis software is inadequate, especially for the noncrystalline scattering patterns of soft matter; the few packages that do exist are designed to analyze individual frames and are limited to particular scattering conditions. There is no support for the hundred- to thousand-frame experiments which can be performed with today's state of the art detectors. Growth and expansion of SAXS programs requires that the facility will explicitly support a data framework including scientific computing support. The infrastructure must include state-of-the-art data storage, retrieval, and visualization, and analysis for at least three applications: SAXS/WAXS of bulk systems; SAXS from complex fluids; and GISAXS of surfaces.

NSLS is moving forward with support for soft matter and biophysics research in the construction of beamline X9. Activities which require new capabilities include the study of directed assembly from nanoparticle and organic molecules, characterization of nano-fabricated devices, and energy-related research areas such as organic semiconductors and photovoltaic devices. The BNL's Center for Functional Nanomaterials has already partnered with NSLS to instrument X9 with support for simultaneous SAXS/WAXS or GISAXS/GIXD, and reflectivity capabilities. NSLS has designed X9 for microbeam focus and a wide energy range on an undulator source, to optimize experiments on thin films, microfluidic devices, small organic crystals, and hierarchically structured materials. X9 will employ some of the most recently developed fast photon-counting area detectors. In all respects, X9 is an excellent test bed for a cutting-edge micro/nano SAXS beamline at NSLS-II, and is designed with an eye to future NSLS-II transition. Scientific computing support would be the most significant additional resource to this program.

Solution SAXS, for the study of protein conformation and related structural biology research, is an area of opportunity for a new program to be nurtured at NSLS and carried forward to NSLS-II. For these experiments, the most important requirements are clean beam, dedicated instrumentation, ease of use, and focused scientific staff support. Up to now, no existing NSLS program has been able to provide a sufficiently strong and coherent effort in this area. Significant interest exists, however, among NSLS staff and users in both the soft-matter and life sciences communities to support a new solution SAXS program. We anticipate that solution SAXS can be an area of real growth if seeded at NSLS and implemented in earnest at NSLS-II.

NSLS programs in liquid interface scattering

Two liquid spectrometers currently operate at NSLS-I: the X22B instrument operating at 8 keV on a dedicated bending magnet source, and the X19C instrument operating at 15 keV sharing a bend source with a topography program. Due to the lower x-ray energy only the vapor or vacuum interface can be studied at X22B, whereas the X19C instrument can be used for studies of liquid/liquid interfaces. The liquid interface scattering community agrees that designing new instrumentation, ideally at NSLS-I, is the most effective foundation for future research. The next generation liquid reflectometer should be designed for on-the-fly data acquisition, and incorporate a 2D pixel array detector with fast readout. Facility-based infrastructure should include Langmuir troughs, liquid-liquid chambers, and other ancillary equipment now typically owned by individual user groups. User-friendly software for analyzing reflectivity and Bragg rods will make the experiments accessible to a broader user base and enhance productivity significantly.

The liquid surface scattering community has identified a need for fast measurements. We anticipate that fast diffuse scattering and grazing incidence diffraction will result naturally from improvements in the source and detectors. The speed of existing measurements can be increased by factors of 10 to 100, compared with the APS. Two different techniques merit discussion: grazing incidence diffraction and reflectivity. The in-plane surface structure of liquid interfaces is obtained by measuring the scattering transverse to a beam which intersects the surface at a small incident angle. Only with microfocusing can the beam footprint be reduced enough to prevent parallax and enable acquisition by a 2D detector. In comparison to present-day instruments which scan a linear detector/Soller slit assembly to achieve the 2D scattering pattern, data acquisition will be orders of magnitude faster, and beam damage correspondingly reduced. Hence, microbeam capability must be designed into the next generation liquid interface station.

Fast reflectivity from liquid surfaces will require one of two new methods that should be developed prior to NSLS-II. Present-day instruments which tilt the beam downward using a single steering crystal move the sample stage, requiring a waiting period to dampen waves produced in the sample. One alternative is to replace the single steering crystal by a two crystal arrangement, in a setup capable of steering the beam to a fixed point for the entire range of incident angles. This enables the sample to be fixed at a single position. In a second method, a broad energy bandwidth beam is reflected from the sample and a fast spectroscopic pixel detector is used in an energy-dispersive geometry. In this case, several serial reflections at different incident angles to cover the complete q range, but experimental signatures over a limited q range can be measured with a single shot. For a typical scan, we estimate that the first method will yield a time resolution of a few minutes while the second will yield sub-second resolution over a limited range in q. This is in comparison to measurements which can take 40 minutes or longer per reflectivity scan using today's spectrometers. The beamline optics and liquid surface scattering instrument at X19C make it an excellent resource for development of a prototype fast reflectometer at NSLS-I.

New liquid interface instrumentation will impact a core community of ~20 PI's who carry out experiments requiring 10 day runs at existing endstations — implying beamtime consumption of 200 days in a year. A larger community measures surface scattering from solid interfaces, notably the liquid–solid interface. For them, liquids instrumentation has certain advantages, such as six-circle reciprocal space access. Outreach to the catalysis and mineral-liquid interface communities is planned. Liquid interface scattering is also conducted with neutrons, in some cases providing critical complementary information to x-ray studies. More neutron scatterers may be attracted to updated liquid surface x-ray instrumentation by the prospect of accessing a larger *q* range for structural analysis, increased time resolution, or the more penetrating high energy x-rays.

NSLS programs in low energy x-ray spectroscopy, microscopy, and scattering

Currently there are at least five NSLS beamlines used for soft x-ray NEXAFS, microscopy, and XPS (U7A, U4B, U12, X24A, X1A). They are used to measure the structure and chemistry of surfaces and bulk, under vacuum and atmosphere for SAMs, DNA, proteins, biomaterials, organic/molecular electronics, polymer surfaces and interfaces, battery materials, catalysts, and many others. These beamlines support a large user community and have substantial scientific impact in basic, applied, and industrial materials research in a broad range of applications. For NSLS-II the existing soft to tender NEXAFS and XPS research communities will utilize the planned NSLS-II soft bend NIST spectroscopy beamline described below. The brightness and intensity of NSLS-II undulators will support high spatial resolution XPS and STXM. Endstations and beamlines are planned for all of these soft to tender x-ray spectro-microscopy innovations.

NSLS already has an excellent x-ray microscopy program at beamline X1A, although much of the soft matter community also uses the ALS x-ray microscopy facilities. The next generation instrument should be constructed at NSLS-II to take advantage of improved polarization control and improved flux. A top-of-the-line instrument for expert users at an EPU beamline and a work horse instrument at a bending magnet beamline similar to the set-up at the ALS is desirable to provide complementary real space information to the various scattering activities. The LOI being submitted by the x-ray microscopy community includes input from soft- and bio-materials researchers, along with life sciences, environmental sciences, and others.

In order to effectively utilize resonant scattering techniques in soft matter systems either at NSLS or NSLS-II, a number of challenges must be addressed. The first is the requirement of an in-vacuum diffractometer, with the corresponding difficulties with efficiently exchanging samples, maintaining hydration of soft materials, and so on. The environmental requirements are quite different from those suitable for hard condensed matter systems, making separate diffractometer chambers necessary.

Significant challenges in the x-ray optics and detection exist. Choices of incident optics include grating monochromators, which preserve the x-ray polarization state, versus the crystal monochromators which do not, but which are required for the higher energies. Suitable phase plates for the higher energies must be developed. Diamond phase plates have already been effectively used down to 3.5 keV. R&D is required for polarization analysis, and for detectors. Silicon pixel array detectors work well for the higher energies, but comparable detectors must be developed for the lowest energies.

Finally, data analysis tools that incorporate full tensor analysis of the complex x-ray structure factor are still at an early stage of development for soft matter systems. The use of changes in polarization state to probe dynamics has yet to achieve proof of concept. There are opportunities for ground-breaking research in this area.

Worldwide, very few facilities are available for research in the 0.2 to 3.5 keV energy range. Establishing such a facility at NSLS would attract an international user community. Construction of this instrumentation at NSLS would motivate the R&D required to effectively utilize the polarization and energy selectivity and high brightness available at NSLS-II. This instrumentation would enable the highly productive NSLS soft matter resonant x-ray program to unambiguously determine orientational order in more complex systems, would launch a soft x-ray resonant reflectivity and scattering program on solar organic polymer films, and would initiate a resonant magnetic scattering program on new magnetic systems (see hard CMP white paper). In addition, the EPU could also serve as an ideal source for the new high-pressure photo-electron spectroscopy instrument under construction at the CFN.

The proposed scattering facilities will complement the spectroscopy facilities under construction at NSLS by NIST to cover the same energy range. The XPS and NEXAFS facilities in particular can be targeted for use by industrial researchers. Since the needs of this user community will likely result from advances in nanoscience and nanotechnology, it will be beneficial to work closely with the nanocenters to develop the most appropriate analytical tools to characterize the systems being fabricated.

3. Proposed Suite of Beamlines

The soft- and bio-materials beamline suite is summarized in the table below. For our community, it is important to recognize the distinction between frontier beamlines where expert users will push the boundaries of what is possible, and the several hundreds of users whose science is adequately served by sources available now. These latter users only require that their programs be supported by dedicated beamlines which are easy to use for the task at hand, and that improvements in detectors and data flow are made. Here, we propose a program with "expert" stations at the frontier, *along with* "high user throughput" stations with dedicated capabilities to support known communities. The development that takes place at the former stations will migrate to larger communities as expertise reaches a wider user group and computing support increases.

Note! The user access model is of significant interest and even *contention* in our community. Huge successes have been built upon the former PRT model, in which the highest performing contributors have guaranteed access to the facility of their own design, and public acknowledgment of the origin of the successful facility. It is perceived, in our community as in others, that without *sufficient intellectual and practical ownership*, designing the new experimental stations will not be an attractive prospect for experts in the field. We urge the facility managements to pursue an ongoing, open-minded dialogue about this, and react to the realities of the research community's needs.

Soft/Bio Beamline	Mission	Source	primary/ secondary	% usage s/bm
1. micro-SAXS	frontier spatial resolution, fast detectors, flexible SAXS/WAXS/GI/XR	hard x-ray undulator	primary; expert users	100
2. SAXS/WAXS	full quadrant state of the art SAXS/WAXS, flexibility in sample environments	3PW on bend	primary; pre-existing SAXS community	100
3. Solution SAXS	dedicated to protein solutions, easy to use	3PW on bend	primary; soft-bio to develop, maintain	50, share with life sciences
4. Liquid reflectometer	state of the art to serve liquid surface community	hard x-ray undulator	primary	100
5. Buried interface spectrometer	frontier instrument to probe buried liquid and solid interfaces	SC wiggler source, 70 keV	primary; soft-bio will develop and maintain	50–100, share *source* (DEI is best partner)
6. Soft / tender x-ray spectroscopy (NEXAFS/XPS)	NIST spectroscopy optimized for light elements and buried interfaces	soft bend 200 eV–5 keV	primary; pre-existing NEXAFS, XPS community	100
7. Tender x-ray resonant scattering	frontier: develop low-energy reflectivity methods	short or long EPU, 1–8 keV	secondary	50, co-develop with hard CMP
8. Soft x-ray resonant scattering	frontier: develop resonant scattering, polarization control	short or long EPU 200 eV–1.5 keV	secondary	50, co-develop with hard CMP
9. High q-resolution surface scattering	Diffractometer with flexible q access; support pre-existing surf. community	hard x-ray undulator	secondary	30–50, share with hard CMP surf. community
10. soft x-ray microscopy	STXM instrument with cryo sample support	EPU and bend instruments	secondary	25, share with life sci, env. sci., hard CMP

4. Beamline Specifications and R&D Needs

Beamline 1: micro-beam SAXS *primary 100%*

This beamline will provide very high flux with micron or submicron spot size, and will be situated on an undulator source in a high-β straight. This beamline will utilize two elliptical focusing mirrors in K-B configuration as the primary focusing optics, and should also be equipped with multiple options for micro-focusing optics such as KB mirror, compound refractive lenses, zone plates, and kinoform lenses. A Si(111) monochromator will be designed to provide 5–30 keV x-rays. R&D efforts will be required to optimize the designs. At present, monochromator and mirror systems tend to provide a slightly lesser energy range, and do not provide good harmonic rejection over the entire energy range specified here. Monochromator crystal gaps, beam offsets, mirror incident angles, and coating options need to be re-examined for future applications. Similarly, although development of new microfocusing optics is active now, parasitic scattering from such optics, especially at low angles, has not been studied in detail.

Modeled on or transitioned from beamline X9 at NSLS-I, the beamline will also have simultaneous SAXS/WAXS, GISAXS/GIXD, and XR capability. R&D is required to optimize detection schemes for these simultaneous scattering modes. Positioning stages with high linear and angular resolution will be implemented. It is also crucially important to develop high quality in-line microscopy to view illuminated beam spots in these experiments. These needs are not unique to NSLS-II beamlines and should be on the agenda for upgrade throughout NSLS-I.

Beamline 2: SAXS/WAXS *primary 100%*

In many cases a double-multilayer monochromator on a 3PW bend source is optimal for soft-matter scattering, where the coarse energy resolution of $\Delta E/E \sim 1\%$ is adequate. However, resonant experiments are also common, in which case a single crystal Si monochromator is required. Both will be incorporated into this beamline. X-ray flux at the sample should be of order 10^{11}–10^{12} ph/s. X-ray energy will be adjustable from 6–20 keV. A collimation system will be designed to determine a maximum spatial resolution of ~100 nm and clean up all parasitic scattering.

Of critical importance is the detector assembly enabling SAXS simultaneously with full-quadrant WAXS detection. The WAXS elements should have a central hole allowing passage of the SAXS signal. R&D is needed to generate a superior design for this, since current designs are sub-optimal. Both detectors should possess single photon counting ability, good spatial resolution, large dynamic range, and 1 to 10 millisecond readout rate. Data throughput in this system will be of order 2 GB/s at a *minimum* (20 MB frame / 0.01 sec). Technology exists which can handle this. The facility must provide it.

The sample stage area will have sufficient space for all the types of chambers in regular use at NSLS-I, with an eye to enabling researchers to dream up new in-situ sample environments for future study.

Beamline 3: solution SAXS *primary, share with life sciences*

A dedicated solution scattering beamline is to be constructed on a 3PW bend. Its priority is to achieve very low scattering background and high throughput for measurements on dilute solutions of nanoparticles, biological molecules, and molecular assemblies. A toroidal focusing mirror is required to capture a fan of 3PW radiation that matches the divergence allowed by collimating slits. This beamline must be equipped with a multilayer monochromator to compensate for the low source flux. The x-ray energy range should be 7–20 keV. Simultaneous WAXS capability here is necessary but not for the full quadrant, since sample scattering is isotropic. Automatic sample handling robotics is required.

Beamline 4: Liquid reflectometer *primary 100%*

The proposed new liquid reflectometer will operate in the 5–20 keV x-ray energy range. The U20 undulator, preferably installed in a low-β straight section, is the preferred source since the typical liquid surface experiment is brightness driven. The experiments require a small, low divergence, high intensity beam because of the very small angles of incidence, the high angular resolution required for small *q*, and the often very weak scattering contrast. A tuning double crystal monochromator and a pair of mirrors in the beamline will maintain a horizontal beam into the hutch. A vertical beam size of 1 to 2 microns is desired with looser specifications on the horizontal size (1 mm might be acceptable). Optical calculations are required for realistic beamline components before deciding on the location and type of mirrors, but it may be possible to use a bimorph vertical focussing mirror and mirror flat, or alternatively two K-B mirrors.

The instrument will be an enhanced version of existing instruments such as the ChemMatCars and CMC instruments at the APS. Modifications will include greatly improved mechanical accuracy to take advantage of the source properties, fast pixel-array detectors, and built-in optical microscopy. A high precision (submicron) sample stage will enable positioning of the small samples made possible by the small vertical beam. The hutch needs to be large enough for the table supporting large sample chambers including a Langmuir trough, and capable of reaching horizontal scattering angles corresponding to molecular spacings at 5 keV. We also plan to install a small ventilated fume hood in the hutch for sample prep or in-situ operations where small amounts of vapors must be routed. The endstation must be sited accordingly for the large hutch.

Beamline 5: Buried interface reflectometer *primary, share source with life sciences (DEI)*

The high energy instrument (greater than 35 keV, with 70 keV preferred) is envisioned as a side station taking part of the fan of a superconducting wiggler source. The DEI beamline being developed by the life sciences community is a good partner for this source. A double crystal monochromator will bring photons to the hutch. Multiple compound refractive lenses in the beamline will focus to provide 5 µm vertical by 50 µm horizontal beams. In the endstation, Laue steering crystals will provide a fixed sample geometry for reflectivity, grazing-incidence diffraction, and diffuse scattering. This will be the only dedicated high-energy beamline in the world for the study of buried interfaces.

Beamline 6: NIST Soft and tender x-ray spectroscopy beamline *primary 100%*

NIST plans to build a soft to tender (~100 eV to ~5 keV) NSLS II soft bend beamline for NEXAFS and variable kinetic energy XPS. Zoom focusing is planned, small beams when needed (~ 1 µm) to probe surface inhomogeneities, and large beams to prevent sample x-ray damage. The beamline will support surface and bulk systems, under vacuum and atmosphere. The proposed beamline has two monochromators, a VLSGM and a DCM to seamlessly span the entire energy range under computer control for fully automated operation. The broad energy range will allow full control of the electron kinetic energy in XPS in order to tune the depth selectivity to probe the length scales of buried interfaces. The NEXAFS endstation will incorporate all of NIST's current and new state of the art detectors for electron and fluorescence yield detection as well as high throughput data acquisition methods pioneered at NIST's U7A beamline. A full field NEXAFS imaging endstation is planned to apply the combinatorial NEXAFS method (currently under development by NIST at U7A) for sample arrays (DNA and catalysts) and gradient structures.

Beamline 7: Tender x-ray resonant scattering *secondary, co-develop with hard condensed matter*

In order to do an unambiguous structural determination of orientational ordering in soft or biomolecular systems, which often include glide and screw plane symmetries where the sense of the screw axis impacts biological function, it is necessary to measure the complete tensor structure factor. This requires the use of linear-horizontal, linear-vertical, and circularly polarized x-rays. This is most effectively done using an EPU. The EPU can be a long or a canted-short EPU. For resonant scattering, precision control of the polarization state and energy of the x-ray beam as well as access to adequate beam time are equally important as brightness; hence, the soft matter resonant scattering program would not be seriously impacted if developed on a lower-brightness canted-short EPU device. The beamline would use crystals with a large lattice-spacing to minimize the Bragg diffraction angles. This is essential to preserve the polarization state of the x-rays. The beamline would also incorporate K-B focusing optics to provide a micron-sized beam to explore changes in orientational texture in the vicinity of defects. In addition to measurements of static structure, the beamline would also be used to probe orientational fluctuations and intermolecular correlations. The beamline could be co-developed and shared with the hard CMP community; however, the different sample environments necessitate the use of separate in-vacuum diffractometers.

Beamline 8: Soft x-ray resonant scattering *secondary, co-develop with hard condensed matter*

A soft x-ray (long or short) EPU beamline in the 200–1200 eV energy range would provide unique capabilities and enable, for example, high brightness techniques such as resonant XPCS to probe orientational dynamics. Multiple grating, grazing incidence beamline optics that preserve polarization, provide a resolving power of >4000, and are capable of microfocusing are required. This beamline would also enable microbeam resonant reflectivity measurements on nanopatterned assemblies. An in-vacuum diffractometer, with an efficient sample exchange mechanism and special sample environmental controls (e.g. hydration) will be developed. Efficient multiplexed data acquisition with an area detector is highly desirable. Due to the large scattering angles at low photon energies (full angular range to 180° backscattering is desired), the incorporation of such detectors for multiplexed data acquisition will be challenging and represents a major opportunity for advancements.

Beamline 9: High *q* resolution surface scattering *secondary, share with hard condensed matter*

The soft- and biomaterials community will retain a need for workhorse scattering and diffraction, which can be performed on the next generation of six circle diffractometers similar in operation to those of the hard condensed matter community. We estimate that our community might use 30 to 50% of the time on a well equipped diffractometer which provides basic high *q* resolution measurements, high reciprocal space access, and support for small chambers.

5. Recommended Transition/Construction Sequence

SAXS instrumentation:

The X9 beamline at NSLS is still under construction. Its bimorph KB focusing mirrors are state-of-the-art quality and its endstation instrumentation is well suited to work with small beams. X9 is therefore a candidate to be moved to NSLS-II and become the micro-beam line. The current X9 monochromator will not be usable at a full sized NSLS-II undulator. More information regarding smaller, canted undulator devices will enable us to determine whether X9 can be appropriately transitioned by sharing a source in that manner. Conversely, optical elements may be upgraded to future designs. MIE funding may be sought for the necessary upgrades.

The SAXS/WAXS beamline should be designed and constructed at NSLS-II as an MIE project with input (BAT participation) by groups who may have relevant sample apparatus to contribute.

The solution scattering beamline is very specialized, and the life sciences community is anticipating a large increase in demand for this capability. Since its performance on a 3PW source at NSLS-II is comparable to that on a bend magnet at NSLS-I, this beamline can be constructed now and transitioned to NSLS-II.

Liquid interface instrumentation:

Beamline X22B will serve users until NSLS is turned off. X22B is fully subscribed by requests, making it important for updated instrumentation to be in place early at NSLS-II, a recommended MIE priority. The high energy instrument is a second priority and should be prepared by R&D beginning at NSLS-I. A transition plan for energy dispersive fast reflectivity instrumentation will depend upon successful prototyping at NSLS-I.

Soft bend and EPU 0.2–5 keV x-rays:

The new NIST soft/tender x-ray spectroscopy beamline will be transitioned to an NSLS-II soft bend x-ray beamline.

We recommend the construction of a short EPU device at the NSLS that would be transitioned to become part of a canted-pair of EPUs at NSLS-II. This short EPU would be designed to cover the energy range from 250 eV to 5 keV, and be used for the development of polarization-analysis techniques, sample cells, detectors, and in-vacuum diffractometers optimized for the soft and tender x-ray ranges.

High q resolution hard x-ray scattering:

Several NSLS beamlines support this technique for hard and soft CMP, and none are planned to be transitioned. The hard CMP community's workshops have described upstream stations with "simple" diffractometers to support basic structure measurements as companions to downstream series installations of large dedicated chambers. We support this model and believe soft CMP experiments would subscribe these upstream diffractometers 30–50%.

6.. Facility Infrastructure at NSLS-II

At full build-out the soft- and bio-materials community will operate nine beamlines simultaneously, hosting a population of approximately 30 experimenters. Planning 10 guest offices for users and 26 offices for beamline staff and laboratory stewards, we arrive at 36 offices for the soft/bio community, which is one half of an upper floor of an LOB of current design. A conference room with display screen and video gear, interaction areas, kitchen, and so on will be shared with the other LOB residents. The building should be "legal" for 150 people: 72 staff and again as many users. All rooms and areas should have telephone and ethernet connections. The building should have one computer room for data retrieval and analysis, with room for 12 workstations. Office support such as photocopy and fax are needed. The building should have a loading dock, with shipping and receiving operations available during business hours. The outdoor dock area needs to have liquid cryogen and dry gas delivery and storage, in accordance with all regulations (including, flammable gases separate from smoking areas!). Bicycle parking and outdoor interaction areas are also desired.

The experiments emphasize chemical preparation and user-contributed sample housings and ancillary equipment. Wet laboratory space must be divided into compatible activities and managed by a laboratory steward. Some apparatus and equipment can be provided by the facility, with sufficient open space reserved for user apparatus. A 1200 square foot block of interconnected lab space can serve this community. A typical layout for a 20 by 20 foot lab has one wall dedicated to fume hoods, sinks, eyewash/shower, and storage. Bench space can be arranged in such a space to create three to four additional work areas where simultaneous activities can take place. Thus, 1200 square feet (three such rooms) can support 10 to 15 work areas divided among (1) harsh chemistry and nanoparticle prep, with properly filtered hoods, toxic gas vent, and appropriate waste storage; (2) biochemistry "clean area" with its own work space, hood, storage, and specialized equipment such as incubators; (3) generic prep space for short-term use; (4) reserved prep space for users requiring ongoing activities prior to or for the duration of a run, as for Langmuir troughs, or in-situ chemical reaction apparatus; (5) small instrument space for facility-owned centrifuge, mass balance, sonicators, etc.; (6) large instrument space for facility-owned microscopes or other instruments. The labs need dedicated space for gas bottles and potentially should have dry nitrogen feeds and compressed air in each work area. Wet labs need networked computers for MSDS retrieval, monitoring of the ring status and experimental floor, and to control experimental equipment.

The need for mechanical setup space is also significant. A space of 800 square feet may be adequate, to subdivide into distinct activities: (1) staff space, with locked-out machine tools, hand tools, hardware storage, and enough bench space to build and fix beamline equipment; (2)

parking space (possibly locked or caged) for wheeled equipment in occasional use: pumps, chillers, spare detectors; (3) generic workbench / hand tool areas for brief use by experimenters; (4) reserved bench space for users with experimental apparatus, prior to or during a run. The latter is particularly important for soft- and bio-materials research, which makes use of presses, rheometers, stress-strain apparatus, and so on which is best housed in a mechanical space and likely to require work and testing prior to the beamline experiment. Mechanical setup rooms need networked computers for web access, reading technical manuals, monitoring of the ring status and experimental floor, and to control experimental equipment.

It seems likely that our combination of chemical and mechanical laboratory spaces, 2000 square feet total, will occupy approximately half of the lab space in the LOB ground floor design currently depicted. Sharing the LOB with hard CMP may be especially efficient.