Development of Dedicated SAXS Beamlines at 12-ID with Canted Undulators

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Introduction

The APS Strategic Plan calls for 12-ID to become a dedicated small angle X-ray scattering (SAXS) facility. Currently the SAXS facility, originally funded from the FY 1996 DOE Scientific Facilities Initiative, has steadily grown to now represent 50% of the usage of the 12-ID undulator beamline. The over-subscription for SAXS experiments at the APS > 100% implies that the fraction of beam time devoted to SAXS experiments would have been much higher if more beam time were available. Since the FY 2002 allocation cycles, the demand for SAXS for users at 12ID has consistently exceeded the available time by a factor of two. With the current instrument configuration oversubscribed, the only way in which the present SAXS community can continue to grow and new users can take advantage of these new capabilities is to provide simultaneous operation of the three 12-ID experimental stations by adding canted undulators and beamline optics which will allow the three experimental stations to operate simultaneously. This proposed enhancement would provide the APS with dedicated SAXS beamlines that would better serve the general user community in a number of areas in materials science, chemistry and biological materials. This plan was endorsed by the SAC review of sector 12 in March 2005.

A canted undulator front end will be installed and the current undulator A and a second 2.7 undulator will be installed. The vacuum chamber for the canted undulators and one of the shorter undulator A's was installed in FY2006. One of the resulting undulator beamlines will use a modified version of the current monochromator and mirror system which includes the pink beam mirror that was installed at the end of FY05. This beam will feed 12-ID-C and be used for anomalous, time-resolved, grazing incidence SAXS and USAXS. The second beamline will serve as a dedicated SAXS instrument in 12-ID-B.

Science

There are a number of important scientific areas where SAXS is and can make important contributions. The scientific impact will be increased by the increase in beam stability and flux as proposed in this project. Descriptions of several important science areas are given below.

Nanoscience - By selectively incorporating chemically modified nanoparticles in one phase of the block copolymer and exploiting the rich phase behavior of the copolymer at nanometer length scales, a number of nanocomposites with ordered 2-D and 3-D arrays in thin film morphology can be synthesized. In order to obtain fundamental understanding of the effects of nanoparticle loading on the phase behavior of block copolymers, nature of dispersion of the nanoparticles and the nature of interaction at the polymer/nanoparticle interface systematic GISAXS experiments are being conducted. This new approach to preparing nanocomposites is to improve the dispersion of nanoparticles so that there is no aggregation. This is extremely important given that dispersion of nanoparticles has continued to be one of the major bottlenecks in the development of nanotechnology.

Catalysis – When applied to high surface area and flat catalytic materials SAXS and GISAXS, respectively, can provide both *ex-situ* and *in-situ* information on cluster size, shape and inter-particle distance. The ability to compare the two classes of materials will validate the formation of identical structures on the two support morphologies. GISAXS can also give depth profile information, and the aspect ratio (height/diameter) of a cluster can be calculated from the GISAXS data to obtain the interfacial energy. GISAXS is ideal for *in-situ* studies since it is very sensitive to surface species and there is less parasitic scattering resulting from the substrate compared to a conventional direct-transmission SAXS experiment. GISAXS has been used to study the thermal stability and reactivity of Pt, Au and Ag clusters deposited on a variety of surfaces with insightful results. ASAXS refers to the extension of standard SAXS experiments in which the energy of the probing X-rays is tuned near the absorption edge of an element in the sample. This method overcomes the problem of separating the scattering of clusters from that of the support. For the first time anomalous GISAXS has been obtained on metal clusters on surfaces and has provided significant insight into the structure of very small metal clusters on surfaces.

Micro SAXS - Often SAXS samples are not homogeneous which blurs scattering patterns and adds difficulty in analyzing data. For example, the sedimented colloidal crystals could be a glassy-aggregate form as well as small-sized crystalline. If the glassy-aggregate form dominates the crystalline one it typically is hard to determine the structure of the nano-crystal. Thus, it is necessary to reduce the scattering volume and find the right crystalline spot by using as small a beam as possible. The idea behind this is identical to micro-crystal diffraction. If a crystal is hard to grow to an acceptable size then the alternative is to reduce beam size. Smaller beam size with fine sample stages will certainly enable searching for the spot on a sample which has the best structure. By combining microscopy it is possible to resolve an area of interest, and by scanning the sample one could get the spatial distribution of the structure as well, which will provide a scattering/diffraction contrast image. The strongest advantage of X-ray over other microscopy sources such as electron, visible light, or cantilever is the high penetration. By using X-ray one could study live samples, which must be kept under different environments, such as aqueous solution, solvents, high humidity, low and/or high temperature.

USAXS - Structural biomaterials such as bones, dentine, cartilage, etc. are generally inhomogeneous and the structural porosity plays a major role in their performance. Indeed, some diseases demonstrate themselves in porosity changes in these structural elements of the human body. Understanding the porosity, and the changes to it caused by various environmental or biological influences, is imperative for new developments in human medicine. Previous tests have shown that current USAXS is not suitable for these studies. However, increasing the X-ray energy will both reduce the effects of multiple scattering as well as improve penetration of the X-rays necessary for these studies to become feasible. Further, while these materials are currently studied by diffraction-enhanced imaging, there have been only limited attempts to use USAXS imaging. Simultaneous availability of diffraction-enhanced imaging, USAXS imaging and selected area USAXS would be an excellent combination resulting in a unique instrument not available anywhere else.

Anisotropic nano materials such as “forests” of carbon nano tubes or carbon nano tubes in suspensions and polymers require use of 2-D collimated USAXS to study this anisotropy. At the same time, these structures can often be highly inhomogeneous and without imaging techniques these studies can be really challenging.

Added Value of the Mid-term Upgrade

By bringing the SAXS user community together at one sector, a gain in efficiency and thus faster experiments and the ability to run more experiments is expected. The increasing demand for SAXS beam time has been driven by research into the structure and dynamics of nanoscale materials for which SAXS is an extremely valuable probe, by an increased interest in soft matter and by increased usage of the 12-ID SAXS facility for time-dependent structural investigations.

General user experiments have included these areas, as well as the extensive use of the 12ID SAXS instrumentation for biological research. Increasing interest in time-dependent SAXS experiments has led the CEP group to add a pink light mirror system to the 12-ID beamline. This provides new experimental opportunities for small angle scattering experiments. With the development of dedicated instrumentation for GISAXS at 12-ID the beamline will also serve as an important complement to the infrastructure for the Center for Nanoscale Materials at Argonne and will further increase the demand for this beam line.

Expected user communities

The over 300 users who have done experiments on the12-ID SAXS instrument represent more than 42 U.S. universities, nine national laboratories and government institutions, six industrial research laboratories, and researchers from 11 foreign universities and laboratories. Over 150 publications have resulted from these studies in such journals as JACS, PNAS, PRL and Macromolecules.

Enabling technology and infrastructure

Modifications of the 12-ID-C beamline will enhance SAXS and ASAXS operations. Based on our experience with SAXS experiments operating under the current configuration, the new design of 12-ID-C will have improved beam stability, lower background and increased flux. A necessary requirement for anomalous experiments is good beam stability as well as high energy resolution. Beam movement makes it very difficult to measure very small changes with changing energies which is usually the case with ASAXS experiments. The monochromator in 12-ID which will be used for 12-ID-C was never designed for spectroscopy, but other designs at 20 and 11-ID have proven to be stable and modifications based on them will be used for 12-ID-C monochromator.

The second goal is lower background which will be accomplished by the reduction of parasitic scattering. The requirements for the beam-defining slits are important for both stability and background issues. For the third goal, flux can be increased by better control of the horizontal mirror and by replacing the vertical mirror with a longer one. At 12-ID-C the mirror has to be changed from an up-reflecting mirror to a down-reflecting mirror. Since the beam footprint of the current mirror, with a length of 40 cm, is only 0.5 mm it would be preferable to have a longer mirror of about 60 cm. At a minimum incidence angle of 2.5 mrad this would accept the full vertical beam size up to the mirror cut-off of ~35 keV.

The final goal is to provide improved detectors which are not part of this project but will be described in this document. One detector is a four CCD mosaic detector which has been constructed by the BTS group in XSD. A design of this detector, which will work in a vacuum, will be constructed for the 12-ID-B instrument where the detector-to-sample distance will be able to be automatically controlled by the user. Another detector will be designed for time-resolved SAXS using the pink beam. This will be highly beneficial for studies on low contrast samples, such as nanometer or subnanometer particles, monolayer thick organic samples, and proteins in solution.

The USAXS instrument of this upgrade can be “faster, smaller and more capable”. Replacement of the main rotational stage by a new generation linear motor-driven stage and associated changes to the crystal system will reduce the current about 15 minutes per sample measurement time, potentially to as low as 1-2 minutes if scanning on-fly is implemented. This will increase the instrument throughput and will significantly reduce the dose in the sample. Change in geometry and new design of the detector and analyzer stages should fully enable automatic geometry changes as fast as a few seconds. This will open the field of USAXS and diffraction-enhanced imaging to all programs and will create an instrument which will be unique world-wide. Installation of USAXS on the beamline, with horizontal focusing and changes to the 2-D collimated geometry, will significantly improve the quality of the data obtained by this mode and will enable studies of anisotropic materials with small beams. This will enable characterization of such materials as advanced layered systems with micron-sized layers. Higher resolution of the new analyzer stage and improved stability of redesigned stages will enable increasing the operating energy range of the USAXS instrument to at least 30keV.

Partnerships

We have over 300 users and expect to grow this community whose growth has been hindered due to the lack of SAXS beamtime at 12-ID. Select current users include:

Sebastian Doniach – Stanford University
Michael Wasielewski – Northwestern University
Barbara Wyslouzil – Ohio State University
Joseph Hupp – Northwestern University
Chad Mirkin – Northwestern University
Joseph Calo - Brown University
Gregory Beaucage – University of Cincinnati
Hector Lorenzana – Lawrence Livermore National Laboratory
Pehr Harbur – Stanford University
Gerard Wong – University of Illinois UC
Balaji Narasimhan – Iowa State University

SAXS Advisory committee - David Cookson, Australian Synchrotron; David Teide, ANL; Andrew Allen, NIST and P. Thiyagarajan, ANL

Industry and technology transfer

We do not anticipate any technology transfer.

Estimated budget

The total cost for this project is estimated to be $3,028K with $623K having been spent on the project.

1. Canted undulator and front end modifications –	685K
2. New optical hutch and shielded beam pipe –	163K
3. New side-bounce monochromator and its vacuum components -	800K
4. New beamline optics (mirrors) –	530K
5. New detectors, detector vacuum chamber and electronics –	560K
6. Beamline components (utilities, vacuum components, motors) –	290K
Total	$3028K

To operate the beamline we will need at least two additional staff members. It is projected that 3.95 FTEs of engineering and technician help will be required.