Beamline 4: Small and wide angle scattering

04

Potential Research Fields

Life sciences

- Biological research and drug design

Physical sciences

- Forensics
- Advanced materials
- Functional polymers
  - Ceramics
  - Nanomaterials and composites
- Earth sciences
- Oil and gas production and distribution
- Agricultural technology
- Food technology
- Chemical reactions and catalysts

Introduction

Studies of the structure and dynamics of large molecular assemblies in environments such as solutions are key to understanding living organisms and complex materials such as polymers, colloids and emulsions. For this reason, small angle scattering (SAXS) and wide angle (WAXS) diffraction, normally combined on one beamline, are recognised as two of the most important synchrotron applications.

At the Advanced Photon Source, nine of the twenty-five undulator beamlines are capable of performing small angle scattering, and the situation is similar in most synchrotrons around the world.

Advantages of a Synchrotron Source

A major advantage of a synchrotron source is the ability to study processes that are changing with time because of the extremely high beam intensities. Dynamic processes are of enormous interest in both the life sciences and physical sciences, and they represent an area where synchrotron sources are contributing valuable knowledge, unobtainable by other experimental techniques.

User Community

There is an active community of researchers using small angle scattering in a number of fields. Currently in Australia there are several laboratory-based SAXS instruments. Access to synchrotron SAXS is available primarily to Australian users through the ASRP to the newly-commissioned instrument at Sector 15 at the Advanced Photon Source (ChemMatCARS) in Chicago. This instrument has not been available for long and already demand exceeds supply as researchers take advantage of the benefits of SAXS on a third generation source.

X-ray and neutron scattering methods complement one another, and the simultaneous refinement of data produced using a combination of both methods on the one system is very powerful. A small angle neutron scattering instrument to be built at the Replacement Research Reactor at Lucas Heights will augment and complement the Australian Synchrotron SAXS/WAXS beamline. The major benefit of such analysis is the removal of potential ambiguity in the interpretation of data.

Research Applications

SAXS/ WAXS studies have broad applicability across a range of materials and biological sciences. Examples of fields of science and technology that can benefit from these methods are:

- polymers
- mesoporous materials
- self-assembled systems
- food science
- fibres
- colloids
- composite materials
- proteins in solution
- membranes
- crystallisation studies

Biological sciences

Small angle scattering (SAXS) can now determine the shapes of macromolecules in solution, and provide information on the assembly of protein components in functional units. This information is being used to determine what changes in association are relevant to the function of these complexes. This has been useful in understanding biochemical regulation by providing insights into domain reorientation and protein–protein interactions in cellular signalling. The shapes of these assemblies can then be used to construct atomic models based on the structure of the individual components, or used in phasing crystallographic data on these complexes.

The dynamics of many important biological processes, such as protein folding, are studied by SAXS. For example, several folding intermediates have been detected in the re-naturation of apo-myoglobin and lysozyme.

The dynamic processes involved in conformational diseases such as prion diseases and neuro-degenerative disorders are important in the understanding of how these diseases arise. In prion diseases such as bovine spongiform encephalopathy (mad cow disease) an 'infectious' form of the prion protein causes nucleation of toxic oligomers of normal protein by altering the conformation of normal proteins. This process is also thought to take place in Alzheimer's and other neurodegenerative diseases. Time resolved SAXS is one of the few techniques that can provide direct information on the rate and mechanism of protein oligomerisation during conformational transformation.

Nanotechnology

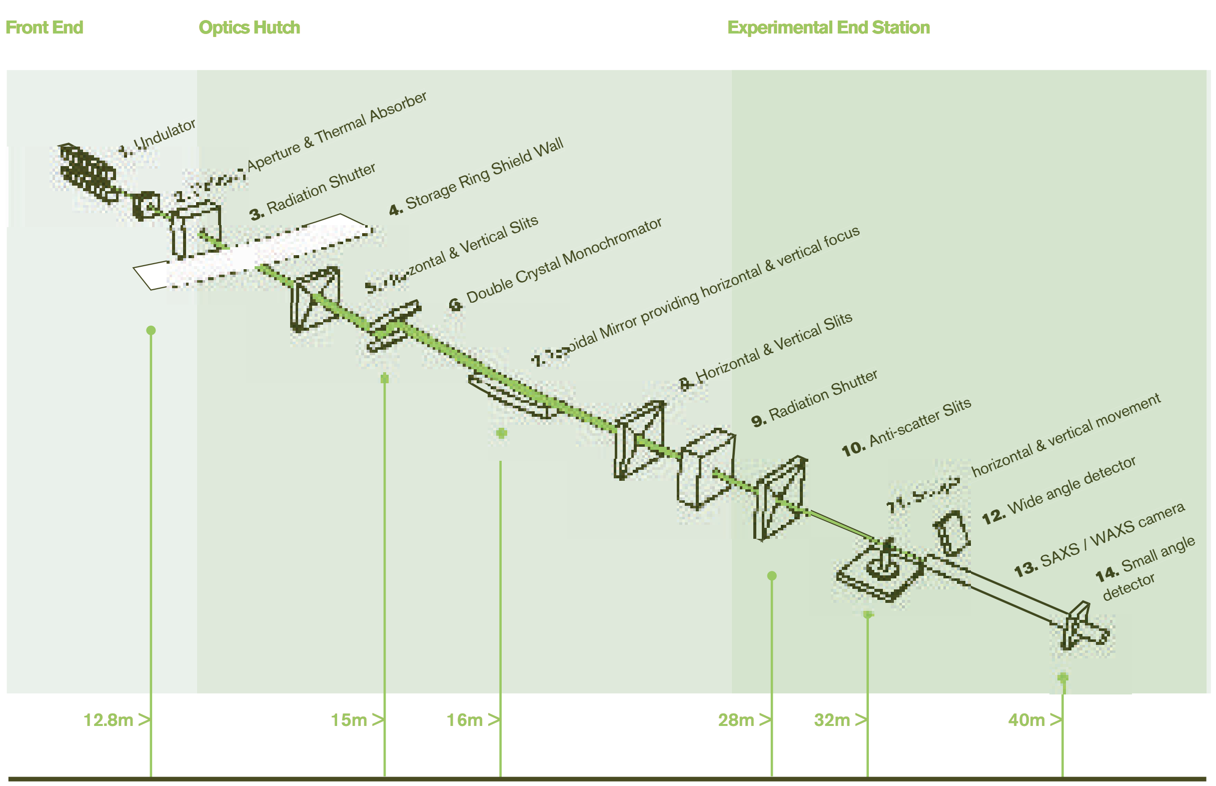
The application of biomolecules to nanotechnology, for example in biomaterials, also relies on an understanding of the basic structures and dynamics of large assemblies, for example the behaviour of liposomes, which is likely to grow in importance for controlled drug delivery. Such systems can be readily studied using SAXS, in a far more direct way than any other method. Self-assembled systems are an important way of manufacturing nanomaterials, for example the clustering of fullerene C60 and higher fullerenes, which has implications in their purification and in building up novel materials. SAXS and WAXS are important techniques for studying the hierarchy of interplay of the components and the formation of complex arrays.

Fibres have a wide range of applications in nanomaterials depending on their degree of entanglement and alignment. For example, the interplay of carbon nanotubes in building up materials with novel function, which again is best studied by SAXS/WAXS techniques.

Micromolecular assemblies and protein folding and unfolding are growth areas in structural biology.

Polymer science and engineering

Polymers are long chain molecules that play an important role in biology, medical applications, new materials, biotechnology, and nanocomposites. The final properties of the polymeric materials are determined by their nanostructure. Australian scientists are at the forefront of many areas of polymer science.



BEAMLINE 4 SAXS / WAXS

Figure BL4.1. *Schematic of the SAXS/WAXS beamline*

An area in which SAXS will contribute greatly is that of polymer blends. Most commercial blend systems contain a polymeric surfactant, or compatibiliser, between the two phases of the blend. These are formed by the in situ reaction of the two components. This affects the chemistry and hence the phase behaviour and crystallisation.

Food science

The melting of gel structures is an important part of the food industry where gels such as amylopectins are used as stabilisers and bulking agents. Recent work has been directed at exploring the mesoscopic structure of starch granules, the impact of biochemical changes in the enzymatic pathways involved in starch deposition in the plant on the internal granule structure and the consequences for breakdown during gelatinisation and other processing treatments.

Other polysaccharide gels need to be further characterised and their melting properties compared to their rheological parameters. In particular, by carrying out x-ray rheology experiments as a function of temperature, it should be possible to quantify the strength and number of the crystalline junction zones as they break down under shear. As rheology is frequently used to characterise food gels, but without the means of unambiguously moving from this information to an understanding of structure, this capability will be invaluable.

Beamline Design

The SAXS/WAXS beamline should make use of the best collimated beam possible with the highest possible flux at the sample position. It will be driven from a 22 mm period in-vacuum undulator.

It will have a useful working energy range of 5.5–20 keV and can be easily tuned.

The beamline will be capable of measuring data at low and high angles simultaneously, and rapidly enough for time-dependent studies. The design will enable incorporation of a USAXS (Bonse-Hart) instrument for ultra small angle x-ray scattering, and allow for the incorporation of additional focussing elements with little or no modification to the underlying optics.

The end station should provide for the ability to change camera length quickly and for rapid flight path evacuation. A hutch large enough to allow in situ measurements of processes, such as extrusion of polymers or food gels, is important.

Beamline 4 – Small and Wide Angle X-ray Scattering	
Source	In-vacuum undulator (22 mm period)
Energy range	5.5–20 keV
Resolution $\Delta E/E$	$< 10^{-4}$
Beam size at sample (horizontal × vertical)	200 × 100 micron (or smaller)
Q range	0.001$Å^{-1}$ – 12.0 $Å^{-1}$ (SAXS); 5 $Å^{-1}$ (WAXS)