Beamline 6: Soft x-ray spectroscopy

Potential Research Fields

Life sciences

- Biotechnology and bio-sensors

Physical sciences

- Forensics
- Advanced materials
  - Functional polymers
  - Ceramics
  - Nanomaterials and composites
  - Metals and alloys
  - Micro-electronic and magnetic materials
  - Biomaterials
- Mineral exploration and beneficiation
- Earth sciences
- Agricultural technology
- Chemical reactions and catalysts

Advanced manufacturing

- Production of micro-devices

Introduction

Soft x-rays are generally understood to be x-rays in the energy range 100–3,000 eV. They have insufficient energy to penetrate the beryllium window of a hard x-ray beamline but have energies higher than that of extreme ultraviolet light. Soft x-rays cover an energy range of importance for spectroscopic studies of many elements in the periodic table. They are well suited to characterising surfaces and near-surface interfacial layers.

The soft x-ray beamline will be set up primarily for XAS of low atomic number elements and x-ray photoelectron spectroscopy (XPS), although there are other significant research applications such as photo-desorption and threshold x-ray excited Auger electron spectroscopy (XAES).

There will be two end stations to enable experimental flexibility, with the second end station capable of XPS and XAS mapping of samples in special atmospheres at pressures of up to 20 torr.

User Community

Australian and New Zealand researchers carry out their synchrotron soft x-ray spectroscopy wherever they can obtain beam time, including Taiwan, Korea, Japan, Germany, USA, Sweden and France. Much of the research is carried out as part of an individual collaboration, and not necessarily funded through the ASRP or even the Access to Major Research Facilities Program. Thus, to some extent, current demand is not fully visible nor fully satisfied.

All soft x-ray beamlines are currently oversubscribed, and very few end-stations available to Australian researchers meet their requirements. Australians are carrying out some cutting edge work, but at present, progress is limited by insufficient access to suitable beamlines.

If only the main user groups currently seeking access to beam time at synchrotrons are considered, it is estimated that the proposed soft x-ray beamline equipped with the ASRP end-station that is currently under construction for initial installation for use on the Taiwan synchrotron would be fully utilised from the outset. The current user base primarily consists of approximately 15 user groups. The limited size of this user base largely reflects the absence of soft x-ray spectroscopic facilities. It is estimated that with better access the potential user base would consist of 30 groups from 18 universities, several CSIRO Divisions and ANSTO, with industry working in association with some of these groups.

Research Applications

Synchrotron soft x-ray spectroscopy provides distinctive information for numerous research areas ranging from fundamental studies in solid state physics and nanotechnology to applied chemical problems in catalysis and coal combustion.

Earth and environmental sciences

Synchrotron soft x-ray techniques are already opening up new ways to address the complex problems arising from earth resource utilisation, and this contribution is expected to increase in areas such as environmentally sustainable ore extraction, mineral processing, coal combustion and soil use.

The surface chemistry of metal sulfides is of major importance in the separation of the valuable and unwanted components in base metal ores, in the hydrometallurgical processing of a concentrate to produce the corresponding metal from the sulfide, and in the leaching of rejected material in waste heaps. Since the application of synchrotron XPS to mineral fracture surfaces, the importance of surface chemical states arising from relaxation of the outermost layer following fracture has become evident.

The enhanced surface sensitivity provided by synchrotron XPS, as well as the ability of angle-dependent x-ray absorption near edge structure (XANES) to reveal orientation, have also assisted elucidation of the mechanism by which flotation reagents interact with the surface of minerals.

Determination of the chemical forms of heteroatoms, such as nitrogen, in a large molecular weight or complex material such as coal is a case where soft x-ray absorption spectroscopy is used to complement conventional XPS for low atomic number non-surface chemical characterisation. This information is sought in research to minimise the generation of undesirable species such as NOx in coal combustion.

The ambient mapping facility on the second end station will enable measurements to be made at pressures of up to 20 torr with high spatial resolution. It will provide maps of surfaces when exposed to a variety of atmospheres including water, oxygen, helium and nitrogen. Thus, for instance, it will be possible to follow mineral oxidation, hydrolysis or collector adsorption in real time.

Physical and material sciences

A niche area in which Australia has been successful to date is the development of thin film materials for electronic and optoelectronic devices. Thin films of materials with particular chemical and/or physical properties such as piezoelectricity are typically deposited onto an appropriate substrate by one form of chemical vapour deposition, and during the development phase for both precursor and deposition conditions, the physical and chemical properties of the film must be determined. Variable-angle XAS from a synchrotron source can augment initial conventional XPS analysis to reveal the orientation of film crystallites.

Chemical and biochemical sciences

In many chemical, biochemical and earth science-related systems it is essential to obtain information about a species while it is in contact with an aqueous or other liquid environment and while it is at a particular electrochemical potential. A wet cell for non-microscopic XAS will be of considerable interest to researchers in a number of fields. In particular, with silicon nitride cell windows, the carbon K-edge near 0.3 keV can be studied, and the region between the carbon K-edges and the oxygen K-edge near 530 eV is often referred to as the 'water window'. It is expected that it would be possible to investigate solid electrode surfaces and even particulate slurries, with the electrochemical potential established by redox reagents in the liquid flowing through the cell.

For materials that can be investigated under ultra high vacuum, XAS is able to provide chemical information that is difficult to obtain by non-synchrotron techniques such as conventional XPS.

For biological samples that need to be kept in damp conditions, or where it is desired to study the action of catalysts in special environments, the ambient mapping facility will be used.

Beamline Design

Energy range

In order to achieve an x-ray beam of adequate stability and energy resolution, it is considered that the energy range sought should be restricted to 100–2500 eV.

The insertion device will be a variable polarisation undulator, capable of supplying linear vertical, linear horizontal, left and right circularly polarised light. The device will work in the 1st, 3rd and 5th harmonics. Limited tapering of the undulator is desirable for producing a broader range of energies in the harmonic peak, but is not essential. It is expected that it will be an 'Apple' type, as fast switching of polarisation is not needed. The device may have either a single period or consist of two modules of different periods to cover the entire energy range required. A design study of the insertion device will provide quantitative data for deciding the preferred specification.

Such an insertion device will meet the needs of the x-ray absorption community by allowing the measurement of linear and circular dichroism without the necessity to rotate the sample.

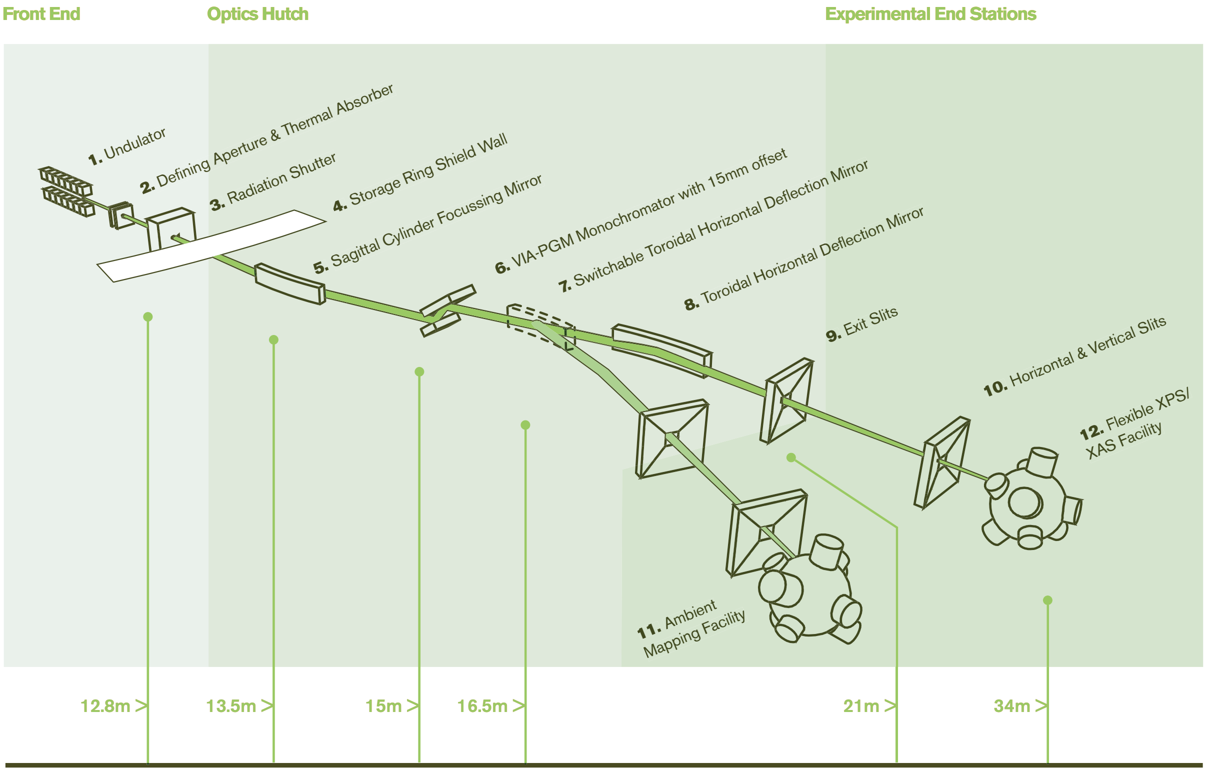
Elliptically polarising undulators are currently the most advanced general-purpose insertion devices. The provision of circularly polarised light ensures that the beamline will be sufficiently flexible to cater for future developments in the scientific areas of interest to the Australian user community.

The resolving power will be 20,000, which is relatively easy to achieve with modern monochromators. In addition there will be provision for lower dispersion gratings with reduced resolution but correspondingly higher flux for experiments that require maximum flux at moderate resolution.

Special features

A soft x-ray beamline and end-station must be maintained under a clean, ultra high vacuum (UHV).

There must be vacuum continuity between the analysis chamber and the synchrotron ring. Therefore, the vacuum in the beamline and end-station must be comparable with that in the ring, in terms of both pressure and quality. It is important that the UHV in both the beamline and end-station be as carbon-free as possible, not only to maintain the reflectivity of the optical components at higher photon energies, but also to facilitate XAS at the carbon K-edge.



BEAMLINE 6 Soft X-ray

Figure BL6.1. *Schematic of the soft x-ray spectroscopy beamline*

The necessity to work under clean UHV conditions means that synchrotron soft x-ray spectroscopy is more demanding and time-consuming than hard x-ray spectroscopy. As a consequence, typical beam access periods are longer, and fewer users can be accommodated. The strategy proposed to address this problem is to have two end-stations, in a branched rather than in-line configuration, sharing one monochromator. The two end-stations proposed are the ASRP soft x-ray spectroscopy end-station, and the other an XPS/XAS end-station optimised for industrial (especially minerals) research. A mirror could switch the radiation between the two end-stations, so that measurements could be made in one end-station while specimen preparation or vacuum recovery was taking place in the other.

In addition, allowance will be made for the ability to connect specialist end-stations in the future.

The soft x-ray spectroscopy end stations

The ASRP soft x-ray spectroscopy end-station is currently being built in Germany at a cost of ~$A1m, and is scheduled to be completed by August 2004. It will be used at the NSRRC in Taiwan until the Australian Synchrotron is operational, when it will be transferred to Australia. Thus, a fully commissioned end-station would be available, at minimal cost in addition to the beamline itself, as soon as beamline 6 becomes operational. This end-station has been designed, and is being constructed, to meet the stringent clean vacuum requirements noted above. In particular, the vacuum system will be as carbon-free as possible, and the end-station will allow the investigation of specimens that must be maintained at a low temperature while they are under vacuum, in order to retain moderately volatile material that might otherwise sublime or desorb.

The ASRP end-station has also been designed to accommodate a cell for the XAS of systems that need to be investigated in their wet environment. A conceptual design for this cell has been developed.

The second soft x-ray spectroscopy end-station will be a modified version of an ambient XPS facility at the Advanced Light Source (Berkeley, USA). The design is a result of collaboration by a consortium consisting principally of the University of South Australia, the Victorian and South Australian Governments, the University of Western Ontario, the Canadian Light Source and researchers at the Advanced Light Source.

Beamline 6 – Soft X-ray Spectroscopy	
Source	Undulator (75 mm period), variably polarising
Energy range	0.1–2.5 keV
Resolution $\Delta E/E$	10^{-5}
Special features	Ultra high vacuum