Beamline 9: Microspectroscopy

09

Potential Research Fields

Life sciences

- Biological research and drug design
- Biotechnology and bio-sensors
- Biomedical and medical imaging

Physical sciences

- Sustainable environment
- Forensics
- Advanced materials
  - Ceramics
  - Nanomaterials and composites
  - Metals and alloys
- Mineral exploration and beneficiation
- Earth sciences
- Oil and gas production and distribution
- Chemical reactions and catalysts

Introduction

This beamline will incorporate the most advanced technologies for focussing the x-ray beam, and will be able to achieve a minimum beam size on a sample approaching 0.1 microns for medium to hard x-rays.

It will be a world class instrument of elemental composition and chemical oxidation states using the two-dimensional mapping μ-XRF, μ-XANES and μ-XAFS providing techniques. It will be the only beamline in Phase I to solve scientific problems requiring micron to sub micron resolution. The spatial resolution is not limited to micron and sub-micron spot sizes and the beamline will be a powerful source for samples of up to 1 mm at sub-micron resolution. This beamline will be of particular importance for the emerging area of nanoscience.

Advantages of a Synchrotron Source

Microprobes using electrons, protons, heavy ions and laboratory based x-rays are in wide spread use. Each of these provides unique and complementary information.

Synchrotron microprobes extend the capabilities of these microfocus techniques with synchrotron light having several distinct advantages, including:

- high brightness, leading to very high photon fluxes on the samples, thereby enabling time-dependent processes and reactions to be followed in real time
- ability to focus the light to very small spot sizes while retaining high brightness that enables high spatial resolution and thus 'imaging' of elemental distribution and structure on a sub-micron scale
- tunable beam excitation energy, which means that individual elements in the sample can be selected for analysis and imaging with total discrimination possible against more energetic K or L x-rays. In the case of this beamline the energy range covers the absorption edges of elements from Ti upwards. This is particularly appropriate for the detection of trace elements in samples, and when coupled with the high brightness of the beam it is possible to analyse down to parts per billion level.

User Community

Australian science has been prominent in the use of all microprobe techniques. A pioneering experiment in 1965 used a microfocus proton beam from a van de Graaff accelerator at AINSE to study oxygen concentrations in small spots on the surface of αTi-O alloys[1]. This established the concept of proton microprobe research, which is now a versatile analytical tool with dozens of facilities throughout the world and a dedicated international conference on its applications. The interest in proton microprobe research continued to develop in Australia. Over the last twenty years or so, researchers at Melbourne University have been recognised internationally as leaders in the development and application of proton microprobes[2]. In fact they have marketed their technology internationally, selling focussing quadrupoles and microprobe systems for analysis worldwide. Similar facilities at CSIRO have focussed on solving issues related to mining and exploration industries and has a world class reputation in this area[3].

1 J. R. Bird, T. M. Sabine, Nature 211 (1966) 739.

2 G.J.F. Legge, P.M. O'Brien. R.M. Sealock, G.L. Allan, G. Bench, G. Moloney, D.N. Jamieson and A.P. Mazzolini, Nucl. Instr and Meth. B30 (1988) 252–259

3 C.G. Ryan, D.J. Jamieson, W.L. Griffin, G. Cripps, R. Szymanski, Nucl. Instr. and Meth, B181 (2001) 12–19

Ion microprobe technology has also been extended to heavy ion beams. One of only three dedicated heavy ion microprobes in the world is located on the tandem accelerator ANTARES at ANSTO[4] in Sydney.

This strong interest and expertise in microprobe analysis has been carried over to microfocus spectroscopy on synchrotron facilities.

The ASRP has joined the APS in Chicago where one of the sectors includes a dedicated high-performance microprobe beamline. Thus Australian scientists have been able to combine high performance micro-x-ray techniques on international synchrotrons with nuclear reaction analysis and heavy ion elastic recoil spectroscopy on national facilities to become world leaders in microprobe applications. However, access to the microprobe facilities at the APS is currently limited to about three days per month and thus demand has outstripped supply, and is increasing rapidly due to the growing importance of micron and sub-micron technologies within Australian research priorities.

There are currently 64 known potential Australian users for the micro/nanofocus beamline on the Australian Synchrotron, representing nineteen universities and research organisations (including three from industry and two government agencies). Organisations with the largest number of identified potential users are ANSTO, CSIRO and the Universities of Sydney and Melbourne. Therefore to satisfy this growing demand, a world class facility is required. There has been widespread discussion in this research community and there is consensus that the proposed beamline will satisfy all their research interests.

Research Applications

Sub-micron technology

Beamline 9 will be an important part of the 'toolkit' required for leading edge research in the emerging field of micron and sub-micron technology. It will be particularly powerful for spectrographic analysis of individual particles and microcrystals of sub-micron size, and will be complemented by the small molecule and powder diffraction beamlines (beamlines 2 and 3) to provide comprehensive characterisation of composition and structure.

Biotechnology

In biotechnology synchrotron microprobes can be used for simultaneous elemental and chemical mapping of tissues, cells and other biological samples providing:

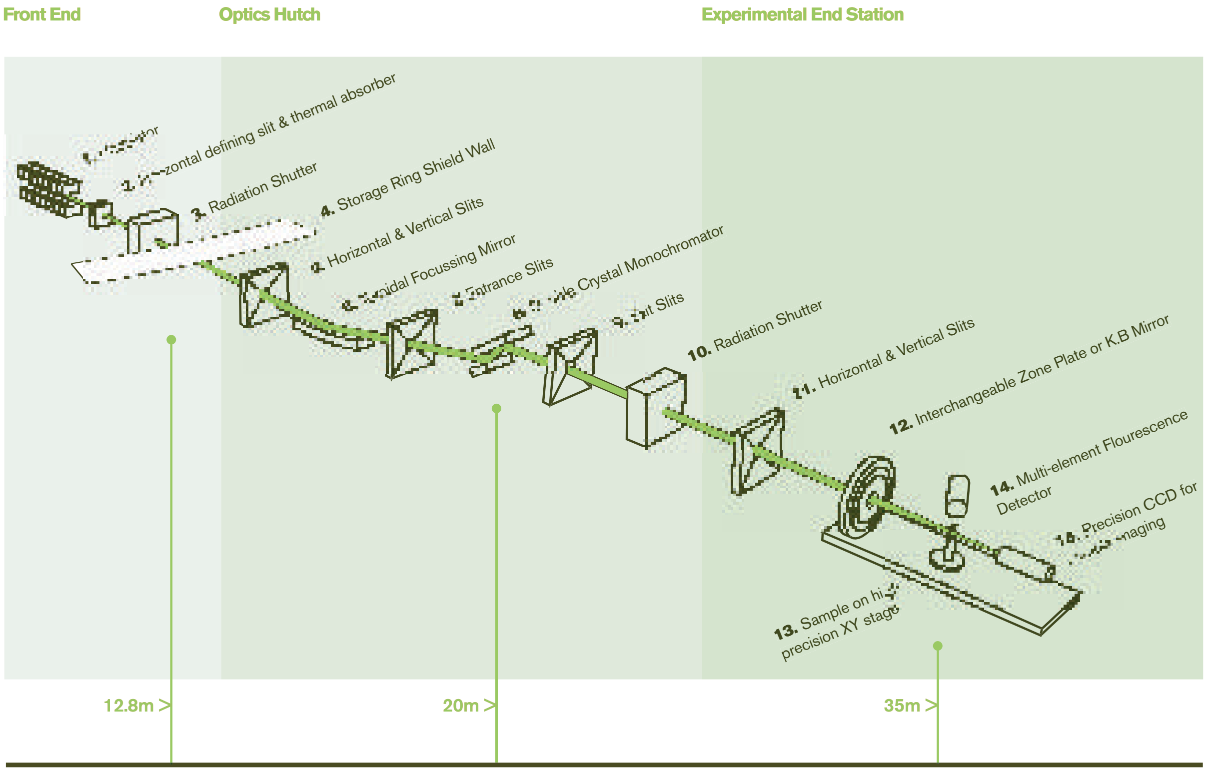
- a better understanding of diseases and immune processes, as well as toxin and heavy metal uptake down to a sub-micron cellular level
- a better understanding of the functionality of a large number of metallo-proteins through the study of the spatial distribution and chemical properties of such species in cells and tissues
- mapping of the distribution and biotransformations of anti-cancer and other drugs in cultured cells and tissues in order to gain a better understanding of drug pharmacology and pharmacokinetics for improved drug design and delivery
- two-dimensional scanning of chromatography gels in which all of the proteins of an organism or an organ are separated. This would enable all of the metallo-enzymes to be mapped, especially those containing Se. XANES and XAFS can be used to determine the local environment about the absorbing atom(s) in order to identify the type of metallo-protein or the sulfur donors in a protein
- a better understanding of the distribution, uptake and metabolism of metal-containing pharmaceutics in cells and tissue for the development of better and safer drugs. Micro-XAS can provide structural chemical information regarding the intracellular biotransformation of the drugs
- gene maps obtained by attaching different metal oxides to DNA and RNA sequences and scanning them by a sub-micron x-ray beam.

Geoscience

Micron resolution is essential in the earth sciences in order to deal with typical sample sizes of relevant structures found in nature. The major analytical techniques such as electron probe, laser ablation ICPMS (inductively coupled plasma mass spectroscopy) coupled and ion probes such as PIXE and SHRIMP also have micron-scale resolution. The ability to produce not only an element map of a sample, but also to determine the oxidation state, as well as how a particular element is bound or coordinated, is the scientific quantum leap offered by the synchrotron microprobe over all other microprobe techniques. It leads in particular to:

- increased efficiency of mineral processing through a better understanding on the micron scale of mineral formation, porosity, chemistry and trace element partitioning in rocks
- more efficient management of mine wastes and mine rehabilitation through a more detailed knowledge of toxicity, chemistry and oxidation states of metals and other mine waste forms
- predictive mineral exploration tools based on a better understanding of ore-forming processes
- determinations of the oxidation state of magmas by XANES to improve our understanding of the geological conditions leading to the release of sulfur as the emission of sulfur during volcanic eruptions can have a profound effect on the Earth's climate
- analysis of tiny inclusions of melt trapped in zircon crystals more than 4 billion years old for chemical indicators of the hydrosphere and atmosphere, with a view to ascertaining when conditions suitable for life first appeared on Earth, and providing a better understanding of weathering processes
- the understanding of the relative abundance, chemical state and distribution of trace elements as indicators of geological processes

4 R. Siegele, David D. Cohen, Nucl. Instr. and Methods B158 (1999) 31



BEAMLINE 9 Microspectroscopy

Figure BL9.1. *Schematic of the microspectroscopy beamline*

- the analysis of extraterrestrial material (dust particles, meteorites, future samples to be returned from Mars) for information on the evolution of the planets and solar system.

Environment

Increasingly the study of toxins in the environment and their pathways requires sub-micron resolution as well as the ability to determine their oxidation state. In particular:

- Increased understanding, on the micron scale, of plants and bacteria that can break down or accumulate toxins or elemental waste, the pathways of the waste and the mechanisms involved can all help optimise processes for waste recovery.
- The transport, origin, chemistry and health hazards associated with micron and sub-micron particulate matter in air and water pollution can be analysed individually by synchrotron radiation induced x-ray emission.

Materials

The high sensitivity, nanoscale resolution and ability to obtain information about chemical binding on a microscale, which will be possible with this beamline, will have many applications in materials research. For example the beamline will be useful for:

- the study of surface corrosion, micro-pitting and wear mechanisms of materials to minimise economic losses and improve the effectiveness of lubricants
- a better understanding of the chemistry and interaction processes of impurities and contaminants in materials and their effects on possible recycling processes
- a better understanding of de-bonding and delamination processes, particularly related to composite materials, involving changes in chemistry and elemental distribution on a micro scale
- a better understanding of polymer crystallisation and seeding of polymers for industry to produce consistent quality and reduce refuse and the study of metal implantation of polymers
- study of grain boundaries, impurities, charge transport and collection efficiency, lifetime, recombination, band gap in photovoltaic materials to improve efficiency in photovoltaic materials
- a better understanding of the structure, porosity and composition of fuel cell electrodes as well as their poisoning, impurity distribution and oxidation to improve the performance and durability of fuel cells.

Beamline Design

The beamline will provide sub-micron spatial resolution (around 0.1 micron) with the highest flux possible and a tuning range of 5.5–25 keV.

The very small beam spot sizes will be achieved by using an insertion device that produces a beam with a high brightness. An undulator (possibly 22 mm period) using the 3rd, 5th and 7th harmonics is the most appropriate choice.

The monochromator will be an adaptation of a large commercial silicon double-crystal unit. The first silicon crystal would probably be cryogenically cooled to operate at the temperature where the expansion of silicon is zero.

The beamline will be designed to accommodate both a zone plate for high resolution microscopy as well as a set of Kirkpatrick-Baez (KB) mirrors. Both will be easily and quickly interchangeable for different applications. Zone plates are intended for high resolution applications (0.1–2 microns), while the KB mirrors are required for chromatic focussing for applications such as EXAFS.

To enable two-dimensional mapping of micro-XRF and XAS data, the end station will have an *XYZ* target mounting stage with sub-micron precision, possibly with a cryo-cooling facility, mounted on a vibration-free optical table. A high resolution CCD camera and light microscope will allow precision placement and monitoring of the samples.

Appropriate x-ray detection systems would be solid state, energy dispersive Ge and Si arrays. An ionisation chamber with high speed capabilities would also be provided.

The multiple detectors will require a multi-parameter high speed data acquisition system as a fast data analysis technique. Data acquisition will include sample XY coordinates and the monochromator settings for μ-XAFS.

Beamline 9 – Microspectroscopy (sub-micron XAS, XANES, XRF)	
Source	In-vacuum undulator (22 mm period)
Energy range	5–20 keV
Resolution $\Delta E/E$	$<10^{-4}$
Minimum beam size at sample (horizontal × vertical)	0.1 × 0.1 micron
Brightness	10^{18} photons/s/mrad^2/mm^2/0.1% band width