Beamline 3: Powder diffraction

Potential Research Fields

Life sciences

- Biological research and drug design
- Plants and crops

Physical sciences

- Sustainable environment
- Forensics
- Advanced materials
  - Ceramics
  - Nanomaterials and composites
  - Metals and alloys
  - Biomaterials
- Engineering
- Mineral exploration and beneficiation
- Earth sciences
- Agricultural technology
- Food technology
- Chemical reactions and catalysts

Introduction

The design and optimisation of new materials and/or mineral processing methodologies is dependent on obtaining precise structural information, especially under non-equilibrium conditions. Such information can be obtained either by single-crystal or powder diffraction techniques. In most laboratories the production of diffraction-quality crystals may be months (or years) behind new discoveries (e.g. high temperature superconductivity). Researchers must then rely on powder diffraction studies. In certain cases, for example in mineral processing or in the study of materials under extreme conditions, powder diffraction can be the optimum method for research.

Advantages of a Synchrotron Source

Laboratory-based powder diffraction techniques are inherently resolution-limited – in the range of observations (d-range), the signal to noise ratio, and the shape and width of observed reflections. Synchrotron-based instruments are the only means whereby these limitation can be overcome to give the resolution required to determine and refine precise and accurate structures of even moderately complex materials from powder samples.

Another advantage of synchrotron light is that it enables the use of anomalous dispersion to be used to obtain information on specific elements, as discussed in chapter 2.

User Community

The past decade has witnessed a staggering growth in the use of synchrotron powder diffraction. The Australian powder diffraction community has made a significant contribution in this area and has an outstanding international reputation.

Powder diffraction plays a key role in the study of nanomaterials, and one of the flagship instruments at the Brookhaven National Laboratory Center for Functional Nanomaterials (USA) is the powder diffractometer at its National Synchrotron Light Source.

The current Australian Synchrotron powder diffraction community exceeds 23 independent research groups from 13 institutions. The total number of Australians using synchrotron powder diffraction methods, including postgraduate research students, is estimated to be greater than 50. The 23 research groups indicate that if beam time and funding limitations were removed they would require 160 days per year for existing programs. This demand is sufficient to use all the time available on a dedicated beamline on the Australian Synchrotron.

The Australian National Beamline Facility at the Photon Factory (Japan) boasts one of the best x-ray diffractometers in the world. Australian scientists are also intimately involved in the development of a diffractometer as part of the ChemMatCARS consortium at the Advanced Photon Source (USA). Australian scientists have also been regular users of similar equipment at many international synchrotron facilities including ESRF (France) and SPring-8 (Japan).

Approximately 25% of all experiments funded by the ASRP utilised the powder diffraction technique. Therefore the installation of a powder diffractometer is a high priority to ensure that this is a world competitive instrument to retain Australia's standing in this fundamental application of x-rays.

Research Applications

The powder diffraction beamline is expected to be used for studies in the following key areas.

Oxide based materials

The majority of advanced materials used in magnetic, conductivity, superconductivity, ferroelectric, catalytic and battery applications are solid metal oxides. This is a very high priority research area in Australia. Metal oxide chemistry is dominated by classes of materials having crystal structures derived from simpler parent structures such as perovskite or rutile. Small lattice distortions, which are critical to the key electronic and physical properties of these oxides, usually lead to lower symmetries and superstructures. These distortions are characterised by subtle peak splittings and the appearance of weak superlattice reflections in diffraction data. Typical examples include polar distortions in bismith oxide ferroelectrics, Jahn-Teller distortions in manganese oxide battery materials and valence ordering in colossal magneto-resistance materials (CMR). The detection and understanding of such distortions requires the high resolution afforded by synchrotron radiation. For more on this, refer to chapter 3.

An underlying feature of many of the most interesting materials is the strongly correlated behaviour of the electrons and coupling of the electronic charge and spin degrees of freedom with those of the electron orbitals and the lattice. The greatest potential for functionality is in materials at the edge of a structure and/or electronic instability where small changes in chemical or physical conditions lead to a major change in properties. Success here requires rapid data collection, which is only possible on a high brightness synchrotron radiation source.

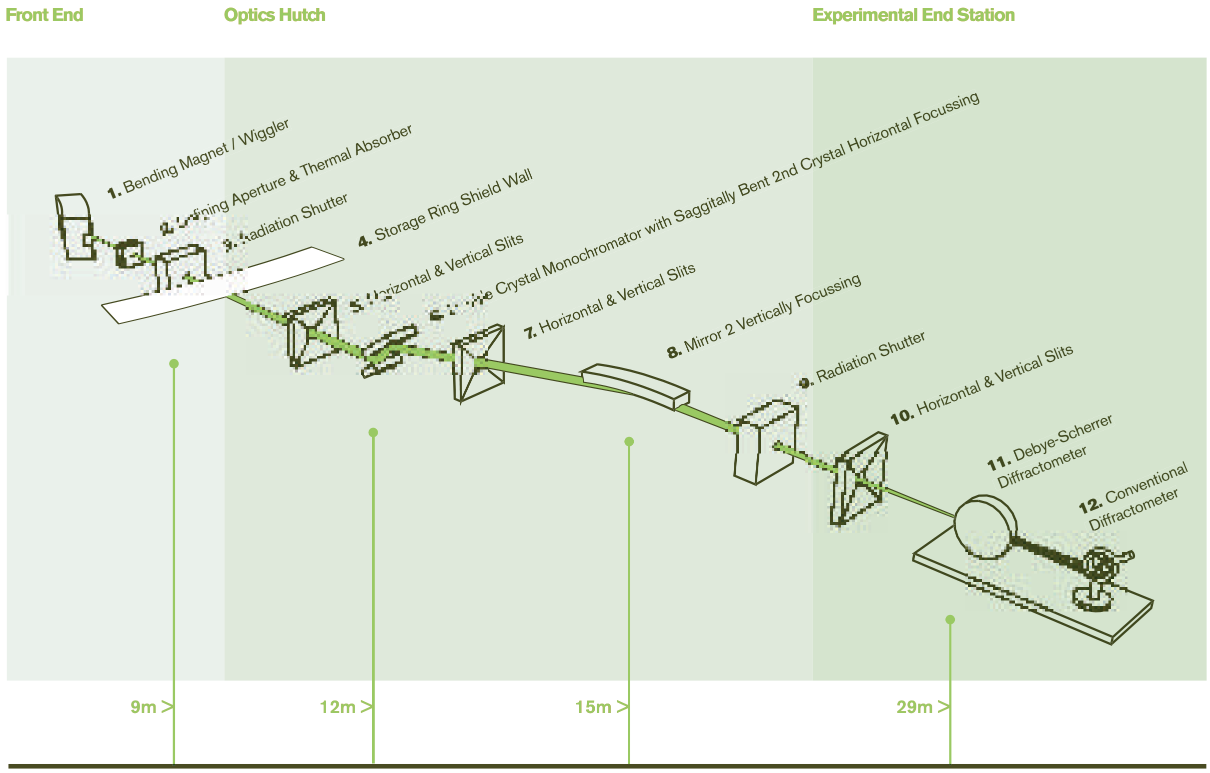
Microporous and framework materials

Detailed knowledge of the crystal structure of microporous materials such as zeolites is required in order to understand their properties and improve their use of catalysts, sorbants and micro-reactors. These materials typically have large unit cells and it is common to observe complex patterns because of low symmetry or subtle distortions. In many cases a very high x-ray flux as delivered by a synchrotron source is the only way these problems can be studied.

Another application is in the study of a novel class of cyanide-bridge coordination framework solids that display negative thermal expansion behaviour. These have diverse potential applications in high precision and low thermal shock materials where the positive thermal expansion exhibited by the vast majority of materials is a hindrance. The ability to access low d-spacing – possible with synchrotron radiation – is critical in the study of the amplitude of such processes.

Mineral processing and soil sciences

Powder diffraction has been applied widely for analysis in the mineral processing industries. Laboratory techniques have traditionally been used; however, as ore grades



BEAMLINE 3 Powder Diffraction

Figure BL3.1. *Schematic of the powder diffraction beamline* become lower with increasing complexity in the mineralogy, improved peak resolution and peak-to-background ratios are required to conduct full characterisation. The synchrotron-based powder diffraction technique provides the inherent high resolution, high sensitivity and high speed capability that is critical in such studies.

This high speed capability will be used in studies where the sample environment emulates the processing conditions found in industry. The availability of a range of sample environments, including high temperatures and pressures, will be a key feature of this instrument. Some examples are provided in chapter 3.

Strain, texture and phase mapping

Powder diffraction has important applications in mechanical engineering, particularly in the mapping of residual strain fields, the detection of phases that degrade the material properties, mapping texture of the material, and determining grain size and the degree of cold work. High-energy synchrotron radiation (60 keV, which will be available from the wiggler source) enables measurement in the depth range of 0.01–1 mm. This depth range is very important as it is where most of the degradation of mechanical components originates. It also covers the thickness range of many protective coatings (e.g. thermal barrier coatings) and surface engineering treatments (e.g. laser shot peening).

The large flux of high energy x-rays available from insertion devices enables the two-dimensional mapping of strain, texture and phase in practical times. At each point of a map, a large area two-dimensional position-sensitive detector collects the rings of the diffraction pattern. Increasing the sample to detector distance provides information on cold work and grain size. Such maps are important in most areas of mechanical engineering (e.g. aerospace and power generation) and enable the integrity of newly developed procedures (e.g. welds) or aged components (e.g. turbine blades) to be assessed.

Furthermore, techniques are now being developed, with spiral or conical post sample collimators, to produce full three-dimensional maps of strain. Coupling the two- or three-dimensional mapping data with imaging data from beamline 10 (imaging and medical therapy) will increase the power of both the mapping and imaging techniques. Thus flaws can be located using beamline 10 and the associated strain fields mapped using beamline 3. Similar synergies may also be possible with other beamlines.

Pharmaceuticals

Powder diffraction has a key role to play in structural studies of pharmaceuticals and their interaction with low molecular weight peptides. Already powder diffraction patterns play a key role in unequivocally establishing the crystalline form of a pharmaceutical in a manufactured drug. Where the data has sufficient resolution, these methods will aid in understanding the solubility and dissolution of these forms.

To ensure that powder diffraction is available on the synchrotron at first light, this beamline will be sourced initially from a bending magnet. Specific techniques can be refined during this start-up period. However, as soon as the electron beam is sufficiently stable, the beamline will be moved to a wiggler source. This will enable an increase in flux and extension of photon energy to 65 keV, so that the capability of this beamline rivals the best in the world.

Beamline Design

Optics

The preferred optics for the beamline are conventional, and consist of a mirror and monochromator. All the optical components should be incorporated into a separate hutch. The first mirror provides vertical collimation and removes high-order harmonics. A double crystal monochromator consisting of two Si (111) crystals is required. At this stage provision has been made for a second mirror in the optics hutch to allow for a future upgrade of the beamline to permit the beam to be focussed (see figure BL3.1).

End stations

Two powder diffractometers on a single beamline are proposed, the first being a relatively compact Debye-Scherrer camera having a large area detector. The second diffractometer would include a large two-dimensional position-sensitive detector for strain and texture measurements capable of being mounted to collect transmitted or reflected diffraction data, as well as being equipped with an array of analyser crystals coupled with scintillation detectors for very high resolution studies.

A positioning table will enable strain, texture and phase mapping and will be able to accommodate moderately large (20 cm) and heavy (5 kg) mechanical components or samples on standard mounting plates. The standard mounting plates will be interchangeable with those on beamline 10 and will facilitate the accurate co-alignment of the sample or component to better than 0.01 mm.

Both diffractometers will be configured to enable an array of environmental conditions to be studied, including high pressure, high and low temperature, and variable atmosphere.

A compact mechanical test machine will also be available to calibrate the strain data in situ to obtain the stresses, and also to load or fatigue the sample or component in situ.

Beamline 3 – Powder X-ray Diffraction	
Source	Bending magnet, then wiggler
Energy range	4–65 keV
Resolution $\Delta E/E$	$<1 \times 10^{-4}$
Beam size at sample (horizontal × vertical)	2 × 5 mm