# Australian Synchrotron Development Plan
# Project Submission Form

## Section A: Summary and Proponent Details

**Project Title**

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| **The Case for the Advanced Diffraction and Scattering Beamline at the Australian Synchrotron** |

**Spokesperson**



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**Executive Summary (approx. 100 words)**


An Advanced Diffraction and Scattering (ADS) Beamline is proposed for construction at the Australian Synchrotron. The beamline will be optimised for a range of leading-edge diffraction and scattering techniques and will enable high-speed data acquisition and sample mapping. The beamline aims to cater to applied, fundamental, and industrial researchers from diverse backgrounds, including materials science and engineering, earth sciences, chemistry, and biology.

The beamline design will allow delivery of monochromatic and polychromatic X-rays over an energy range of 30-120 keV, with a minimum spot size in the order of 10's of microns. Existing and prospective users will benefit from the availability of high energy X-rays which enable bulk materials analysis, wide *q*-space sampling, simultaneous collection of multiple scattering vectors, and reduced radiation damage.


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## Section B: Detailed Description

Attach a document using the following headings (max 10 pages):

# B1: Description of Proposed Beamline/Development Project

## Scientific Context and Objectives

The Australian chemical, materials and engineering research communities have developed a strong diffraction and characterisation background extending across a broad range of research areas and have made significant impacts on the world stage. In addition, the synchrotron user community has been built up over many years through access to the Australian National Beamline Facility at the Photon Factory in Japan. Consequently, one of the initial beamlines to be constructed at the Australian Synchrotron was the bending magnet powder diffraction beamline, where the community has continued to mature and flourish. However, there is a large portion of the diffraction community, particularly in materials development and characterisation, that cannot be serviced by the capabilities of existing beamline infrastructure and, as such, these groups continue to seek access to international facilities. This scenario is doubly true for materials engineering researchers who, at present, only have local access to neutron facilities. The capabilities of the proposed **Advanced Diffraction and Scattering** (ADS) beamline compare well with that on offer at world leading international facilities such as the ESRF (ID15), the APS (1-ID and 11-ID), and more recently, DLS (JEEP) and Petra (P02 and P07), and in the future, NSLS-II (XPD).

In addition to applied and fundamental research opportunities, it is also expected that the ADS beamline will attract legitimate commercial interest. A high energy X-ray (HEX) beamline, such as that proposed herein, provides a unique opportunity to examine engineering components for various industries, including automotive and aeronautical. On ID15 at the ESRF, the value of beamtime sold for proprietary research in these areas was *ca.* €75k per year for over the past 5-10 years. In 2005, the beamline's gross income for these beamtime applications exceeded €200k; only limited by the ESRF due to the priority placed on science, versus industrial use.

There are several advantages to using HEX, in the context of engineering and materials research and also, perhaps surprisingly, to biological samples. The characteristics of HEX enable the following:

- Bulk materials analysis, through use of micron sized beam(s); bulk sample information can be obtained, but also allows mapping etc.;
- Wide *q*-space sampling; the short wavelengths are ideal for sampling large a *q*-space range;
- Use of complex sample environments; high energies enable penetration of large and elaborate sample cells;
- Simultaneous collection of multiple scattering vectors; without HEX this is not achievable unless using a spallation neutron source; and
- Reduced radiation damage; due to reduced absorption and faster data acquisition times.

The proposed beamline will provide opportunities for Australasian scientists to explore problems in a broad range of areas, which largely fall into two main categories relating to beamline functionality:

**1.** Monochromatic diffraction, using large area detectors, such as:
   - **a.** Ultra-fast 'traditional' powder diffraction;
   - **b.** Total scattering analysis or Pair Distribution Function (PDF) analysis of crystalline and amorphous materials;
   - **c.** Single crystal studies at high energy for unique structural and charge density analyses; and
   - **d.** Rapid texture analysis and 2D materials mapping.

**2.** Polychromatic studies, including:
   - **a.** Energy dispersive diffraction studies from an isolated 3D gauge volume; and
   - **b.** Laue diffraction.

A high-energy X-ray probe is a unique and powerful instrument for a vast number of scientific and industrial studies, and is currently accessible only at international facilities. Even with this being the case, there is already a large and active user community utilising HEX scattering techniques, as shown by the number of proponents above. Given the advantages of such scattering techniques, it is certain that the number of users would dramatically increase given local access to such a beamline, enabling the community to be competitive in a broad range of research areas at an international level. The techniques outlined above are well developed and the Australian Synchrotron and its user communities have close collaborative contacts with international institutions, allowing all techniques to be implemented quickly at the Australian Synchrotron with little development time.

The potential commercial prospects, mainly through non-destructive residual stress analysis, are large. Australasian industry will no doubt benefit from such techniques. In addition, limited access to such facilities means that potential commercial clients world wide outweigh the available beam time.

## Overview of Design Elements

**Source and Optics Discussion**

The ADS beamline will require installation at an insertion device straight section, most likely utilising a superconducting wiggler in order to achieve the upper energy of the design ideal. The beamline will be designed for use over the energy range of 30-120 keV and will be capable of allowing both monochromatic and polychromatic modes of operation.

Also crucial to the performance of the beamline is the optimisation of flux density. A number of experiments that will be conducted on the beamline are reliant on high flux; this includes *in situ* PDF measurements. Therefore, the optical focussing components will ideally be as far up stream as practicable in order to avoid loss of flux density through horizontal (and vertical) divergence of the beam.

One of the challenging aspects of a beamline on such a source is the high heat load that is incident on the optics, the sample and ultimately the detectors. This issue has already been addressed by beamlines at the Australian Synchrotron such as the XAS beamline and more recently, and relevantly, the IM beamline. By removing or limiting the throughput of lower energy radiation (i.e. <30 keV) through the use of filters at the front end of the optics it will be possible to minimise the incident heat load downstream of the source.

As dedicated micro-focus beamlines are proposed elsewhere, it is not anticipated that the beamline will attempt to make any records in terms of the smallest spot size achievable. Again, this design aspect relates to flux density and also the practical realities of the experiments being undertaken on the beamline. It is therefore expected that a spot size on the order of 20 (V) × 100 (H) µm is readily achievable.

**Detectors**

Detector selection is critical to the performance of any synchrotron beamline and it is often detector performance that lags behind the technology of the other beamline components. On this beamline, detector selection will be a challenge due to the high energies involved, but with the on-going detector development around the world, such as the PILATUS area detector and the MAR amorphous Se detector, it is expected that there will be a range of options when this beamline comes on-line, in addition to those already in use, such as the Thales and GE flat panel area detectors.

Although the detector resolution requirements will be modest (e.g. 100 µm pixel size), the detectors must allow rapid, real-time data acquisition for time resolved and *in situ* studies and have a high dynamic range to complement the available X-ray flux[1].

A highly collimated, high resolution energy-resolving detector system is also required for energy-dispersive diffraction studies. For Laue diffraction, an area detector would be needed with good efficiency over the utilised energy range. Energy-sensitive area detectors are currently under development in several groups around the world, and may be a viable option when this beamline is built.

**Experimental Equipment**

A variety of sample stages and sample environment ancillaries for *in situ* experimentation must be made available in-house for use at the beamline. In addition, the end station must also incorporate the flexibility to accommodate a wide variety of user-supplied sample apparatuses.

## B2: Applications and Potential Outcomes to Australian Scientific Community

The ADS beamline will provide a range of opportunities for fundamental, applied and commercial research to advance knowledge in fields such as materials development and characterisation, engineering, biology, energy production and storage, earth sciences, manufacturing and minerals processing.

The beamline will open fields of research previously untapped or underutilised in Australia and will maintain Australia's place at the forefront of diffraction-based research and pioneering. Some of the research areas that will be enhanced include: liquid total scattering analysis which provides complementary species coordination information to that obtained by EXAFS; high energy *in situ* macromolecular crystallography; complex *in situ* materials processing studies; charge density studies; and energy dispersive diffraction experimental technique development.

The ADS beamline seeks to address the shortfall in local capability that affects a range of research areas. These are reiterated below, along with case studies from some of the existing research groups.

## Monochromatic diffraction, using large area detectors

**a. 'Traditional' powder diffraction (PD), including: structural characterisation, *in situ* studies, phase identification/quantification, etc.**

***PD Case Study: In situ diffraction studies of mixed ionic conductors - Ling, University of Sydney***

Materials that exhibit significant mobility of different types of charge carriers (*e.g.* oxide ions, protons, and electrons) have diverse potential applications as fuel cells, membranes, electrodes, batteries, and sensors. Mixed conductivity is often associated with a high uptake of water at moderate temperatures (below *ca.* 600 °C), leading to complex sequences of phase transitions as functions of temperature, pressure and humidity. Characterising these sequences is crucial to understanding their functionality[2].

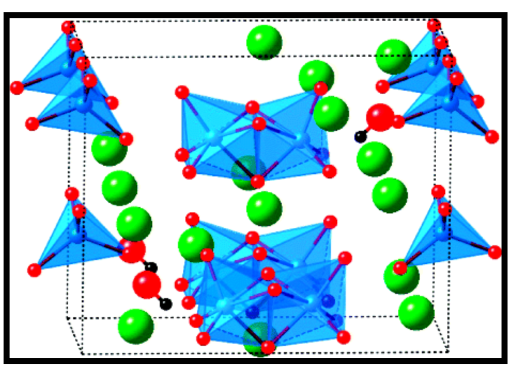

***Figure 1:*** *Crystal structure of* $\gamma$*-III-*$Ba_4Nb_2O_9.1/3H_2O$

In order to extend this research, it is necessary to collect data under conditions of high water vapour /oxygen/hydrogen pressures, to provide information about the relative stability of the various phases under fuel cell operating conditions. *In situ* experiments require the use of sample cells with thicker walls than can be penetrated by X-rays on the PD beamline, but are well suited to the ADS beamline.

Furthermore, we have so far only solved the average structure of the γ-III phase - see Figure 1 - our single crystal diffraction data show that the true structure is a 4x2x4 superstructure of this average, while the γ-II and γ-I phases have different superstructures. The solution of these superstructures may well be intractable problems, given the likelihood that they coexist with some local disorder. The best way to characterise the conducting layers, and thereby understand the conduction mechanisms, would be through Pair Distribution Function (PDF) analysis. The ADS beamline will uniquely permit several experiments that advance understanding.

***PD Case Study: Ultra-Fast Kinetics Using Time-Resolved Diffraction - Riley, University of Melbourne***

Intense high-energy X-rays provide sufficient transmission of samples and diffraction intensities to directly measure chemical kinetics of reactions. In many industrially relevant processes, the surface to volume ratio of the sample will influence the rate constants of reacting compounds, which, if sufficiently high, will completely inhibit the reaction. Using time-resolved diffraction, optimisation of novel material synthesis, simulated service environments and dynamics of phase transitions have all been verified in a non-destructive, unobtrusive manner. These applications have, respectively, achieved significant reductions in the developmental costs of novel discovery, allowed for improvement of industrial process efficiencies and provided direct observation of previously unknown phenomena - see Figure 2.

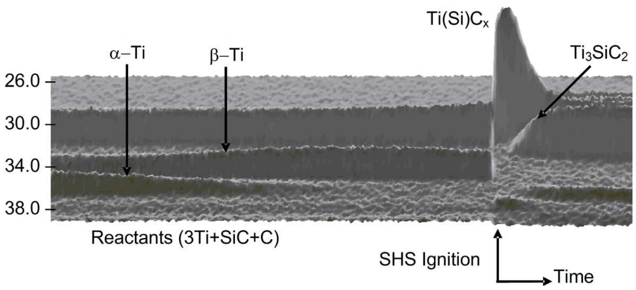


***Figure 2:*** *Fundamental phase transitions (α-Ti → β-Ti) during an ultra-fast reaction (time resolution <0.1s), resulting in the optimisation of a novel compound, $Ti_3SiC_2$[3].*

To date in Australia, only neutron diffraction provides sufficient penetration of both sample and ancillary equipment to allow routine measurement of reaction kinetics. However, several distinct advantages of X-Rays arise from the ability to achieve greater spatial resolution (i.e. ability to use focusing optics with greater precision) and chemical selectivity using absorption edge contrast. In combination, these attributes of X-Rays allow for volumetric mapping of reaction kinetics with possible correlation to localised chemical inhomogeneities, which, if absent, can result in inaccurate interpretation of data. Furthermore, the high diffraction peak resolution and significantly high fluxes of X-Rays provide unrivalled solution to crystallographic cell parameters and the shortest time resolutions for kinetic analysis.

***PD Case Study: In situ studies of gas-solid reaction mechanisms under high gas pressure - Gray, Griffith University***

Powder diffraction is a powerful tool for elucidating the mechanism and pathway of a reaction via the crystallography of the reactants and products. This group has used neutrons and, to a lesser extent, X-rays and muons, to study a number of hydrogen storage materials under deuterium pressures up to 2600 bar. Access to the ADS beamline will provide an opportunity to set up a world-class facility for studying such materials under medium-high gas pressure. We are building an advanced X-ray sample holder based around a single-crystal sapphire capillary embedded in a high-pressure gas-handling system. The concept has been realised at pressures up to about 150 bar by Chupas *et al*.[4] and Ravnsbæk *et al*.[5] and used with great effect to study the thermal desorption spectra of hydrogen storage materials such as borohydrides.

The experiments to be undertaken require *ca.* 700 bar hydrogen gas pressure at 600°C. Due to the large thick-walled sample environment, the X-ray energy available on the current PD beamline is too low for such experiments, but the energy range of the ADS beamline is ideal.

***PD Case Study: High pressure engineering properties of nanoparticles - Gu, Australian Synchrotron***

Access to high pressures opens new horizons for tuning material structures and properties. Studies of condensed matter under static high pressure conditions extending into the 100 GPa range, under simultaneous very high temperature or low temperature conditions[6,7] can be used for fields ranging from minerals, earth and planetary science, to solid state physics and chemistry, and material science[8,9]. Various types of sample cell are now available for these studies such as diamond anvil cells (DACs)[10] and Paris-Edinburgh cells[11].

High pressure X-ray diffraction (HP-XRD) was used to investigate the engineering properties of Ag and Au nanoparticles materials under extreme conditions[12]. This work used the small spot size and high intensities available at the Materials Science beamline at the Swiss Light Source. The compressibility of silver and gold nanoparticles suspended in a methanol-ethanol mixture was studied by diffraction at pressures up to 30 GPa. Unexpectedly for that size, the nanoparticles show a significantly higher stiffness than the corresponding bulk materials. The bulk modulus of n-Au shows an increase of *ca.* 60% and is in the order of W or Ir. The unexpected high stiffness may open new fields of applications to these noble metal nanoparticles and it could be expected that other metal nanoparticles show a similar trend. To carry this work out in Australia requires small spot sizes and high intensities that access to the ADS beamline will bring.

**b. PDF: solid- and liquid-state total scattering analysis**

Pair distribution function analysis uses an entire diffraction pattern, not just peak positions and intensities, to recover structure information in the form of a radial distribution function of inter-atomic distances from both crystalline and amorphous materials. The PDF is a radial distribution function related to the probability of finding two atoms at a distance *r*, including those which may deviate from the average long-range structure, providing insight into the instantaneous structure. PDF analysis has been used in the study of nanoporous host-guest systems[13], amorphous aluminosilicates[14,15] and nanocrystalline titania[16].

The incident beam energy must be as high as possible to maximise the accessible *q*-space. In addition, the high intensities at the ADS beamline will also enable *in situ* experiments.

***PDF Case Study: Negative thermal expansion materials - Kepert, University of Sydney***

Negative thermal expansion (NTE) is an exotic material property with attractive potential applications, most notably in moderating the predominantly positive thermal expansion of materials, particularly those in high precision applications where instability associated with temperature fluctuation often reduces performance. This behaviour has been identified in a range of oxide-based materials, as well as molecular framework materials containing linear diatomic bridges such as the cyanide anion.

We have carried out an *in situ*, PDF study of $Zn(CN)_2$ which showed an increase in average transverse displacement with increasing temperature (Figure 3) giving direct confirmation of this mechanism for NTE[17] - structural information extracted directly from the PDFs. The rich structural and compositional diversity of cyanide-bridged molecular framework materials is expected to yield a vastly expanded range of NTE materials with enhanced properties and, as such, the study of the mechanism underlying the NTE in the cyanide-bridged $Zn(CN)_2$ structure is of particular relevance.

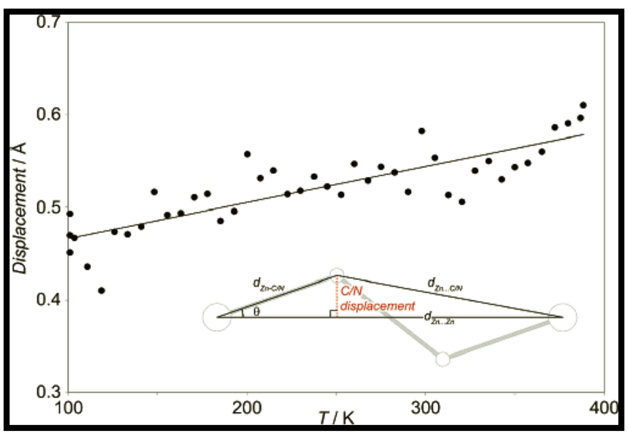


***Figure 3:*** *Average displacement of the C/N bridging atoms from the line connecting the $Zn^{II}$ centres*

The ADS beamline would give access to high intensity X-rays with energies of *ca*. 90 keV required to carry out this experiment.

***PDF Case Study: Local ordering in Jahn-Teller materials - Kennedy, University of Sydney***

The Jahn-Teller (JT) distortion in manganites is associated with the unique colossal magnetoresistance of these oxides. Typically, the JT distortion involves lengthening of two Mn-O bonds in the $MnO_6$ octahedra, with a concomitant shortening of the other four. This is accompanied by orbital ordering of the occupied Mn 3d orbitals, which introduces new periodicities into the system, possibly accompanied by lattice distortions. Understanding the complex interplay between this orbital ordering and the charge and magnetic ordering that generally accompanies this, whilst exploring their coupling to the lattice, has emerged as a major challenge in condensed matter science in recent times.

The formation of fluctuating OO domains just below the orbital melting temperature has been postulated and direct experimental observation of these has required the use of a local probe such as PDF analysis. PDF has the enormous advantage over other techniques such as EXAFS in that it can readily be carried out at high temperatures and/or pressures and thus invaluable in the study of complex materials.

### c. Single crystal (SX) studies

***Diffuse scattering***

Diffuse scattering is the weak background scattering that occurs in the diffraction patterns (X-ray, neutron or electron) of all real crystalline materials from the simplest, e.g. NaCl, to the most complex macromolecule, e.g. protein.

In conventional crystallography, the position and intensity of the sharp Bragg diffraction peaks are used to deduce the average repetitive arrangements of atoms in crystals on the unit cell level. Diffuse scattering, the intensity between the Bragg peaks, contains information about the local deviations from the average - giving structural details on the 1-1000 Å scale. It is often just these deviations from the average, rather than the average structure itself, that give materials their unique or novel properties.

***SX Case Study: Relaxor ferroelectrics and other functional oxide ceramics - Welberry, ANU***

A renewed interest in the field of ferroelectricity has taken place in recent years since the finding of exceptional piezoelectric properties in the lead-oxide class of relaxor ferroelectric (RF) materials typified by the disordered perovskites $PbMg_{1/3}Nb_{2/3}O_3$ (PMN) and $PbZn_{1/3}Nb_{2/3}O_3$ (PZN). RFs are materials having an extremely high dielectric constant that has significant dispersion over a broad range of frequency and exists over a wide range of temperature. When doped with $PbTiO_3$ (a conventional ferroelectric material), PMN and PZN can exhibit high strain levels, making them promising candidates for the next generation of solid-state transducers and actuators. Although PMN, PZN and numerous related materials have been extensively studied over a long period, a detailed understanding of the exact nature of their polar nanostructure has still not emerged.

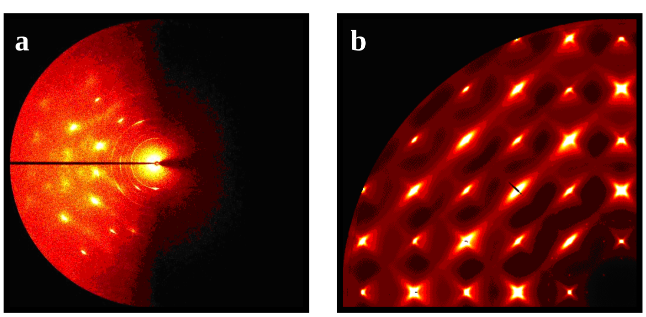


***Figure 4:*** *(a) A single frame of data for PZN-PT using 25 keV X-rays. (b) Part of a full 3-dimensional data set of PZN recorded using 58 keV X-rays. The relative intensity scales are 1:150*

All of the important materials of this kind are based on Lead, or more recently, on Bismuth. Both Pb and Bi are strong absorbers of low-energy (< 25 keV) X-rays, so in order to obtain good diffuse scattering data, much higher-energy X-rays must be used. Figure 4(a) shows data collected at 25 keV at the AS PD beamline; most of the field of view is blocked by the shadow of the crystal, the signal level is very low and there is strong powder scattering from surface damage on the outside of the cut crystal. In contrast, Figure 4(b) is of a comparable sized sample of PZN recorded at the APS using 58 keV X-rays. Here, the diffuse scattering signal is recorded with excellent signal to noise and shows much fine-detail that is crucial to the analysis.

***MX Case Study: Anomalous diffraction at ultra-high energy for protein crystallography - Jakoncic et al.***

Recently, an alternative solution to the phasing problem has emerged with the use of high energy X-rays for the acquisition of diffraction data from protein samples.

*"With the advent of third-generation synchrotrons, cryogenic protection of crystals was shown to be insufficient to prevent crystal deterioration due to radiation damage, the major cause of unsuccessful phasing at third-generation synchrotrons. The total linear mass absorption coefficient calculated for a generic protein sample is about 5 $cm^2g^{-1}$, compared with 0.2 $cm^2g^{-1}$ at higher energies, leading to potential lower cumulated absorbed dose; i.e., less radiation damage at high X-ray energies. With an optimised source, an implemented macromolecular crystallography experimental setup and high-energy optimised detectors combined together, it will be possible to collect almost ideal high-quality data without radiation damage, nearly free of absorption errors."*[18]

## d. Materials mapping and texture analysis

***Case Study: Study of residual strain and texture in thermal barrier coatings - Thornton, DSTO***

Thermal barrier coatings (TBCs) typically consist of a 0.3 mm layer of partially stabilised zirconia over a bond coat of 0.2 mm of NiCoCrAlY applied to a metal substrate. TBCs are applied to components that are exposed to hot combustion gases within gas turbine and piston engines in order to thermally insulate the components. While TBCs are now widely used, their lifetime cannot be predicted reliably. The ability to predict TBC lifetimes will enable a greater utilisation of the coating's temperature reduction properties and requires a full understanding of the failure mechanisms of TBCs.

Through the use of high energy (*ca*. 80 keV) X-rays in transmission geometry, it has been possible to map the phase composition, texture and structure, and obtain information on the magnitude and distribution of the strains[19]. The observations of out-of plane tensile strains at and above the interface supports the proposed mechanisms for TBC failure based on localised swelling of the material underlying the zirconia.

To continue this work requires access to high energy X-rays, as would be available at the ADS beamline, in order to penetrate the thick samples. The use of area detectors, enabling the imaging of complete diffraction rings, will allow the distribution of intensity about the rings to be analysed, providing texture information. Reflection-geometry methods using lower energy X-rays are not feasible due to the limited volume sampled by that mode.

***Case Study: Phase transitions in metal alloys - Liss, ANSTO***

Low density, high specific yield strength, high oxidation resistance, and good creep properties at elevated temperatures make intermetallic γ-TiAl-based alloys excellent candidates as structural materials for advanced jet and automotive engines as well as for future hypersonic vehicles. The mechanical properties depend strongly on composition, thermo-mechanical processing, and subsequent heat treatment. The processes occurring during heat treatment are complex and difficult to study directly. Therefore, characterisation and measurements are necessary with different techniques such as macroscopic stress and strain measurements, hardness testing, optical and electron microscopy, calorimetry, as well as diffraction methods with neutrons and X-rays, to mention but a few.

High-energy X-rays (> 100 keV) can penetrate centimetres into light and medium-dense materials as investigated here. A high-energy synchrotron X-ray diffraction study was undertaken at ID15B at the ESRF to characterise *in situ* phase transitions, recrystallisation behaviour, and phase evolution in an intermetallic Ti–46Al–9Nb alloy up to 1400 °C[20]. The combination of the penetration power into a bulk sample and the large high intensity allowed for sophisticated *in situ* investigations

## Polychromatic studies

### a. Energy dispersive diffraction

In energy dispersive diffraction, a well-defined polychromatic incident X-ray beam is used in conjunction with a highly collimated diffracted beam and an energy-dispersive detector. The intersection of the incident beam with the field of view of the collimator defines an elongated gauge volume or lozenge in the sample from which a full scattering pattern is collected. The distribution of intensity vs energy in the incident beam and the angle between the incident and diffracted beams determines the range of observable *d*-spacings. To map a region of interest the sample is raster scanned through the beam so that the entire region of interest is examined. The experimental arrangement is illustrated in Figure 5[21].

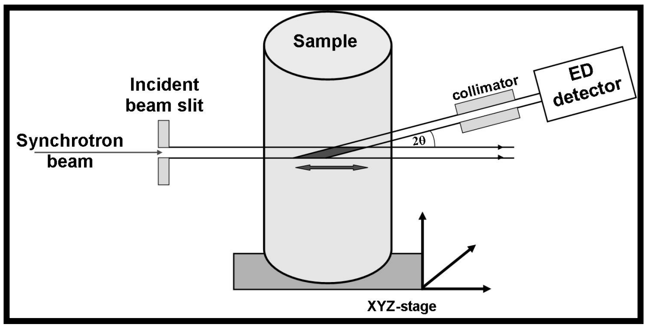


***Figure 5:*** *The experimental arrangement for energy dispersive diffraction.*

***Case Study: Energy dispersive diffraction studies of inert anodes - Rowles, CSIRO***

Traditionally, most characterisation of starting materials and products for electrochemical investigations in molten salts relies upon *ex-situ* techniques. Information obtained in this manner is often subject to experimental artefacts brought about by changes that may take place during preparation of samples for analysis. Whilst *in situ* techniques are common in aqueous electrochemistry, equivalent methods for molten-salt electrochemistry are particularly challenging, although McGregor *et al*.[22] have implemented a "see-through" cell for observing the cell contents during electrolysis.

Previous work[21] developed a method whereby the internal features of an operating electrochemical cell could be phase mapped quantitatively without destroying the cell. We carried out energy dispersive diffraction (Figure 5) studies of pre-prepared electrochemical cells that had been operated for 10 min, 1, 2, 4 and 7 hours. Line scans - see Figure 6 - were taken across the anode at various positions along the anode, and an analysis method was developed that allowed the full quantification of the data via the Rietveld method.

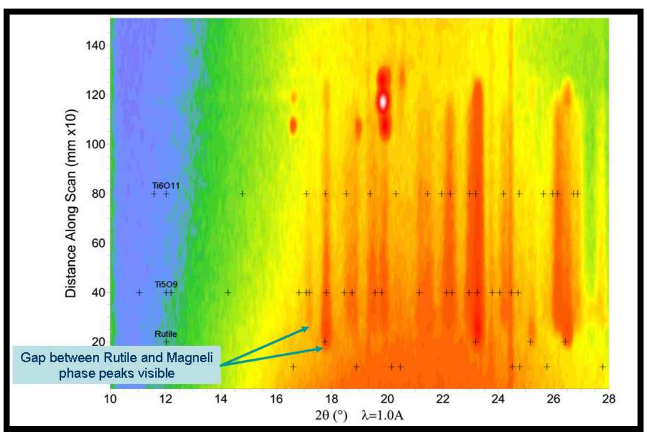


***Figure 6:*** *Accumulated patterns collected for a cell cycled for 10 minutes. The thickness of the rutile layer can be seen directly. The energy scale has been converted to 2$\theta$ using a dummy wavelength to allow peak identification.*

This research must be conducted at overseas synchrotron facilities due to the lack of suitable local facilities. The materials and physical dimensions of the cells used in this experiment demand access to high energy X-rays in order to penetrate the sample cell and electrolyte, typically $CaCl_2$. Additionally, energy dispersive diffraction simplifies the sample cell design as it demands only a single, small beam exit window. Access to a high-energy beamline capable of delivering polychromatic X-rays, with an energy dispersive detector would enable this experiment to be carried out in Australia and greatly simplify travel arrangements - removing the requirement to transport entire sample environments overseas.

*Case Study: Geopolymerisation kinetics determination by energy dispersive X-ray diffraction - Provis, University of Melbourne*

Geopolymers are a class of cementitious aluminosilicate binder materials with a variety of applications including as a high-performance, low-$CO_2$ alternative to ordinary Portland cements. The need to understand and control geopolymeric setting behaviour has led to the application of a variety of techniques in the analysis of the early stages of geopolymerisation. In particular, *in situ* energy-dispersive diffraction allows the investigation of bulk materials inside of reaction chambers.

Previous experiments[23] have provided a remarkably accurate quantitative measure of the extent of reaction as it progresses towards completion and yielded valuable information about geopolymers as they solidify. Access to the ADS beamline would allow such experiments to be undertaken in Australia enabling the use of 'laboratory-sized' sample cells in transmission geometry, greatly simplifying experimental design.

**b. Laue diffraction**

Laue diffraction represents the only 'single shot' diffraction technique. In this instance the polychromatic nature of the synchrotron X-ray source is fully utilised in order to capture the diffraction pattern in a single exposure, removing the need to rotate the sample to meet the Bragg diffraction condition. Consequently, the methodology is ideally suited to probe real world samples, in real environments and in real time[24]. Moreover, application of high energy polychromatic diffraction represents a particularly interesting case. An increase in the energy band-pass to the high energy X-ray regime will provide true 3D data, up to several millimetres in most bulk materials.

*Case Study: Electronically driven phase transitions in Ni-Al alloys - Riley, University of Melbourne*

A high-energy (*ca.* 100keV) beamline offers the potential to study phase transitions in bulk crystals of current interest to materials science. Of particular interest are crystals for which the phase transition is believed to be driven by the electronic contribution to the crystal free energy. A typical example would be $Ni_{1-x}Al_x$ ($0.615 \le x \le 0.64$), which undergoes a martensitic transformation from a CsCl-structure parent phase to a pseudo-orthorhombic phase on cooling[25] below a transformation temperature, $T_m$, which is strongly composition dependent[26]. Previous studies using inelastic neutron scattering[27] have revealed phonon softening as a precursor to this phase transformation in the parent phase but the strongly composition-dependent behaviour is suggestive of an electronically driven phase transition.

While temperature dependent neutron diffraction from a single crystal (using the Wombat beamline at the OPAL reactor) gives a clear indication of the complexity of the phase transformation and will provide an understanding of crystal structure for such materials, high-energy X-ray Laue diffraction has the potential to image the electron density within a bulk crystal and thus provide important information concerning transformation mechanisms.

## B3: Match to Selection Criteria

### a. Meet the demands of an identified group of researchers for new techniques

The proposed ADS beamline is a highly sought after capability and, as demonstrated, it is well supported. There are a significant number of researchers interested in the installation of this beamline; an initial list of proponents is given at the beginning of this proposal. The beamline design aims to meet the existing needs of the current user community and provide for development opportunities and frontier research. It is also anticipated that there will be strong interest in the beamline from the commercial sector.

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## b. Take advantage of the existing third generation light source

The research to be undertaken at this beamline is largely impossible by other means and currently inaccessible in Australia; the ADS beamline will provide a range of opportunities for development of research programs in the Australasian region. The beamline will take advantage of the characteristics of the Australian Synchrotron third generation source, including the use of an insertion device, high spectral brightness, broad spectrum radiation, access to reasonably hard energy X-rays and the ability to obtain high flux density.

## c. Will position Australasian scientists at the leading edge of their field

The ADS beamline will provide the opportunity for Australasian researchers to be at the leading edge of many research areas, including the use of energy dispersive diffraction and materials characterisation techniques. The beamline will provide capabilities previously unavailable in Australia and will remove barriers to world class science conducted locally. The limited number of similar facilities around the world will also contribute to the nature of the forefront research being undertaken.

## d. Can be demonstrated to be feasibly constructed within a 3 year time frame

It is anticipated that this beamline will utilise standard components and systems. The distinctiveness of the beamline arises from the combination of accessible X-ray energies, the choice of detector systems and the use of various sample-related stages and ancillaries. When considering the high heat load and optical design requirements, it will be possible to take advantage of the experiences of the Australian Synchrotron Imaging and Medical beamline, in addition to a number of international facilities.

# B4: Potential Users

This project has been undertaken to address an existing need within the Australasian research community. A list of proponents of the beamline has been provided. Many are within active research groups that have current and on-going research projects requiring access to high energy diffraction capabilities as proposed herein. The experience of the existing powder diffraction beamline has shown that there is an extensive and growing user community awaiting appropriate facilities. The bending magnet beamline continues to receive approximately 25% of its applications from new users and demonstrates the wealth of research being undertaken in Australasia that requires access to facilities for materials characterisation and development, *in situ* studies, engineering studies and earth science research, to name a few. The research being undertaken has implications for energy production and storage, the environment, functional materials development and improvements to the mining and manufacturing sectors. However, the existing community is currently not satisfactorily serviced by the bending magnet beamline alone, due to the relatively low upper energy limit.

In addition to the applied and fundamental researchers it is predicted that there will be interest from the commercial manufacturing and engineering sectors in using this beamline, as has been observed at facilities such as ID15 at the ESRF.

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