# Diamond Phase III Beamline Proposal 058- S

# Large Area Detector Powder Diffraction Beamline

## A proposal prepared for the SAC March 2011

## Acknowledgements

The outline proposal was presented to the SAC by the Scientific Directors Trevor Rayment and Dave Stuart. This full proposal was produced by:

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| Dr Matthew Johnson | Industry | GSK |
| Dr Alistair Lennie | Earth Science | DLS |
| Prof Colin Pulham | Chemistry | Edinburgh |
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We thank the following DLS staff for their contributions to this proposal

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| Maura Launchbury | Head, Project Team |
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## Summary

The beamline set out in this proposal is a dedicated powder diffraction facility designed to complement the existing high-resolution powder diffraction beamline I11 by offering a radically different design concept. It is based on a bending magnet rather than undulator source, utilises large 2-dimensional (2D) area detectors rather than a 1-dimensional multi analyser-crystal array and will have the novel set up of two experimental hutches to provide versatility for both short and long duration experiments. The overall design will enable rapid (~ms to minutes) data collection for dynamical studies while simultaneously permitting long duration (days to months) experiments crucial for *in situ* studies (e.g. battery and fuel cell lifetimes). The higher energies will be suitable for PDF studies of, for example, amorphous organics of pharmaceutical interest, which exhibit virtually no signal at very high *Q*. Large 2D area detectors with variable sample-detector distances also make the proposed beamline well suited to those studies requiring access to low *Q*, e.g. powder protein studies, surface adsorbates and large superstructures. The new facility will make a massive impact in the crucial fields of materials related to energy (e.g. batteries, hydrogen fuel cells for the next generation vehicles, storage for nuclear waste, catalysts), the environment (e.g. $CO_2$ storage and mineralisation, corrosion issues), health care (pharmaceuticals, biominerals), and defence of the nation (e.g. energetic materials, forensics). With a versatile design and many automated features such as robotic sample changers, this beamline will be used by many academic researchers from diverse scientific backgrounds as well as from the pharmaceutical, nanotechnology, petroleum, minerals, defence, and speciality chemical industries.

# 2. Scientific Case

## 2.1 Powder Diffraction

The impact of powder diffraction on the UK economy and quality of life has never been greater: supporting studies of new materials for energy (fuel cells, batteries, nuclear, catalysts, gas storage substances)[1-21] to materials and minerals central to environmental issues (green chemistry, mineral dynamics, climate change, bio-mineralisation, corrosion)[22-25], health care (pharmaceuticals, biominerals)[26-36], homeland security (explosives, forensics)[37-40], electroceramics[41-42], new superconductors[43-44] and surface monolayers[45-47]. From the outset of SR source development, synchrotron X-ray powder diffraction (SXRPD) has become the crucial tool to probe structures of condensed matter on length scales ranging from sub-Angstrom, through the nanometre regime, to the micron level; at temperatures ranging from 2 to 2000 K; on timescales ranging from the order of seconds to tens of minutes; under conditions of ambient or applied pressure; under varying degrees of humidity, chemical environment, magnetic or electric field; and as materials are synthesised and self-organise from their constituent elements or building blocks. This is evidenced by the estimated number of publications from a Web of Science search based on SXRPD as shown in Fig. 1.

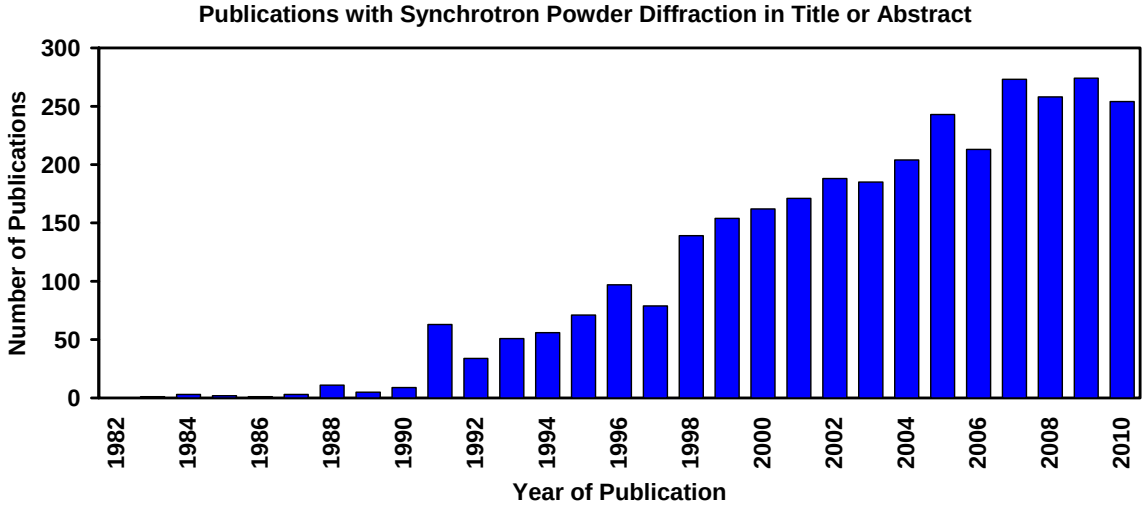


Fig 1. The number of publications specifically mentioning "synchrotron powder diffraction" in titles or abstracts.

With the development of the new Diamond Light Source (DLS), the user community requiring SXRPD facilities prioritised the build of an ultra high-resolution powder X-ray diffraction beamline for crystal structure solution and refinement. This specialised approach led to construction of the highly successful beamline I11[48-50]. This new proposal addresses aspects of powder diffraction facilities required by the UK community that could not be addressed in the original proposal for I11; these are therefore not currently being served by existing DLS beamlines. While I11 is designed for ultra high resolution studies of capillary samples over a 5–25 keV energy range, the proposed powder diffraction beamline would be based on the use of versatile large 2D area detectors, have more uniform flux from 8–40 keV, and be equipped to easily accept online analytical instruments and complex *in situ* and *in operando* sample chambers. This would enable and improve studies including:

- Rapid turnaround experiments and industrial applications (easy and fast access);
- Large scale structures ($d$ = 30–300 Å);

- Energy storage systems and fuel cell materials;
- High-energy powder diffraction and total scattering (PDF) studies;
- In-situ and fast kinetic studies (ms–μs) at non-ambient conditions (e.g. cryogenic and high temperatures, laser heating, and microwave);
- Crystal growth and design;
- Anomalous and amorphous scattering (DAF and PDF);

It is now standard practice for synchrotron sources worldwide to provide a range of powder diffraction beamlines optimised for different experiments in much the same way that neutron facilities such as ISIS have done with HRPD, GEM, and POLARIS. Thus, at ERSF, the versatile microfocus high-energy beamline ID11 with a large area detector complements the ultra high-resolution powder diffraction beamline ID31.

With regards to industrially relevant studies, many researchers from pharmaceutical companies supplement their in-house X-ray powder diffraction capabilities with SR diffraction, for example when tqckling particularly challenging cases such as solving structures of new active pharmaceutical ingredients, obtaining the highest quality data for phase quantification or determining accurate levels of crystallinity. Another industrial area supported by the technique is the study of catalysts, both transition metal based such as supported nanoparticles and also solid acids such as nanoporous zeolites, aluminophosphates, and metal-organic frameworks (MOFs). A third industrial area supported by SXRPD studies is electroceramics (ferroelectrics, capacitors, piezoelectrics etc.), where the structures of these often very complex and multiple-doped systems are critical for their operation, both exact compositions and processing conditions are determined by the information provided. Specific examples of scientific importance will continue to develop while this beamline is commissioned, although it is certain that powder diffraction will continue to provide the crucial insights allowing the rapid progress of a wide variety of physical sciences.

### 2.2 Community

The UK has a vibrant, mature and extensive SXRPD community. This has been built on research undertaken on stations such as 9.1, 2.3 and MPW6.2 at the SRS, Daresbury, and continues with beamlines I11 at DLS and ID31 at the ESRF. User community researchers come from disciplines including Chemistry, Physics, Materials Science/Engineering and Earth Sciences. It is strong in both Academia and Industry, including many cases of collaborative research undertaken in Universities by postgraduate students or postdoctoral research associates that is of direct relevance to supporting UK industry, often via Case studentships. An industrial powder diffraction community already uses I11 and we anticipate it further increasing in size and activity with this new beamline. One of the UWG members is from GSK and has provided invaluable advice with regards design and operation to ensure the beamline meets the needs of this community whilst still maintaining a strong, underlying focus on the needs of the Academic community.

### 2.3 Experimental Strengths of the Beamline

This line will complement the existing RCUK funded high-resolution powder diffractometers I11 (DLS), ID11 and ID31 (ESRF). The most important four areas where it will be superior to these instruments are highlighted below. The overall design with two experimental hutches and the ability to accommodate long duration experiments will be unique in the world, although we do note that the conceptual design for the new powder diffractometer at NSLS-II includes similar hutches as part of a possible future upgrade.

### 2.3.1 Rapid Data Collection for Dynamical Studies

Powder diffraction is ideally suited for studying the change in crystalline solids as a function of time under a range of experimental conditions. With the advent of brighter synchrotron sources and better detectors, there has been a push to monitoring changes at ever faster rates and in ever more complex cells. Notable recent examples are studies of batteries under electrochemical control[7,8], hydrogen storage materials under variable gas and temperature conditions[14], piezoelectric ceramics under electric fields[42], minerals or high strength alloys at high temperature[51,52] and the optimisation of industrial hydrothermal processes[53]. This beamline will be ideally suited to these and related studies (e.g. precipitation/crystallisation of catalyst particles or minerals, following fast reactions of energetic materials) as it will have good flux at a range of energies and rely on area detectors. Area detectors not only facilitate fast data collections, they have the additional benefit that integration of full Debye-Scherrer rings will reduce the texture problems that often occur *in situ* as samples rarely become/stay random powders in many cells.

### 2.3.2 Long Duration Experiments (LDEs)

An important design feature is to have two experimental hutches, where the second will be optimised to house complex sample environment cells and/or experiments that need to be monitored periodically over long (weeks-months) time periods. LDEs will be set up and left in place with programmed automated data collections. There will be a high precision table and robot to ensure reproducible sampling of exactly the same place(s) in the cells. This will be a particular strength for certain research areas such as batteries and fuel cells (section 2.4.1) where important information on the development of phases over time will allow full lifetime modelling and design optimisation. Other research areas that will benefit from this provision include studies of crystallisation, mineral evolution, thermal cycling and corrosion science (section 2.4.2). A means to manage this unique mode of operation is set out in section 4.3.

### 2.3.3 High Energy/PDF Studies

The ability to collect routine powder diffraction patterns to 40 keV with an optimised area detector is a key feature of the second end station. High energy photons are much preferred for many *in situ* studies as reduced absorption coefficients allow more flexibility in the choice of materials and design (a smaller aperture for diffraction patterns) of *in situ* cells. At small sample-to-detector distances and with a currently available 2D flat plate detector (PE, Pixium), it will be possible to collect data to a $Q_{max}$ of ~25 Å$^{-1}$. This will open up the possibility for medium resolution Pair Distribution Function (PDF) studies[54].† We believe 40 keV is a reasonable working energy and there should only be a handful of elements that will need to be avoided due to fluorescence.

### 2.3.4 Low-*Q* Studies

To study any material with a large unit cell it is necessary to be able to accurately measure diffraction peaks at low *Q* values. Examples include work on proteins[36], surface adsorbates[45-47] or materials with very large superstructures such as the classic example of the "simple" binary alloy $Cu_3Sn$ with an orthorhombic unit cell with one edge of nearly 48 Å[55]. This is very challenging on many diffractometers relying on the use of an analyser crystal arrangement where the sample-to-detector distance is fixed and usually optimised to collect high resolution data to large *Q* values. For example, although the recent work by Clarke and his co-workers of C6-C13 monolayers on

† Given the SAC is considering a dedicated PDF beamline we will not describe the technique, scope or utility.

graphite substrates was successful using I11[45-47], the studies were restricted by the low angular limit of the instrument ($Q \sim 0.2$ Å$^{-1}$). With the use of a 2D detector and variable sample-to-detector distance one can optimise the *Q*-range and angular resolution to obtain better data than would currently be possible on lines such as I11 or ID31.

### 2.4 Scientific Impact

#### 2.4.1 Energy Materials

Novel and improved methods for energy storage are urgently required to make more efficient use of our dwindling and *finite* supply of fossil fuels, to increase energy autonomy and to enable effective use of renewable energy sources. Two aspects of this beamline will be of particular utility for underpinning energy research, the ability to run long duration studies with periodic monitoring and PDF studies.

**Fuel cells** are an active area of research in many academic and industrial laboratories in the UK, predominantly Solid Oxide (SOFC) and Proton Exchange Membrane (PEMFC) designs. Powder diffraction can be used in fuel cell research to study the anode in a SOFC which is typically a crystalline ceramic oxide ion conductor such as yttria-stabilised zirconia (YSZ)[1] or the catalysts at the heart of the PEMFC anode and cathode, typically finely divided platinum and its alloys. With respect to the metal catalysts, most research is focussed on increasing the stability and activity through means such as changing alloy compositions and producing stable nanoparticles[2]. With respect to the SOFC electrolyte, research is generally focussing on finding new ceramics with increased oxide ion mobility at lower temperatures (the standard operating temperature for YSZ is ca. 950°C) as this is one of the major limitations for wide-scale use. A variety of other systems are currently being explored in the UK including doped apatites and cerium niobates[3,4]. Great progress could be made through studying active fuel cells over long time periods in order to fully understand the changes in composition and structure that occur during operation and ultimately lead to failure.

**The lithium ion battery** (LIB) is, in principle, the most practical battery for most energy storage applications since it is fabricated from light, Li-containing electrodes, and provides high working cell voltages (typically over 3.5 V). Although significant progress has been made in battery technology, applications such as transportation (e.g., in hybrid and electric vehicles) and grid storage impose tremendous requirements on these devices in terms of capacity, capacity retention, rate, safety and cost. Continuing improvements will not come without a more thorough understanding of how these materials function[5,6]. This requires studying how electrode materials transform as lithium is repeatedly extracted/inserted. SXRPD methods allow structural transformations to be monitored in real time for functioning LIBs[7,8].

A major challenge is the need to understand electrode processes over multiple time and length scales. For example, the need to monitor a structural transformation that may take hours, days or even years (for transformations that occur gradually over multiple cycles), does not readily mesh with the design of high energy sources that are optimized for processes that occur very rapidly. One solution to this is to design sample holders that can hold and allow multiple batteries to be continuously electrochemically cycled over periods of days to months and periodically returned to exactly the same position in the synchrotron beam, via a robot arm, so as to collect another diffraction pattern and monitor slow chemical processes.

**Nuclear power** is again on the agenda as a medium term solution to meet a significant fraction of the UK's energy needs through new reactor build, and there is an increased emphasis on involving

UK Universities in the research process[56]. A key aspect of ensuring the safety and public acceptance of new reactors is to plan appropriate measures to account for the long term issue of radioactive waste disposal. High level waste (HLW) accounts for ca. 95% of the total activity from power generation and is typically a mixture of fission products and transuranics. The current preferred wasteforms in the UK for the HLW produced from reprocessing are made by incorporating it into a borosilicate or phosphate glass or cement mixture. Another type of nuclear waste to be dealt with is the significant quantity of spent ion exchange materials, e.g. zeolites or titanosilicates, which have been used to decontaminate storage ponds or aqueous effluent.

Finding answers to key research questions leading to better wasteforms would be facilitated by this beamline[9]. Vitrification rarely incorporates all of the elements in a HLW and there are issues of devitrification or leaching with time due to radiation and/or heat damage. Cemented HLW suffers similar problems leading to a complex mixture of crystalline and amorphous phases. In both cases it is important to determine the exact nature of the crystalline phases, the degree of element incorporation and doping and how both of these change with time. This underpins both the science and the technical safety case. A high throughput approach to examine many variations of wasteforms with powder diffraction data of sufficient quality for the quantification of phases present at very low levels is important. The ability to perform long duration experiments under relevant repository conditions would be unique to this beamline. Temporal changes within the samples could be monitored, especially unwanted crystallisation, and be used to form the safety case of a geological disposal facility avoiding the potential pitfalls of using accelerated alteration experiments or predictive modelling. PDF experiments would lead to determination of the short and medium range structures within the glasses and coordination environments of the radionuclides. Long duration studies with periodic collection of PDF data would be a means to investigate radiation damage mechanisms where α-recoil leads to partial amorphisation of the initially crystalline ceramic host[10]. Any of these studies would guide formulation design and processing.

For ion exchange materials, such as the currently used zeolite clinoptilolite or titanosilicate CST found in IONSIV IE-911 or others being assessed for future use including the titanate SrTreat, the layered metal sulphide KMS-1 and layered M(IV) phosphates, rapid *in situ* data acquisition would be used to study the exchange processes as has been done for pure CST[11]. Cation locations within the frameworks and how these change as a function of time and temperature are critical to develop a fundamental understanding of the process and improve materials. In many porous solids the cations of interest are poorly ordered, so complimentary PDF studies could be used as a way to determine local environments and binding sites.

**Heterogeneous catalysis** underpins the chemistry industry, being used in the synthesis of 80% of its products. A 1% improvement in the activity of a catalyst will lead to increased efficiency in energy usage and will save a company millions of pounds. SXRPD has long been used, particularly powerfully in conjunction with other techniques[12-13]. For metal catalysts, there is an ever increasing emphasis on high surface area nanoparticles. Fast time domain diffraction studies of the formation and change of metal particle catalysts, especially *in operando* in suitably designed reaction cells, followed by examination of PDF patterns after formation can be used for intelligent design of catalytic systems and operating conditions. A second type of important heterogeneous catalyst is a nanoporous solid with the active sites within the pores, such as an acidic site in a zeolite or aluminophosphate or transition metal site in a metal organic framework (MOF). Understanding the structures, how they change during catalytic cycles and in favourable cases observing the binding of reactants is best achieved using powder diffraction in combination with spectroscopy and/or

physical measurements. The ability to set up complex reaction cells to study these systems *in operando* with simultaneous monitoring by, e.g., IR or Raman spectroscopy will make this beamline particularly suited.

**Gas storage** studies will include those relevant to the use of fuel cells (hydrogen storage) and also to produce energy more cleanly (carbon dioxide storage). Systems where the gas is chemically locked into the solid, such as B-N-H sytems for hydrogen storage[14] or carbonates for $CO_2$ storage, as well as porous solids[15], e.g. MOFs, that chemisorb or physisorb relevant gases will both be studied at this beamline[16-20]. The quality of the diffraction patterns will be suitable to solve unknown structures of moderately complex systems, data from this beamline will also be used for *in situ* studies to follow changes during gas sorption and desorption. This will allow a fuller understanding of the factors and conditions favouring the locking up of these gases. For hydrogen storage materials, it is important to develop an understanding of the absorption and desorption mechanisms and the structures of the desorbed solids at realistic pressure and temperature conditions.

**Phase-change materials** such as salt hydrates, hydrocarbons and metal eutectics are receiving increasing interest as heat storage materials in domestic and industrial applications. The technology has the potential to make a very significant impact on worldwide use of energy with concomitant reductions in the level of $CO_2$ emissions. Such chemical compounds absorb heat and undergo a phase transition, e.g. dissolution or melting. On cooling, the reverse occurs, e.g. crystallisation or freezing, and heat is released. Whilst conventional phase-change materials have an acceptable level of energy density to support development of a compact heat store, there remain challenges that include: reproducible performance over many cycles, ensuring changes occur at appropriate temperature ranges, tuning the volume-energy density ratio, ensuring efficient thermal conductivity and maintaining long term chemical stability. The issue of compromised performance is particularly important and has been attributed to: crystallisation of other, less desirable phases; nucleation and crystal growth processes; and the presence of short-lived intermediate phases. An example of an intermediate phase found in the crystallisation of aqueous solutions of sodium sulfate has recently been described[21]. Hence the ability to monitor and study phase changes *in situ*, in real time and under controlled temperature and humidity conditions using the new beamline would allow researchers to address these issues.

### 2.4.2 Environmental Science

With increased public awareness of the impact of human activity on both the climate and environment (waste treatment, remediation and pollution) SXRPD is well placed to make significant contributions to many different aspects in this area.

**Corrosion** of built structures, plant machinery and transport has a very obvious economic impact as well as potential environmental and health impacts. As a predominantly electrochemical process, much attention has been given to applying electrochemical techniques in the laboratory to understand, prevent and treat corrosion. In the real world, corrosive processes take place in hostile environments and until very recently, potential electrochemical conservation/preservation methods have always been evaluated using *ex situ* techniques requiring samples to be transferred out of their host environments. This change in environment invariably alters the surface and near-surface layers so the development of SXRPD *in situ* techniques via specialised corrosion cells are critical to accurately understand reactions occurring at electrodes[22]. Furthermore, the electrode potential can be maintained during the whole measurement process while simultaneously applying traditional

electrochemical analyses. All of these aspects are crucial to the interpretation of time dependent reactions. *In situ* corrosion studies would be ideally suited to the LDE facility proposed for this beamline, while the focussed beam introduces the possibility of monitoring the spatial aspects of corrosion growth in any given sample.

**$CO_2$ sequestration** has gained in importance with the acceptance that the increased emission of greenhouse gases, such as $CO_2$, during the industrial era has resulted in a rise in the average surface temperature of the Earth. Rising temperatures may, in turn, generate changes in precipitation patterns, storm severity and sea level, commonly referred to as climate change and there is currently much emphasis being placed on $CO_2$ sequestering technologies. Although many schemes have been proposed, a large number rely on either slow methods (e.g. forestation, plant/algae or microbe uptake), or long term storage (e.g. injection into the wellbores of depleted oil and gas reservoirs) which pose long term stability risks in terms of chemical stability and sudden leakage. Over slow geological time $CO_2$ is sequestered in rocks via weathering processes involving the carbonation of minerals, which occurs naturally by the reaction between alkaline materials and atmospheric $CO_2$. The development of materials that undergo accelerated carbonation[23] over a time scale of hours is very attractive and have been applied to the stabilization of solid wastes generated from coal fired power stations and other types of combustion residues, including de-inking and paper mill ash and municipal solid waste ash[57]. Carbonation here involves injecting high purity $CO_2$ at high pressure to promote the formation of stable phases such as Ca or Mg carbonate[23]. Currently accelerated carbonation materials are almost entirely composed of the mined Ca-silicate mineral wollastonite and large ore deposits of economic value are rare. Thus to turn accelerated carbonation into a viable technology requires the development of new materials whose structures, compositions and form are optimised for rapid carbonation. Optimisation of for maximum gas uptake is well matched to the provision of *in situ* facilities for studying incorporation of gases into solid structures under real-world operating conditions using the long duration facility.

**Mineral precipitation and dynamics** in the ocean environment are particularly important to understand for carbonate phases given their prevalence in the biosphere and important role in climate change. One of the first minerals predicted to precipitate during the formation of sea ice is calcite. However in the natural environment it is the hydrated carbonate ikaite ($CaCO_3.6H_2O$) that precipitates. This highlights a lack of knowledge about both the phase(s) and the dynamics of $CaCO_3$ minerals in sea ice that currently limits our understanding of carbon dynamics and, most importantly, their contribution to the carbon cycle in the polar oceans[58,59] which directly effects carbon dioxide drawdown[24] and hence climate change[60]. Using the proposed beamline, the dynamics of anhydrous and hydrated carbonate polymorphs could, for the first time, be studied under realistic long term environmental conditions (i.e. lower temperatures, increasing ionic strength) and time scales using a large sample chamber with a controlled partial pressure of $CO_2$.

### 2.4.3 Health and Society

**Pharmaceutical** research remains one of the United Kingdom's most important scientific sectors with all of the biggest companies (e.g. GSK, Pfizer, Sanofi-Aventis, AstraZeneca, Novartis[61]) having major research laboratories in the UK. These companies add significantly to the wealth of UK plc and represent a scientific sector in which the UK is a world leader. Powder X-ray diffraction is one of the major techniques used for the characterisation of pharmaceuticals and a XRPD pattern is now a USA FDA requirement for polycrystalline substances with medical applications, and is particularly crucial for patent protection. In the pharmaceutical industry,

currently only the most challenging samples are sent to SR facilities, due to cost/time/access considerations. There are analytical gaps to be resolved: crystallisation studies (growth in solvent, morphology variation, conversion of solvate to non-solvate forms, formation of intermediate forms) with improved time and spatial resolution; studying amorphous compounds (phase identification and stability control); and studying complex formulations (characterising the drug compound in a product, e.g. crystalline v amorphous). In contrast to academic studies, rapid access to SR facilities is crucial for SXRPD studies for both the development of new pharmaceuticals or for protecting existing ones in intellectual property disputes. SXRPD data is already being used as evidence in patent cases and such data will appear more frequently as such facilities become more common worldwide.

In academic pharmaceutical research, XRPD has played a major role: methods for *ab-initio* structure solution from XRPD have been developed by various UK groups[62] and the most successful software package from this research has been spun out commercially here in the UK[63]. Recent applications of SXRPD in academic pharmaceutical research include: the development of a synchrotron X-ray diffraction method to monitor phase transitions during entire freeze-drying cycles[26], grinding studies on α-lactose monohydrate (one of the most common excipients in pharmaceutical tablets)[27], determination of pharmaceutical crystal structures[28,29], and *in situ* dehydration studies[30]. One of the conclusions from the latter study by Nunes *et al.* is that two-dimensional powder X-ray diffractometry, using a high intensity source, is a powerful technique to study the kinetics of rapid solid-state reactions: their experiments required a time resolution ranging from 40 ms to 30 s and an area detector covering the angular 2θ range of 3° to 27°. These exemplars demonstrate the wide variety of applications of powder diffraction to scientific problems in pharmaceutical research.

A recent development in the pharmaceutical sector is the production of kinetically-stable amorphous products produced, e.g. by spray drying. This is a hotly debated research area, and has even been the subject of patent disputes in USA federal courts (e.g. Hoffmann–La Roche v. Ranbaxy over the drug valganciclovir sold by the plaintiffs in its crystalline form as Valcyte®, but developed by Ranbaxy in an amorphous form). There is even the current question of whether amorphous products can have different structures and whether these can be characterised reliably by PDF methods[31]. This beamline will produce suitable PDFs to support this area of research.

**Biominerals/biomaterials** is another increasingly important research area in the field of human health care with hip and knee joint replacements now becoming routine operations in an ageing population. There is an increasing body of evidence that prosthetic implants based upon titanium alloys attain improved performance when coated with calcium hydroxyapatite. Such coatings appear to promote osteointegration and bone in-growth. Detailed chemical and structural characterisation is essential for (i) coating technology development, (ii) ensuring consistent material quality and (iii) assessing performance. Combined conventional powder diffraction and glancing angle synchrotron diffraction can be used to examine the *in vitro* performance of apatite coatings formed by plasma spraying[32–34]. The use of micro beams now available at SR sources allows the *in situ* kinetic study of nanocrystals in biomineralization processes such as bone formation as a function of pH, under macroscopic strain, or in the presence of organic molecules. The proposed beamline will be ideally suited to such studies.

### 2.4.4 Energetic Materials

Energetic materials such as propellants, pyrotechnics and explosives have a wide range of applications including mining, space exploration, car safety and emergency signalling. These materials release heat and/or gaseous products at a high rate upon external stimulus. In order to model and understand the characteristics and performance of these materials, it is essential to obtain detailed structural information over a range of conditions of temperature and pressure, including the structural changes that occur during fast chemical reactions. This information can be used to explore aspects of energetic materials that include: sensitivity to shock, heat, and friction; chemical decomposition mechanisms; energy transfer through the solid; detonation velocities; and testing the efficacy of theoretical modelling techniques. This enhanced understanding is of key importance for the development of less sensitive compositions for safer operations. For this reason, the areas of modelling, material characterisation, and test and evaluation have been highlighted as priority technologies for the UK Ministry of Defence[64]. Recent work at I11 has focussed on the effects of pressure and temperature on the structures of the explosives RDX[37-39] and CL-20[40]. In addition to the determination of the crystal structures of the high-pressure phases, the research has also highlighted the potential for the recovery of high-pressure/high-temperature phases back to ambient conditions – such recovered phases may display enhanced properties such as higher density and reduced sensitivity.

An emerging challenge is to study *in situ* structural and chemical changes in energetic materials during pre-combustion (slow decomposition) and combustion (rapid decomposition) processes. Reaction fronts that span the velocity range $10^{-2}$ to $10^{5}$ mm $s^{-1}$ are typically encountered in such processes and recording diffraction data in order to map the evolution of crystal structures at these higher reaction rates is clearly challenging, but crucial for the development of reliable models to predict the energy-transfer and chemical processes that are invaluable for the prediction of the onset of the *deflagration-to-detonation transition* in which a fiercely burning reaction accelerates to become a detonation – with obvious implications for the safety of surrounding personnel.

## 2.5 Relevance to Sponsoring Bodies and UK Industry

There are currently seven well-defined RCUK cross-council research themes. Of these, the research areas outlined above support:

- *Ageing, Lifelong Health and Wellbeing* are key priorities for pharmaceutical companies, which have a representative on the UWG and letters of support have been obtained from researchers at GSK and AstraZeneca.
- *Energy* is stated on the RCUK web site as "at the top of our national and international policy agenda". This beamline would support research into batteries, fuel cells, nuclear power, catalysts and hydrogen gas storage. It will also support the development of new lower energy processing routes. Key industrial support comes under this heading with letters from researchers at Johnson Matthey and Infineum.
- *Living with Environmental Change* can be better predicted or mitigated by research this beamline will facilitate to help understand some of the fundamental environmental science problems such as biogeochemical processes leading to mineral formation and climate change, particularly carbonate species in aqueous environments where $CO_2$ is a major component. It would also support research into the design and use of novel materials for carbon capture and storage such as porous inorganic solids (zeolites, MOFs).

- *NanoScience through Engineering to Application* will be supported by powder diffraction studies to help to understand the formation process and atomic structure of nanoparticles.
- *Global Uncertainties; Security for All in a Changing World* where the beamline would support studies into the characteristics and performance of explosives to enhance the UK's energetics capability and its ability to counter potential terrorist threats.

We note that powder diffraction studies of active pharmaceutical ingredients and biomaterials feature in the research plan of the Wellcome Trust. Solving crystal structures of small proteins using powder diffraction data collected with area detectors has been demonstrated at the APS[36], and we believe this beamline will attract this type of study.

Many UK companies are operating in an increasingly competitive, global environment. It is therefore vital that they maintain their competitive edge through continual innovation; inevitably this will require increasingly closer links with world-leading expertise in UK universities and at Diamond Light Source. As has been highlighted earlier in this proposal, powder diffraction studies are of relevance to a wide range of industries and the proposed beamline would allow access either directly or via academic collaborators to an unrivalled facility.

## 2.6 References

1. A Atkinson, S Barnett, RJ Gorte, JTS Irvine, AJ McEvoy, M Mogensen, SC Singhal, J Vohs, "Advanced anodes for high-temperature fuel cells." *Nature Mater.* 3, 17–27 (2004).
2. CJ Zhong, J Luo, B Fang, BN Wanjala, PN Njoki, R Loukrakpam, J Yin, "Nanostructured catalysts in fuel cells." *Nanotechnology*, **21**, 062001 (2010).
3. JR Tolchard, PR Slater, MS Islam, "Insight into doping effects in apatite silicate ionic conductors." *Adv. Functional Mater.* **17**, 2564–2571 (2007).
4. RJ Packer, SJ Skinner, "Remarkable oxide ion conductivity observed at low temperature in a complex superstructured oxide." *Adv. Mater.* **22**, 1613–1616 (2010).
5. KS Kang, YS Meng, J Breger, CP Grey, G Ceder, "Electrodes with high power and high capacity for rechargeable lithium batteries." Science, 311, 977–980 (2006).
6. AR Armstrong, M Holzapfel, P Novak, CS Johnson, SH Kang, MM Thackeray, PG Bruce, "Demonstrating oxygen loss and associated structural reorganisation in the lithium battery cathode $Li[Ni_{0.2}Li_{0.2}Mn_{0.6}]O_2$." *J. Am. Chem. Soc.* **128**, 8694–8698 (2006).
7. J McBreen, "The application of synchrotron techniques to the study of Li-ion batteries." *J. Sol. St. Electrochem.* **13**, 1051–1061 (2009).
8. JB Leriche, S Hamelet, J Shu, M Morcrette, C Masquelier, G Ouvrard, M Zerrouki, P Soudan, S Belin, E Elkaïm, F Baudelet, "An Electrochemical Cell for *Operando* Study of Lithium Batteries Using Synchrotron Radiation.", *J. Electrochem. Soc.* **157**, A606–A610 (2010).
9. T Schafer, MA Denecke, "Nuclear waste repository research at the micro- to nanoscale." *AIP Conf. Proc.* **1221**, 181–187 (2010).
10. I Farnan, H Cho, WJ Weber, "Quantification of actinide α-radiation damage in minerals and ceramics." *Nature*, **445**, 190–193 (2007).
11. A Clearfield, A Tripathi, D Medvedev, AJ Celestian, JB Parise, "In situ type study of hydrothermally prepared titanates and silicotitanates" *J. Mater. Sci.* **41**, 1325–1333 (2006).
12. SDM Jacques, O Leynaud, D Strusevich, P Stukas, P Barnes, G Sankar M Sheehy, MG O'Brien, A Iglesias-Juez, AM Beale, "Recent progress in the use of in situ X-ray methods for the study of heterogeneous catalysts in packed-bed capillary reactors." *Catalysis Today*, **145**, 204–212 (2009).
13. MA Newton, W van Beek, "Combining synchrotron-based X-ray techniques with vibrational spectroscopies for the in situ study of heterogeneous catalysts: a view from a bridge." *Chem. Soc. Rev.* **39**, 4845–4863 (2010).
14. DB Ravnsbaek, Y Filinchuk, R Cerny, TR Jensen, "Powder diffraction methods for studies of borohydride-based energy storage materials", *Z. Kristallogr.* **225** 557–569 (2010).
15. AK Cheetham, CNR Rao, RK Feller, "Structural diversity and chemical trends in hybrid inorganic-organic framework materials." *Chem. Comm.* **46**, 4780–4795 (2006).

16. J Rabone, Y-F Yue, SY Chong, KC Stylianou, J Bacsa, D Bradshaw, GR Darling, NG Berry, YZ Khimyak, AY Ganin, P Wiper, JB Claridge, MJ Rosseinsky. "An Adaptable Peptide-Based Porous Material." *Science,* **329,** 1053–1057 (2010).
17. T Tozawa, JTA Jones, SI Swamy, S Jiang, DJ Adams, S Shakespeare, R Clowes, D Bradshaw, T Hasell, SY Chong, C Tang, S Thompson, J Parker, A Trewin, J Bacsa, AMZ Slawin, A Steiner, AI Cooper. "Porous organic cages." *Nature Materials* **8**, 973–978 (2009).
18. MT Wharmby, JPS Mowat, SP Thompson, PA Wright, "Extending the pore size of crystalline metal phosphonates toward the Mesoporous regime by isoreticular synthesis." *J. Am. Chem. Soc.* in press.
19. Z Han, AL Picone, AMZ Slawin, VR Seymour, SE Ashbrook, W Zhou, SP Thompson, JE Parker, PA Wright, "Novel large-pore aluminophosphate molecular sieve STA-15 prepared using the tetrapropylammonium cation as a structure directing agent." *Chem. Mater.* **22**, 338–346 (2010).
20. PM Forster, J Eckert, BD Heiken, JB Parise, JW Yoon, SH Jhung, JS Chang, AK Cheetham, "Adsorption of molecular hydrogen on coordinatively unsaturated Ni(II) sites in a nanoporous hybrid material." *J. Am. Chem. Soc.* **128**, 16846–16850 (2006).
21. IDH Oswald, A Hamilton, C Hall, WG Marshall, TJ Prior, CR Pulham, "In-situ characterization of elusive salt hydrates - the crystal structures of the heptahydrate and octahydrate of sodium sulfate." *J. Am. Chem. Soc.* **130**, 17795–17800 (2008).
22. K Leyssens, A Adriaens, MG Dowsett, B Schotte, I Oloff, E Pantos, AMT Bell, SP Thompson, "Simultaneous in situ time resolved SR-XRD and corrosion potential analyses to monitor the corrosion on copper". *Electrochem. Comm.* **7**, 1265–1270, 2005.
23. M Lim, GC Han, JW Ahn, KS You, "Environmental remediation and conversion of carbon dioxide ($CO_2$) into useful green products by accelerated carbonation technology". *Int. J. Environ. Res. Public Health* **7**, 203-228, 2010.
24. GS Dieckmann, G Nehrke, S Papadimitriou, J Göttlicher, R Steininger, H Kennedy, D Wolf-Gladrow, and DN Thomas, "Calcium carbonate as ikaite crystals in Antarctic sea ice". *Geophys. Res. Lett.*, **35**, L08501, 2008.
25. S Kellici, K Gong, T Lin, S Brown, RJH Clark, M Vickers, JK Cockcroft, V Middelkoop, P Barnes, JM Perkins, CJ Tighe, JA Darr. "High-throughput continuous hydrothermal flow synthesis of Zn–Ce oxides: unprecedented solubility of Zn in the nanoparticle fluorite lattice." *Phil. Trans. Royal Soc.* **368**, 4331–4349 (2010).
26. DB Varshney, P Sundaramurthi, S Kumar, EY Shalaev, SW Kang, LA Gatlin, R Suryanarayanan, "Phase transitions in frozen systems and during freeze-drying: quantification using synchrotron X-Ray diffractometry." *Pharm. Research* **26**, 1596–1606 (2009).
27. S Garnier, S Petit, F Mallet, MN Petit, D Lemarchand, S Coste, J Lefebvre, G Coquerel. "Influence of ageing, grinding and preheating on the thermal behaviour of α-lactose monohydrate." *Int. J. Pharmaceutics*, **361,** 131–140 (2008).
28. P Fernandes, K Shankland, AJ Florence, N Shankland, A Johnston. "Solving molecular crystal structures from X-ray powder diffraction data: the challenges posed by γ-carbamazepine and chlorothiazide N,N,-dimethylformamide (½) solvate." *J. Pharm. Sciences.* **96,** 1192–1202 (2007).
29. E Nishibori, T Ogura, S Aoyagi, M Sakata. "Ab-initio structure determination of a pharmaceutical compound, prednisolone succinate, from synchrotron powder data by combination of a genetic algorithm and the maximum entropy method." *J. Appl. Cryst.* **41**, 292–301 (2008).
30. C Nunes, A Mahendrasingam, R Suryanarayanan. "Investigation of the multi-step dehydration reaction of theophylline monohydrate using 2-dimensional powder X-ray diffractometry." *Pharm. Research* **23**, 2393–2404 (2006).
31. F Atassi, C Mao, AS Masadeh, SR Byrn "Solid-State Characterization of Amorphous and Mesomorphous Calcium Ketoprofen." *J. Pharm. Sciences.* **99,** 3684–3697 (2010).
32. K Rogers, S Etok, A Broadhurst, R Scott. "Enhanced analysis of biomaterials by synchrotron diffraction." *Nucl. Inst. Methods in Phys. Res.* **A548**, 123-128 (2005).
33. CJS Ibsen, H Birkedal. "Modification of bone-like apatite nanoparticle size and growth kinetics by alizarin red S." *Nanoscale*, **2**, 2478–2486 (2010).
34. A Carrado. "Structural, microstructural, and residual stress investigations of plasma-sprayed hydroxyapatite on Ti-6Al-4V." *ACS Appl. Materials & Interfaces* **2**, 561–565 (2010).
35. KC Stylianou, R Heck, SY Chong, J Bacsa, JTA Jones, YZ Khimyak, D Bradshaw, MJ Rosseinsky (2010) "A Guest-Responsive Fluorescent 3D Microporous Metal-Organic Framework Derived from a Long-Lifetime Pyrene Core." *J. Am. Chem. Soc.* **132,** 4119–4130 (2010).
36. JA Doebbler, RB Von Dreele, "Application of molecular replacement to protein powder data from image plates." *Acta Cryst.* **D65**, 348–355 (2009).

37. DIA Millar, IDH Oswald, C Barry, DJ Francis, WG Marshall, CR Pulham, AS Cumming, "Pressure-cooking of explosives - the crystal structure of ε-RDX as determined by X-ray and neutron diffraction." *Chem. Commun*. **46,** 5662–5664 (2010).
38. DIA Millar, HE Maynard-Casely, AK Kleppe, WG.Marshall, CR Pulham, AS Cumming, "Putting the squeeze on energetic materials - structural characterisation of a high-pressure phase of CL-20." *CrystEngComm.* **12**, 2524–2527 (2010).
39. DIA Millar, IDH Oswald, DJ Francis, WG Marshall, CR Pulham, AS Cumming, "The crystal structure of β-RDX - an elusive form of an explosive revealed." *Chem. Commun.* **5**, 562 (2009).
40. AJ Davidson, IDH Oswald, DJ Francis, AR Lennie, WG Marshall, DIA Millar, CR Pulham, JE Warren, AS Cumming, "Explosives under pressure - the crystal structure of γ-RDX as determined by high-pressure X-ray and neutron diffraction." *CrystEngComm*. **10**, 162 (2008).
41. KE Johnston, CC Tang, JE Parker, KS Knight, P Lightfoot, SE Ashbrook. "The Polar Phase of $NaNbO_3$: A Combined Study by Powder Diffraction, Solid-State NMR and First-Principles Calculations." *J. Am. Chem. Soc*. **132**, 8732–8746 (2010).
42. M Hinterstein, M Knapp, M Hölzel, W Jo, A Cervellino, H Ehrenberg, H Fuess "Field-induced phase transition in $Bi_{1/2}Na_{1/2}TiO_3$-based lead-free piezoelectric ceramics." *J. Appl. Cryst.* 43, 1314–1321 (2010).
43. AY Ganin, Y Takabayashi, P Jeglič, D Arčon, A Potočnik, PJ Baker, Y Ohishi, MT McDonald, MD Tzirakis, A McLennan, GR Darling, M Takata, MJ Rosseinsky, K Prassides. "Polymorphism control of superconductivity and magnetism in $Cs_3C_{60}$ close to the Mott transition." *Nature* **466**, 221–225 (2010).
44. MJ Pitcher, T Lancaster, JD Wright, I Franke, AJ Steele, PJ Baker, FL Pratt, WT Thomas, DR Parker, SJ Blundell, SJ Clarke, "Compositional control of the superconducting properties of LiFeAs." *J. Am. Chem. Soc.* **132**, 10467–10476 (2010).
45. TK Phillips, T Bhinde, SM Clarke, SY Lee, "Adsorption of Aldehydes on a Graphite Substrate: Combined Thermodynamic Study of C6-C13 Homologues with a Structural and Dynamical Study of Dodecanal." *J. Phys. Chem.* **C 114**, 6027–6034 (2010).
46. T Bhinde, SM Clarke, TK Phillips, T Arnold, JE Parker, "Crystalline Structures of Alkylamide Monolayers Adsorbed on the Surface of Graphite." *Langmuir*, **26**, 8201–8206 (2010).
47. TK Phillips, SM Clarke, T Bhinde, MA Castro, C Millan, S Medina, "Monolayer Structures of Alkyl Aldehydes: Odd Membered Homologues." *Thin Solid Films*, in press.
48. CC Tang, SP Thompson, TP Hill, GR Wilkin and UH Wagner. "Design of powder diffraction beamline (BL-I11) at Diamond". *Z. Krist, Suppl.* **26** 153-158 (2007).
49. SP Thompson, JE Parker, J Potter, TP Hill, A Birt, TM Cobb, F Yuan, and CC Tang. "Beamline I11 at Diamond: A new instrument for high resolution powder diffraction". *Rev. Sci. Inst.* **80**, 075107 (2009).
50. JE Parker, SP Thompson, TM Cobb, F Yuan, J Potter, AR Lennie, S Alexander, CJ Tighe, JA Darr, JC Cockcroft and CC Tang. "High-throughput powder diffraction on beamline I11 at Diamond". *J. Appl. Cryst.* **44**, 102–110 (2011).
51. S Ori, E Mazzucato, G Vezzalini, "Dehydration dynamics of barrerite: An in situ synchrotron XRPD study", *Am. Mineral.*, 94, 64–73 (2009).
52. K-D Liss, RE Whitfield, W Xu, T Buslaps, LA Yeoh, X Wu, D Zhang, K Xia "*In situ* synchrotron high-energy X-ray diffraction analysis on phase transformations in Ti-Al alloys processed by equal-channel angular pressing." *J. Synchrotron Rad.* **16**, 825-834 (2009).
53. NAS Webster, IC Madsen, MJ Loan, NVY Scarlett, K S Wallwork, "A flow cell for *in situ* synchrotron x-ray diffraction studies of scale formation under Bayer processing conditions." *Rev. Sci. Instrum.*, **80**, 084102 (2009).
54. T Egami and SJL Billinge, *Underneath the Bragg Peaks: Structural Analysis of Complex Materials*, ed. R Cahn, Pergamon Press, Oxford, 2004
55. Y Watanabe, Y Fujinaga, H Iwasaki, "Lattice modulation in the long-period superstructure of $Cu_3Sn$." *Acta Cryst.* **B39**, 306–311 (1983).
56. CA Fairhall, "The National Nuclear Laboratory and collaborative University research in the UK." *Scientific Basis for Nucl. Waste Management XXXII, MRS Symp. Proc.* **1124**, 65–76 (2009).
57. I Costa, R Baciocchi, A Polettini, R Pomi, CD Hills, PJ Carey, "Current status and perspectives of accelerated carbonation processes on municipal waste combustion residues". *Environ. Monit. Assess.* **135,** 55–75 (2007).
58. CH Fritsen, V Lytle, SF Ackley, CW Sullivant, "Autumn Bloom of Antarctic Pack-ice Algae", *Science* **266**, 782-784, 1994.
59. E W Wolff, H Fischer, F Fundel, U Ruth, B Twarloh, GC Littot, R Mulvaney, R Röthlisberger, M de Angelis, CF Boutron, M Hansson, U Jonsell, MA Hutterli, F Lambert, P Kaufmann, B Stauffer, TF Stocker, JP Steffensen, M

Bigler, ML Siggaard-Andersen, R Udisti, S Becagli, E Castellano, M Severi, D Wagenbach, C Barbante, P Gabrielli and V Gaspari, "Southern Ocean sea-ice extent, productivity and iron flux over the past eight glacial cycles", *Nature* **440**, 491-496, 2006.

60. BW Selleck, PF Carr and BG Jones "A Review and Synthesis of Glendonites (Pseudomorphs after Ikaite) with New Data: Assessing Applicability as Recorders of Ancient Coldwater Conditions". *J. Sediment. Res.* **77**, 980-991, 2007.
61. "The Top 20 Pharmaceutical Companies" *http://www.allaboutmedicalsales.com/dir/top20_pharma_companies.html*
62. WIF David, K Shankland, J van de Streek, E Pidcock, WDS Motherwell, JC Cole. "DASH: a program for crystal structure determination from powder diffraction data." *J. Appl. Cryst.* **39**, 910–915, 2006.
63. "DASH - Crystal structure solution from powder diffraction data.", available from CCDC, *http://www.ccdc.cam.ac.uk/products/powder_diffraction/dash/*
64. *Defence Technology Strategy*, Section B8 (General Munitions and Energetics Technologies), published by MoD, October 2006.

## 3. Beamline requirements

The community requires a versatile powder diffraction beamline designed to allow the wide range of experiments described in the science case with transmission geometry and both very short and extremely long measurement timescales (ms – months). The key requirement is for a broad range of energies between 8-40 keV, this can be achieved with X-rays sourced from a bending magnet (BM). Standard optical elements will give stable focused monochromatic X-ray beam, continuously tuneable between the specified energy ranges for the end stations, with white beam and gas bremsstrahlung from the storage ring being confined to the optics hutch. High flux concentrated at the sample can be achieved with the latest developed focusing optics and, when combined with integration of the data from area detectors, maximum intensity and rapid data collection will be easily achievable. The conceptual design of the beamline is illustrated in the Appendix.

This facility will require an optics hutch with a standard EXAFS-quality monochromator using pairs of Si (111) and (220) crystals to adequately filter the incident beam. Mirrors will be used for harmonic rejection and for focusing both vertically and horizontally to increase the flux density at sample. Additional space is designated for a high-bandpass Laue-Laue monochromator, and for compound refractive lens assemblies for focusing. Thus the configuration of a number of intensity-wavelength combinations will be possible. New optical elements including compound refractive lenses, kinoform lenses, and small focusing mirrors are available or are being developed at DLS. For example, CRL's prepared in diamond substrates [1] can focus beam down to sub-micron focal spots, and automated systems for changing focal length have been developed at ESRF [2]. Commercial beryllium CRLs have been used successfully on both undulator (I11) and bending magnet (B16) beamlines at DLS. Here, foci are in the region of 20x10 μm.

Two separate experimental hutches will be required and each will be equipped with a large area detector and a range of sample environments and robotic sample changers. These (EXP-H1 and EXP-H2) will operate at "low" and "high" energies for short (ms-hours) and long duration (hours – months) experiments, respectively. The normal operational beam size at the sample should be 200 $\mu m^2$. In the first hutch, the detector would be easily set at either short or long distances (200 – 1000 mm) from the sample, either in line or off-axis to the X-ray beam so that large Q-ranges (0.04 - 30 $Å^{-1}$) can be accessed. It will be equipped with a variety of sample environments (e.g. cryostream, cryostats and furnaces) to cover a large range of non-ambient conditions. In the second hutch, in addition to facilitating long term studies, the combination of X-ray diffraction with other on-line techniques (e.g. Raman, IR spectroscopy and laser illumination) can be explored. Maximum flexibility and ease of configuration is to be provided, with highly automated experimental systems to allow set up and switching of beam between the two end stations. The key requirements are the unique arrangements of the two end stations and area detectors described below. An important use of this beamline will be for developing novel experiments.

To meet the scientific aims of this beamline, fast larger area detectors built using pixellated photon counting technology will be required. These allow rapid data collection with textural information at high signal-to-noise by whole pattern integration. The high quality data will be used for detailed structural analysis (Rietveld and LeBail refinement methods). For Day 1, we will procure the latest commercially available detector technology, matched with the energy range and resolution requirements of each end station. Readout time, frame rate, dynamic range and physical size will be constraining criteria. Near-horizon technology, and Diamond involvement with development projects (e.g. Medipix), will ensure an upgrade path to maintain competitive performance. With

small contributions from the beamline optics, high quality PD data for structure refinement will require detectors with an intrinsic resolution of $\Delta d / d \sim 10^{-3}$-$10^{-4}$. This will be achieved with off the shelf detectors such as Si pixel and flat-panel devices To achieve fast and time resolved measurements, detectors must offer high intrinsic frame rates at full pixel binning, with the option for higher burst-mode rates. This is achieved by increasing pixel binning and/or bit depth (ADC resolution) and entails a trade off in crystallographic resolution and/or dynamic range respectively. Frame rates of between 10 and 500 frames $s^{-1}$ are achievable using currently available technologies.

Rapid area detectors will result in large volumes of data. High speed processing will be necessary to prevent data overwriting or loss. A suite for visualisation of these data is required, and software for area detector data processing during and after collection will be provided to ensure the best outcomes of experiments. Thus in addition to standard computing and display facilities, networking to beamline instrumentation and site data storage is necessary.

A decent size control room at the end of the beamline is required to accommodate users and beamline staff during operation. Although users will require a support laboratory for chemistry and materials science facilities for off-line sample preparation, we envisage a cooperative arrangement with the existing I11 (ultra-high resolution powder diffraction) support laboratory and workshop facilities. Significant gain will therefore achieved with little extra cost being incurred. We expect that further cooperation will develop with I12 and I15 communities (Materials Science, Extreme Conditions) and I19 (Single Crystal Diffraction).

[1] L Alianelli, K Sawhney, A Malik, O Fox, P May, R Stevens, I Loader, M Wilson (2010) A planar refractive x-ray lens made of nanocrystalline diamond. J Appl. Physics, 108. DOI: 10.1063/1.3517060

[2] GBM Vaughan, JP Wright, A Bytchkov, M Rossat, H Gelyzolle, I Snigereva and A Snigirev (2011) X-ray transfocators: focusing devices based on compound refractive lenses. J. Synchrotron Radiation, 18. DOI: 10.1107/S0909049510044365

# 4. Beamline Specifications

## 4.1 Beamline X-ray Optics

We propose X-ray optics which utilise both the BM spectrum and beam divergence, the latter is 0.15 mrad vertically and 3 mrad horizontally. We will place the double-crystal monochromator (DCM) first, then focus the diverging monochromatic beam with a meridionally-bent double toroid mirror (horizontal and vertical focusing) and a plane mirror to return the beam parallel to the horizontal plane providing a fixed height beam to experimental hutches. Our design has similarities to those of B16 (DLS) and XMAS (ESRF BM28).

| | |
|---|---|
| **Source:** | Bending magnet |
| **Energy range:** | 8 – 40 keV (tuneable) |
| **Optics:** | (a) Si(111) double crystals (8 – 30 keV)<br>(b) Si(220) double crystals, Laue-Laue crystals (25– 40 keV)<br>(c) Harmonic rejection, focusing mirrors and refractive lenses<br>Energy resolution: (a) $\Delta E/E \sim 10^{-4}$ and (b) $\Delta E/E \sim 10^{-3}$ |
| **Beam size:** | 1st Exp Hutch: 200 μm$^2$<br>2nd Exp Hutch: 200, 50 and 5 μm$^2$ (with various focusing optics) |
| **Photon flux:** | $\sim 10^{12}$ phs/s/0.1% at 15 keV and $\sim 10^{11}$ phs/s/0.1% at 30 keV. |
| **Detectors:** | Pixel area detectors (x2): active area of 420x420 mm$^2$ or bigger,<br>pixel size = 200 μm or less, speed = minutes – ms per frame (pattern) |

### 4.1.1 Optics Hutch (OH)

The first element will be the DCM in Bragg-Bragg geometry (20 m from BM). Here, two crystal sets will be mounted side by side, with Si 111 and Si 220 for lower (8-30 keV) and higher energy (25-40 keV) ranges, respectively. The intrinsic energy resolution ($\Delta E/E$) of Si 111 is ~ 1.5 x $10^{-4}$, while 220 crystals give a resolution of ~ 0.6 x $10^{-4}$. The first crystal will take white beam, requiring direct water cooling. Similar DCM's are in routine operation on BM beamlines at DLS; the same design used from Day 1 will present no risk.

The second element, at 24 m from BM, will be a toroidal mirror meridionally bent to focus vertically and horizontaly. The toroid will be coated with Pt for harmonic rejection. At an angle of 2.5 mrad, a 1.2 m mirror will take a beam height of 3 mm, collecting most of the vertical diverging beam. Changing meridional bend and incidence angle allows focusing in either experimental hutch. A focused beam size of ~200 µm (FWHM) is our target for Day 1 for each experimental hutch. For high resolution experiments, the beam collected will be limited to <0.5 mrad (11.75 mm horizontal) to prevent loss of resolution from diffraction at the area detector. The third element at 26 m is a sideways translation plane mirror, with Rh or Pt stripes for harmonic rejection. This mirror will be angled to make the output beam to EXP-H1 or EXP-H2 horizontal.

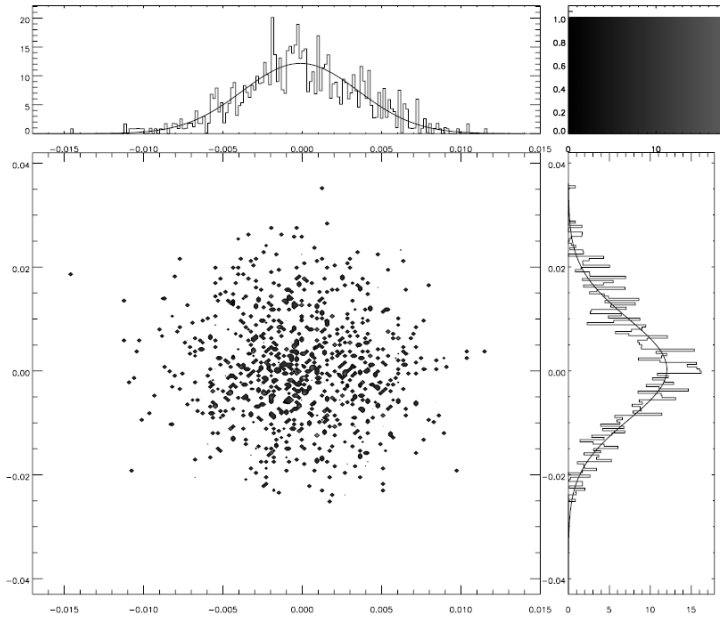

Fig. 1. Ray tracing for DCM/Toroid/Plane Mirror assembly with rms slope errors; meridional=0.31 arcsec and sagittal=2.07 arcsec (focus = 47m from BM, meridional bend = 12.8 km, toroid radius = 43.2 mm, and mirror angle of 1.84 mrad). The acceptance angle is 0.5 mrad (h) and 0.15 mrad (v). Scale on axes (cm) showing FWHM of ~230 µm (v) and ~ 86 µm (h) to the focused beam.

With this optics train the monochromator can be used alone, or in combination with mirrors for harmonic rejection and focusing. Ray tracing (SHADOW) of the above optical scheme is shown in Fig. 1. Other arrangements are possible, e.g. mirror(plane)-DCM-mirror(cylindrical) and DCM-mirror(plane)-mirror(cylindrical), the final choice would be decided after detailed ray tracing analysis at the Technical Design Review stage if this beamline is approved.

Our design allows space for a Laue-Laue DCM (21.5 m) and compound refractive lenses (CRL's) for fine focusing (< 20 μm). For Day 2, we will install a double-bounce bent-crystal Laue-Laue monochromator, with a bandpass of $\Delta E/E \sim 10^{-3}$. This will provide increased flux between 30 and 40 keV. DLS already has experience in designing and building such devices. We will also reserve 0.5 m in OH for a CRL assembly designed to focus over ~ 15-20 m, available from Day 2. At 40 keV, fewer than 10 individual lenses are required.

## 4.2 Experimental hutches

### 4.2.1 Hutch 1 (EXP-H1) - Low energy end station (8-25 keV) – centred 40 m from source.

EXP-H1 will accommodate fast turn-around experiments (~1-3 days) on removable, light-weight breadboards on which sample stages/cells can be mounted. A robotic arm for high-throughput/rapid-access for academic and industrial work will also be used to pick up and hold in place small cells with positional repeatability (< 60 μm). It will be equipped with non-ambient sample environments: cryostream (80-500 K), cryostats (4 – 295 K), hotair blower (RT – 1273 K), DSC, humidity chamber and furnaces (295 – 2000 K), high pressure gas ($H_2$, $CO_2$, $CH_4$ and others) supply system. A motorised rail system, side mounted on the optical table, will translate the detector along and across the beam. To transfer beam to EXP-H2, the optical table will translate to one side and a transfer pipe section will be held in place via a robotic actuator. For high energy beams, small air gaps will not adversely affect beam intensity and Kapton-type windows will be sufficient.

### 4.2.2 Hutch 2 (EXP-H2) - High energy end station (20-40 keV) – centred 47 m from source

EXP-H2 will house longer duration experiments (LDE) with sets of sample cells and combined with other techniques, e.g. laser or Raman spectroscopy, slow and/or fast reactions and micro-focussing for small or differentiated samples requiring high spatial resolution measurements. These experiments will be mounted on a large optical table equipped with adjustable linear drives, to automatically and periodically move sample cells in and out of the beam. The area detector will be driven by a motorised stage and one position on the optical table used for "interactive" experiments. Sample environments such as cryogenic systems, incubators, heating stages, climate simulators and high pressure gas cells will be accommodated for user operation.

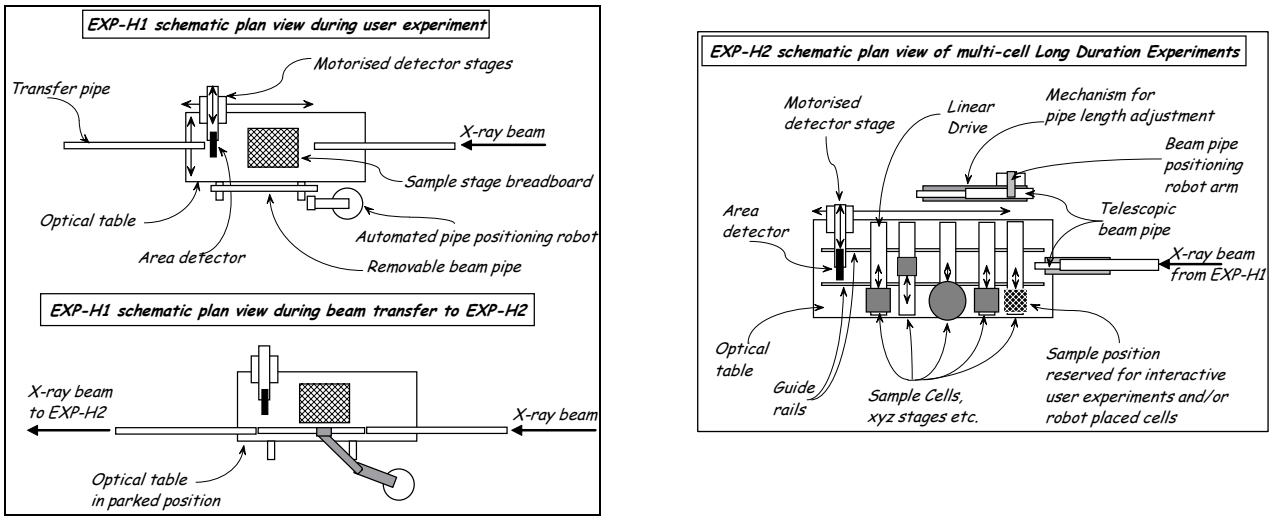


Fig. 2. Experimental arrangements: EXP-H1 (left) and EXP-H2 (right)

### 4.3 Management of Long Duration Experiments

Multiple unattended LDE may seem challenging for maintaining continuity of services and ensuring personnel safety in the hutch. However, services management will be minimised by standardised connections at the sample cells, with umbilicals to allow cell translation, while racking and easily accessible conduits will be provided for controllers and interface cables. A backup power generator will be installed for supplying cells and controllers. An auto-filling $LN_2$ reservoir and closed-circuit water chiller will ensure continuity of cooling.

The data collection time step in LDE will be less critical and programming will have to account for the user schedule and shutdowns. During user beam, automatic measurements can be scheduled on a regular basis or automatically interleaved at planned intervals during EXP-H1 user experiments (e.g. requiring ~30 min to switch beams, select wavelength, collect data and switch back) and will be interlocked with the EXP-H1/H2 PSS and hutch shutters. Control will be via logistics scheduling software, while a "delay LDE" command issued at the start of data collection for non-ambient or time sensitive EXP-H1 measurements would prevent interference between the two systems.

### 4.4 Beamline Optical Elements for EXP-H2

The field of X-ray optics is developing very quickly at present, with novel micro- and nano-focusing optical systems emerging. Stable monochromatic X-rays will allow novel optics, such as kinoform lenses to be exploited in the experimental hutches close to sample environments for Day 2 operation.

### 4.5 Beamline Detectors for EXP-H1 and EXP-H2

For 8-25 keV Si sensors offer efficient stopping power and the PILATUS area detector is now well established and used at DLS. The pixel size is 172 μm, with a frame rate (6M model) of 12 Hz. However, the newly available XPAD3 chip (130 μm pixels, 500 Hz), or the soon to be available Medipix3 chip (55 μm pixels, up to 1 kHz in burst mode, 100 Hz in continuous mode), may make effective alternatives to PILATUS. For energies of 20-40 keV, scintillators become preferable to Si. Here, compact flat panel devices are replacing the traditional scintillating plate/CCD camera. DLS already has a Thales (Pixium) device with 150 μm pixel size (I12) and a Perkin-Elmer 200 μm pixel device on order (I15). These offer frame rates up to 30 Hz. Resolution of $\Delta d / d \sim 10^{-4}$-$10^{-5}$ will be possible in the future with devices having a pixel size ≤ 100 μm$^2$. Improved resolution at high angle is important as this is where diffraction features increasingly overlap. Future possibilities include direct detection amorphous Se flat panels, which should have a wider dynamic range. ANRAD's detector has a pixel size of 85 μm, but is untried in a synchrotron context. Future high energy detection that would benefit this and other beamlines at DLS (I12, I15 and I11) is a large area detector based on a high Z semiconductor. Recent achievements by the HiZPAD joint research activity on CdTe sensors could prove highly suited, and DESY is currently developing an area detector based on Medipix3 using Ge sensors.

## 5. Budgetary Requirements (Estimated)

| BL Section | Item | Comments | Cost/unit | Qty | Total |
|---|---|---|---|---|---|
| **Front End** | Front End with X-ray shutter | Funded by FE800 | | | |
| | FE XBPM | | | | |
| **Hutches** | Optics & Expt Hutches (EH1 & EH2) | Extra cost for design and construction has been added for contingency. Value to be revised at TDR stage | | | |
| **Cabins & Services** | Design | Funded by ME600 | | | |
| | Construction | Extra cost for design and construction has been added for contingency. Value to be revised at TDR stage | | | |
| | Control cabling (purchase, installation, termination) | Funded by SE800 | | | |
| | Mechanical pipework (supply and installation) | Funded by SE800 | | | |
| **Optic** | Monochromator, double crystals (Day1) | | | | |
| | Monochromator, Laue (Day2) | | | | |
| | Mirrors, X-rays | | | | |
| | CRL (Day 2) | | | | |
| **Slits** | Slits, white beam | | | | |
| | Slits, general purpose in-vacuum | | | | |
| **Absorber & X-ray shutter** | Absorber | | | | |
| | Shutter | | | | |
| **Diagnostic** | Intensity monitor | Use I11 spare Sens-Tec detectors | | | |
| | Fluorescence screen | | | | |
| | Cameras (general viewing and monitors) | | | | |
| | Ion chamber | | | | |
| **Detector System** | Area Detector 1, Pilatus 6M (low energy) | | | | |
| | Area detector 2, PerkinElmer (high energy) | | | | |
| **Computing** | Network | | | | |
| | Electronic racks | | | | |
| | Data Acquisitions (computers, monitors and storage) | Area detectors requirements - see next 4 items | | | |
| | Fast 3D storage | TBC by budget holder for CS800 | | | |
| | Fast data processing (3D-2D) | TBC by budget holder | | | |

| | | | | | |
|---|---|---|---|---|---|
| | | for CS800 | | | |
| | Permanent storage | TBC by budget holder for CS800 | | | |
| | Workstations (super screens - 2D/3D display) | TBC by budget holder for CS800 | | | |
| | Data analysis software (TOPAS, Origin, etc) | | | | |
| **BL Vacuum** | Pumps, gauges, valves and controllers) | | | | |
| **Controls & Motors** | Controls & Motors | TBC by budget holder for CS800 | | | |
| **Safety** | PSS | TBC by budget holder for CS800 | | | |
| | Radiation monitor (portable, installed) | | | | |
| | Oxygen level monitor | | | | |
| **End Station – EH1** | Heavy duty goniometer | | | | |
| | Sample table | | | | |
| | Robotic arm | | | | |
| | Online spectrometers (mass, laser, Raman) | | | | |
| | Standard sample environments (furnace, cryogenic systems, DSC, etc) | Use I11 equipment, no cost | | | |
| **End Station - EH2** | Large sample table | | | | |
| | Robotic arm | | | | |
| | Emergency power generator | | | | |
| | Long duration sample cells and support system | | | | |
| | Online spectrometers | Share with EH1 | | | |
| **Peripheral Lab** | Shared I11 Labs | Cost to refurbishing I11 lab & add specific sample preparation equipment | | | |
| **Furniture** | Desks, chairs & draws, etc | | | | |
| **Miscellaneous** | Tools | | | | |
| | Beamline tubes, stands, windows materials, power standards (Si, $LaB_6$), etc | | | | |
| | | | | | |
| | **Total** | | | | |
| | | | | | |

# 6. Expressions of interest (EOI)

## 6.1 Community

As outlined in the science case, we expect the user community to be drawn from a wide range of disciplines ranging from fundamental/academic research, through to applied science and industrial development. The gap in the current provision of powder diffraction facilities at Diamond were discussed in a workshop held in April 2010 with speakers covering the following areas: high throughput materials synthesis (J Darr, UCL); fast reactions (C Pulham, Edinburgh); *in situ* studies of hydrothermal processing (R Walton, Warwick); thin films (G Hyett, UCL); 2D monolayers (S Clarke, Cambridge); crystal growth (F Meldrum, Leeds); total scattering/PDF (A Hannon, ISIS); batteries and fuel cells (C Grey, Cambridge); Industrial Applications (T Hyde, Johnson Matthey and M Johnson, GSK).

## 6.2 EOI responses

### 6.2.1 Areas of research:

67 written EOIs were received, a number of which were multi-authored, and in broad terms could be broken down according to:

- 38% chemistry; 19% Materials Science; 10% Energy storage materials; 8% Industrial users; 8% Physics; 6% Earth & Environmental Sciences and 11% other (or unstated).

### 6.2.2 Industrial use and application:

There are obviously strong overlaps between subject areas since research regularly migrates from one field to another as it develops from academic problem to applications and products. A significant proportion of EOI were from researchers working on materials systems that would ultimately result in industrial applications. Furthermore, the percentage of EOI from direct industrial users, although strong, under represents total industrial involvement since many academic researchers who sent EOI, particularly in Materials Science, are actively engaged in project work directly funded by industrial partnerships, including defence organisations.

### 6.2.3 Technical requirements

EOI authors were asked to highlight/comment, where applicable, on the specific technical aspects of the beamline that would be of direct benefit to both their own research programmes and/or to their fields of enquiry in general.

Beyond the baseline requirement of providing SXRPD facilities suitable for structural analyses, the following aspects were considered by correspondents as important to their current work, or of direct benefit to the planning and execution of future work:

- **Non-ambient environments**: 68% of EOI identified the provision for providing facilities for performing diffraction measurements on samples in non-ambient or hostile environments. Although the majority discussed high and low variable temperature experiments, a number were concerned with other non-ambient environments including gas-solid interactions and mechanical deformation. There has recently been a noted increase in the number of user applications involving gas-solid cells on I11, which due to space constraints on the I11 instrument, restrict either the size of cell, or the data range. Space restrictions would be less stringent on the large area detector beamline.

- **Time resolved, fast measurement, kinetic studies:** 61 % of the EOI stated that short data collection times and time resolved measurements were central to their work. Such applications obviously overlap with the need for non-ambient environments, but fast data collection times are also essential for materials (particularly with high organic content) that are beam sensitive
- **Fast measurement, experiment turnaround, high throughput and rapid access:** Overlapping with a need for fast data collection times was a 48% EOI for fast experiment turnaround times, high throughput facilities and rapid access. All direct industrial users identified these as important, as did a large proportion of academic users. Faster turnaround times will be achieved by the use of extensive automation.
- **High energy, high flux:** 55% of all EOI specifically identified the need for high energy and high flux as important. Again there are obvious interdependencies (e.g. high energy for penetration of non-ambient cells, high flux for time resolved work). However many EOI drew a distinction between high energy diffraction for structure refinement and high energy for total scattering (PDF) studies. Nearly all EOI that identified high energy as important, indicated a requirement for performing both types of measurement. Although each technique has differing data requirements (statistics, angular ranges), hardware requirements are similar and there is clearly a perceived benefit to providing the capability for both on the same beamline.
- **Large scale structure, nano-to-micron domain diffraction**: 39% of the EOI stated a requirement for a diffraction facility capable of probing large-scale structures, nano-to-micron scale diffraction, spatially differentiated materials or single particles. Such studies will make extensive use of the micro focus beams produced on this beamline
- **Anomalous techniques**: 11% expressed an interest in being able to utilise anomalous scattering techniques such as DAFS.
- **Long duration experiments**: 15% of EOI explicitly indicated a use for, or described work than could clearly map onto, the LDE capability. Given its uniqueness and, that by its very nature user turnover will necessarily be lower than normal mode usage, this represents a strong percentage of the existing Diamond SXRPD community. We expect this to increase once the facility is established. Specific examples cited were: long term (years) in situ measurements of the corrosion of nuclear waste forms (cements and glasses) under simulated repository conditions; multiple duty cycle (heating and cooling) studies of the influence of crystallite formation on the durable performance of phase change materials for heat storage technology; long term studies of chemical interaction in metal-organic framework materials for gas storage, chemical separations, molecular sensing and catalysis. Other areas of activity described by various EOI that would profit from this facility include: real time studies of functioning Li-ion batteries; formation and production of pharmaceutical and other compounds and phases by gelation; ocean environment mineralisation; corrosion processes and formation of salt phases in metals.

The following Expressions of Interest were received from the outline proposal

| Name | Affiliations | Research areas and techniques |
|---|---|---|
| Alexander, S | Department of Chemistry, University College London | High throughput characterisation of Fuel cell materials, in situ non-ambient measurements, combined techniques (diffraction with Raman and IR spectroscopy) |
| Almeida Paz, F | Departamento de Quimica, Universidade de Aveiro (Portugal) | Chemical crystallography, design and synthesis of novel functional materials |
| Anderson, P | School of Chemistry, University of Birmingham | nano structured materials based on framework materials, hydrogen storage materials, high throughput, fast turnaround time, in situ fast kinetic studies under non-ambient conditions, nano-micron domain particles, total scattering (PDF) |
| Arnold, D | School of physical sciences, University of Kent | multiferroic and functional oxide materials, high throughput, fast turnaround, total scattering (PDF), high energy diffraction, fast kinetic studies under non-ambient conditions, nano-micron domain particles |
| Attfield, M | School of Chemistry, University of Manchetser | Metal organic frameworks (MOFs), negative thermal expansion, crystal growth of MOFs and Isolation of metastable MOFs, in situ and fast kinetic studies under non-ambient conditions, Diffraction data for Rietveld refinement and total scattering (PDF) studies |
| Attfield, P | Centre for Science at Extreme conditions, School of Chemistry, University of Edinburgh | Partially ordered oxynitride perovskites, high energy Powder diffraction and total scattering (PDF), high and low temperature phase changes, single particle experiments |
| Azough, F | School of Materials, University of Manchetser | Electroceramics, microwave dielectrics, ferroelctrics, multiferroics, thermoelectrics, grain boundary property controlled ceramics, structure-property relationships, high energy powder diffraction, total scattering (PDF), large scale structures, fast in situ kinetic studies under non-ambient conditions, nano-micron domain particles, properties of thin films |
| Brammer, L | Department of Chemistry, University of Sheffield | metal-organic framework materials, in situ solid-gas interactions, fast in situ measurements, combined techniques (diffraction laser spectroscopy), long duration studies of chemical interactions |
| Brandao-Neto, J | Diamond Light Source | Biological materials, fast turn around, high throughput, large scale structures nano-micron domain particles |
| Callear, S | ISIS, STFC | hydrides and metal-organic framework materials for energy storage, fast turnaround experiments, high throughput, high energy powder diffraction and total scattering (PDF) studies, large scale structures, in situ and fast kinetic studies under non-ambient conditions, anomalous scattering methods, study of thin films |
| Catlow, R | Department of Chemistry, University College London | crystallography: catalysts, nanoporous zeolites, structure-function relationships |
| Cernik, R | School of Materials, University of Manchetser | reacting ceramics, thermo electric responses for energy storage, time resolved measurements, high energy, total scattering (PDF) non-ambient environments |

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|---|---|---|
| Chong, S | Department of Chemistry, University of Liverpool | Design and synthesis of porous organic and hybrid metal-organic materials, in situ studies of loaded framework materials, fast data collection to alleviate sample decay and for desolvation studies, structure refinement of weakly scattering organics, high throughput, in situ studies, high energy powder diffraction and total scattering (PDF) |
| Clarke, S | Department of Chemistry, University of Cambridge | structure of molecular adsorbed layers using diffraction, high flux for weakly scattering systems, large scale structures |
| Clarke, S | Department of Chemistry, University of Oxford | Magnetic materials, high temperature iron-based superconductors, thermoelectric materials, battery materials, fast turnaround measurements, in situ studies of battery materials, high energy powder diffraction and total scattering (PDF) studies, in situ fast kinetic studies under non-ambient conditions |
| Davies, P | School of Chemistry, Cardiff University | role of chloride ions in the corrosion of archaeological artefacts, micro focus diffraction from nano-microin domain structures |
| Evans, A | Lennard Jones Laboratory (Astrophysics Group), Keele University | laboratory astrophysics, high throughput, fast turnaround, high energy diffraction and total scattering (PDF), crystal growth, nano-micron domain particles |
| Evans, J | Department of Chemistry, Durham University | Oxychalcogenide materials with novel electronic and magnetic properties, framework inorganic materials, structure-property relationships in amorphous oxides, fast data collection, kinetic studies, studies of highly textured materials, total scattering (PDF) |
| Evans, I | Department of Chemistry, Durham University | structural chemistry of functional materials, oxide ion conductors, fast diffraction data collection for rapid parametric studies of samples under non-ambient conditions, total scattering (PDF) |
| Fern, G | Wolfson Centre for Materials Processing, Brunell University | luminescent materials and electronic materials, organic and polymer processing to improve structural and mechanical properties, using powder diffraction to identify new luminescent phases, anomalous diffraction, fast data collection, combined techniques (diffraction and Raman) |
| Freer, R | School of Materials, University of Manchetser | Electroceramics, microwave dielectrics, ferroelctrics, multiferroics, thermoelectrics, grain boundary property controlled ceramics, structure-property relationships, high energy powder diffraction, total scattering (PDF), large scale structures, fast in situ kinetic studies under non-ambient conditions, nano-micron domain particles, properties of thin films |
| Ganin, A | Department of Chemistry, University of Liverpool | molecular superconductors in the Cs-C60 system, fast turnaround experiments, in situ and fast kinetic studies under non-ambient temperature conditions |
| Grey, C | Chemistry Department, University of Cambridge | Litium-ion batteries & fuel cells, high throughput measurements, total scattering (PDF), fast in situ kinetics, anomalous scattering, nano-micron domain particles, real time studies of Li-ion battery functioning |
| Grovenor, C | Department of Materials, University of Oxford | thin film chalcogenides and pnictides, nano structured steels for future nuclear applications, high energy powder diffraction, nano domains |
| Hannon, A | ISIS, STFC | structure of glasses and disordered materials, the role of lone pair ions, total scattering (PDF) |

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|---|---|---|
| Harris, K | School of Chemistry, Cardiff University | structure determination of organic molecular solids, time resolved studies of structural transformations in organic materials - solid state chemical reactions, desolvation and polymorphic transformations, structural characterisation of order-disorder phase transitions in incommensurate organic inclusion compounds, fast data collection, time resolved studies |
| Hogg, S | Department of Materials, Loughborough University | ultra high strength nano-scale and metastable alloys for aerospace, ultrafine microstructure, intermetallic phases, fast turn around, high throughput, in situ fast kinetics under non ambient conditions, nano-micron domain particles |
| Hutchins, P | Infineumm UK Ltd. | Fast turnaround experiments, high throughput, high energy powder diffraction and total scattering (PDF) studies, large scale structure, in situ and fast kinetic studies under non-ambient conditions, crystal growth, anomalous methods, nano-micron domain particles, properties of thin films |
| Hyatt, NC | Dept of Materials Science & Engineering, University of Sheffield | treatment, disposal & remediation of radioactive wastes, high throughput, low temperature in situ studies, long term in situ studies of nuclear waste forms |
| Hyde, T | Johnson Matthey Technology Centre | in situ & ex situ measurement of industrial catalysts, energy storage materials, characterising amorphous and poorly ordered materials fast turnaround, rapid access, high energy powder diffraction and total scattering (PDF), in situ fast kinetic studies of catalysts under operating conditions, nano-micron domain particles for nano-engineered materials |
| Hyett, G | School of Chemistry, University of Leeds | |
| Johnson, M | GlaxoSmithKline | Pharmaceuticals design and development, phase identification in crystalline compounds and formulations, rapid access, high throughput |
| Jones, M | Department of Chemistry, University of Oxford | Energy materials, fast turn around and high throughput, high energy powder diffraction and total scattering (PDF) studies, in situ and fast kinetic studies under non-ambient conditions, nano-micron domain particles, properties of thin films |
| Jones, T | Department of Chemistry, University of Warwick | Organophotovoltaic materials for solar cells, high flux, focussed beam, weakly scattering poorly ordered structures in thin films |
| Kavanagh, A | AstraZeneca | isolation and characterisation of small-molecule pharmaceuticals, rapid access |
| Kenndy, H | School of Ocean Sciences, Bangor University | mineral precipitation in marine environments, formation of carbonates in sea ice, in situ studies, kinetics, in situ measurement of mineral growth using long duration simulation of oceanic conditions |
| Kirk, C | Department of Chemistry, Loughborough University | Anion exchange materials for radionuclide encapsulation, fast turn around, high throughput, high energy powder diffraction and total scattering (PDF), in situ and fast kinetic studies under non-ambient conditions |
| Lennie, AR | Diamond Light Source | Mineral studies and related inorganic phases, in situ combined diffraction and optical spectroscopy in non-ambient environments for studying kinetics, phase transformations and crystallisation |
| Leoni, M | Trento University (Italy) | nano materials, structure-microstructure relationships, whole powder pattern modelling, large scale structure |

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|---|---|---|
| Lightfoot, P | School of Chemistry, University of St Andrews | Local and long-range structure in perovskite ferroic materials, crystallographic studies of multiferroic and ferroelectric materials, total scattering (PDF) studies |
| Lu, Z | Department of Earth Sciences, Syracuse University (USA) | Calcium carbonate mineralogy and its impact on past climatic and oceanographic conditions, in situ studies under non-ambient conditions |
| Martin, C | CRIMAT, CNRS Caen (France) | metal transition oxides as functional materials, fast turn around, high throughput, high energy powder diffraction and total scattering (PDF), large scale structures, in situ fast kinetic studies under non-ambient conditions, crystal growth and design |
| Parker, J | Diamond Light Source | calcium carbonate mineral system and biomineralisation, in situ studies of phase change structures under non-ambient temperatures and gas atmospheres, micro focus diffraction of biomineralised carbonate structures |
| Parson, S | School of Chemistry, University of Edinburgh | chemistry: total scattering (PDF), hydration phase changes, amorphous materials, pharmaceuticals |
| Portius, P | Department of Chemistry, University of Sheffield | High energy metal based compounds, energy storage systems and fuel cell materials, high energy powder diffraction and total scattering (PDF) studies, in situ and fast kinetic studies under non-ambient conditions |
| Prassides, K | Department of Chemistry, Durham University | Strongly correlated magnetic and superconducting materials, fast turn around, high throughput, high energy powder diffraction and total scattering (PDF) studies, large scale structure determination, in situ and fast kinetic studies under non-ambient conditions |
| Pugh, E | Department of Physics, Cavendish Laboratory, University of Cambridge | structural changes in strongly correlated electron materials, in situ low temperature measurements, nano-domain powder diffraction |
| Pulham, C | School of Chemistry, University of Edinburgh | energetic materials: in situ fast kinetic studies, in situ studies of long duty cycle performance of phase change materials for energy storage |
| Rennie, A | Department of Physics & Astronomy, Upsalla University (Sweden) | Soft matter and colloidal materials, in situ studies of rheology, growth of nano particles and composites |
| Rochford, L | Department of Chemistry, University of Warwick | Organophotovoltaic materials for solar cells, high flux, focussed beam, weakly scattering poorly ordered structures in thin films |
| Rogers, K | Department of Engineering and Applied Science, Cranfield University | thin film polycrystalline photovoltaics, effects of environmental agents on biological systems (bone mineral chemistry), coherent scattering for rapid imaging, high energy diffraction and total scattering (PDF) |
| Sankar, G | Department of Chemistry, University College London | crystallography: catalysts, nanoporous zeolites, structure-function relationships |
| Schoder, M | School of Chemistry, University of Nothingham | development of metal-organic, porous organic and supermolecular framework materials, in situ non-ambient structure determination, structure & phase changes on desolvation of porous hosts, structural and chemical change upon gas and substrate uptake with temperature and gas pressure |

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|---|---|---|
| Serghiou, G | School of Engineering, University of Edinburgh | Materials synthesis using extreme conditions, high spatial resolution structural & chemical micro to nano-analysis and mapping of alloys and ceramics including chemistry from low-Z materials, fast turn around, high throughput, high energy powder diffraction and total scattering (PDF), in situ studies under non-ambient conditions, anomalous scattering, nano-micron domain particles |
| Shankland, K | Reading School of Pharmacy, University of Reading | methodologies for structure determination from powder diffraction data, high throughput, fast turn around, high energy powder diffraction and total scattering (PDF), large scale structures |
| Shotton, E | Industrial Liaison Manager, Diamond Light Source | fast turn around, high throughput, high energy powder diffraction and total scattering (PDF), large scale structures, in situ and fast kinetic studies under non-ambient conditions, crystal growth and design anomalous scattering |
| Skinner, S | Department of Materials, Imperial College of Science, Technology and Medicine | Development of new electrolytes and electrode materials, investigation of their performance under operating conditions, reaction kinetics at high temperature |
| Smith, A | Department of Chemistry, University College London | nucleation and growth of zeolites, fast turnaround, high throughput, high energy powder diffraction and total scattering (PDF) studies, in situ and fast kinetic studies under non-ambient conditions |
| Steed, JW | Department of Chemistry, Durham University | Switchable gels as media for pharmaceutical crystal growth, structure determination |
| Stone, H | Department of Materials Science & Metallurgy, University of Cambridge | high temperature alloys for gas turbine engines, fast in situ high temperature processing measurements at high energies of phase changes and metastable phases combined with mechanical testing |
| Sullivan, P | Department of Chemistry, University of Warwick | Organophotovoltaic materials for solar cells, high flux, focussed beam, weakly scattering poorly ordered structures in thin films |
| Tang, CC | Diamond Light Source | calcium carbonate mineral system and biomineralisation, in situ studies of phase change structures under non-ambient temperatures and gas atmospheres, micro focus diffraction of biomineralised carbonate structures |
| Thompson, SP | Diamond Light Source | Solid state astrophysics & planetary materials: structural-spectral relationships in cosmic silicates, structural evolution in thermally processed amorphous silicates, formation of gas phase carbonates in the early solar nebula, morphological modification of carbonates by biotic molecules, non ambient in situ studies, long duration processing, combined techniques |
| Walton, R | Department of Chemistry, University of Warwick | characterisation of complex structures, porous materials for gas separation & catalysis, fast in situ measurements of crystal growth kinetics |
| Warren, M | Mathematical, Physical and Life Sciences Division, University of Oxford | nano-micron domain structures, anomalous scattering, high energy powder diffraction and total scattering (PDF), in situ measurements of materials processing |

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|---|---|---|
| Wright, P | School of Chemistry, University of St Andrews | Nanoporous solids, synthesis and structural characterisation of zeolite catalysts, metal organic frameworks materials, porous hosts in pigments analysis, fast turnaround experiments, large scale structures, in situ studies under non-ambient conditions |
| Yang, S | School of Chemistry, University of Nottingham | Energy storage and Carbon capture in metal-organic framework materials, in situ studies under non ambient conditions of gas capture and storage, in situ non-ambient studies of gas sorption in poorly diffracting materials, fast turnaround experiments, high throughput, high energy powder diffraction and total scattering (PDF) measurements, in situ fast kinetics under non-ambient conditions |

Appendix 1 – Conceptual Design