

**Science
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Diamond Year 3 Outline Proposal 037

**Proposal for a Year 3 Diamond beamline for
X-ray Diffraction from Surfaces and Interfaces**

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1. Scientific Case

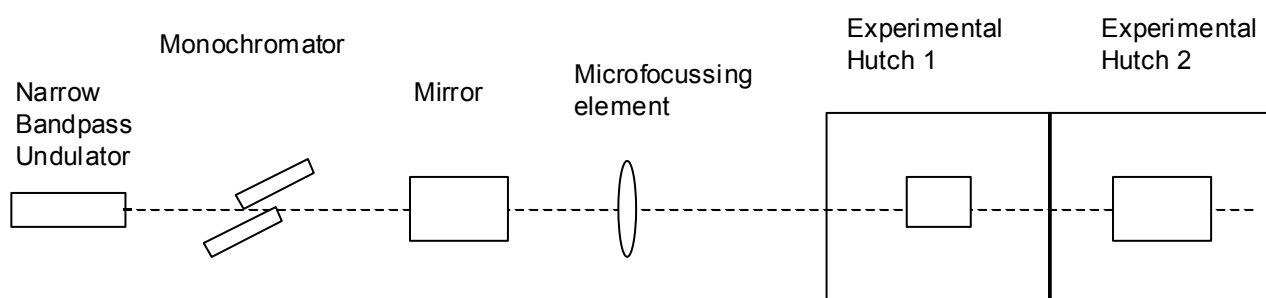
This proposal is a development and resubmission of the case to build a high resolution x-ray diffraction station for the study of surfaces and interfaces under well defined and controllable environments, which was presented for consideration as a year 2 beamline. The proposers are convinced that interface science including the study of solid-liquid interfaces found in many bio-related problems will play a significant role in future research. At present, most surface scattering beamlines around the world focus solely on ultrahigh vacuum (UHV) related research. Here we propose a complementary beamline for surface scattering that specifically allows the study of solid-liquid and solid-solid interfaces at the required higher photon energies. We have incorporated this higher energy range, together with related sample environments, into this revised version of the proposal. In response to comments from the referees and the DIAMOND Scientific Advisory Committee, the beamline layout has been simplified to remove the side branch. The impact of this on the science case has been minimised by retaining a two-hutch design, but with the hutches in-line. This arrangement allows for the separate accommodation of vertical and horizontal scattering diffractometers, optimised for solid and liquid samples respectively. Only one would be used for x-ray diffraction studies at a time, but with only a small impact on the research programme as the rear hutch will be available for sample preparation in an offline mode. The range of samples that will be studied remains extensive and includes; gas-solid interfaces, liquid-solid interfaces, mesoscopic particles, organic and polymer films, semiconductor and metal interfaces and ceramic and complex surfaces.

X-ray diffraction is established as a powerful technique for the determination of structural arrangements of low dimensional materials, such as surfaces and interfaces. It has been used in a wide range of environments including solid surfaces, solid-solid and liquid-solid interfaces. In well-ordered arrangements, X-ray diffraction is able to establish the positions of the atoms to an accuracy of $\pm 0.01\text{\AA}$. The power of the technique is based on two factors: the weak scattering of X-rays by matter and the brightness of modern storage ring insertion devices. The first allows simple data analysis in terms of the Fourier sum of plane waves, whereas the second means that many, often weak, reflections can be recorded giving the high information content necessary to characterise complex structures. In bio-related structures where in-plane crystallinity is less common, reflectivity measurements (both specular and diffuse) are a powerful probe of the out-of-plane layering.

There is a growing trend for the structures that produce unique or unusual properties in low-dimensional systems to be complex or occur in buried interfaces. Diffraction from such samples results in many peaks in reciprocal space or weak features due to absorption by the material that the X-rays must penetrate (e.g. solids or liquids). Most Surface X-ray Diffraction facilities are optimised to an energy range of 10-14keV, which is ideal for the solid-vacuum studies that have dominated this research field for the past decade. The increased penetration and reduced radiation damage of harder x-rays, together with accurate diffractometers would make a surface diffraction beamline on DIAMOND a world leading facility to study more complex structures (by allowing more access to reciprocal space) and buried interfaces. We therefore propose to raise the energy range from the previous proposal to 8-30keV, with the flux optimised between 15-20keV. Such an arrangement would still enable solid-vacuum surfaces to be studied but also give unique access to scattering features from samples with buried interfaces (solid-liquid, solid-solid) that are at present very difficult to study. These would include molecules (including biomolecules) on surfaces, where there is predicted to be a massive demand for structural information to understand such processes as protein denaturation, emulsification and phase separation, binding and unbinding and the control of interfaces. The use of microfocus techniques at DIAMOND to produce a well-defined beam will extend the range of samples that can be studied.

2. Beamline Arrangement

A potential layout for the beamline is illustrated below:



The key aim of this beamline is the provision of high-intensity hard X-rays for diffraction, (anomalous) scattering and other experiments on surfaces of solid materials and at buried interfaces. The samples need to be held under environmental conditions varying from ultra-high vacuum (UHV) to pressures of several bars using a variety of sample chambers. The two different scattering geometries of interest, each with environmental cells that vary substantially in weight, necessitate two different instruments each with their own experiments hutch. Microfocus will be required to illuminate very small samples, while minimizing the scattering from the surrounding substrate. We propose that the beamline will use a small gap in-vacuum undulator to produce a high flux of hard x-rays in the energy range 8-30keV. A range of double crystal monochromators will be incorporated allowing a selectable energy resolution. Even at these energies, the calculated transverse coherence lengths for a source size of $\sigma_{\text{horiz}}=79.9\mu\text{m}$ and $\sigma_{\text{vert}}=7.82\mu\text{m}$ are $20\times 205\mu\text{m}$ respectively for 15keV x-rays and a 40m long beamline; figures comparable with other 3rd generation synchrotron sources.

Outline specification:

Source: Undulator

Optics: double crystal monochromator with interchangeable crystals and focussing mirror.

Energy range: 8-30keV

Resolution $\Delta E/E$: 1×10^{-4} to 1×10^{-2}

Photon Flux: $1\times 10^{14}\text{s}^{-1}$ in 0.1% bandwidth at 10keV

Image Size: $10\mu\text{m}(\text{v})\times 100\mu\text{m}(\text{h})$ with mirror; $2\times 2\mu\text{m}$ with microfocussing

Diffraction: Two in-line hutches operating independently and containing:
1 six-axis, heavy duty diffractometer for mounting large chambers, vertical scattering geometry
1 six-axis, medium duty diffractometer for mounting medium sized cells horizontal scattering geometry
solid state and area detectors

Sample Environment: large scale environmental chambers (inc. UHV)
Medium sized cells, easy interfacing of complex sample environments.

3. User Community

The beamline will support a wide user community encompassing current users of facilities including stations 9.4 and 16.2 at the SRS; ID3, ID10, ID11, ID32 and XMaS (the UK CRG) at ESRF; beamlines W1 and BW1 at HasyLab and the Surface Diffraction Beamline at ANKA.

| Name | Institute | Area | Comments |
|-----------------------------|----------------------------------|---|---|
| Berry | Open Univ | Multilayers | Reflectivity, roughness, interfaces |
| Schreiber | Oxford | Solid surfaces, Organic thin films | time-resolved studies of the growth processes and structural changes in ordered organic thin films |
| Nicklin | Leicester | Solid surfaces/ Nanoparticles | use coherent nature of the beam to study a range of nanoparticle assemblies and dynamics |
| Thornton | Manchester | Solid surfaces/ Nanoparticles | self-assembled metal nanoclusters supported on metal oxide surfaces, which have potential in a number of technologies from visual displays to gas and biosensors |
| McGrath | Liverpool | Solid surfaces/ quasicrystals | open up new opportunities for the study of nanostructures at surfaces and extend the applicability of the diffraction technique to a much wider range of quasicrystal samples. |
| Meenan | Ulster | Bioceramic materials and coatings | X-ray diffraction, environmental chambers inc. biological media |
| Moriarty | Nottingham | Dynamics of nanocrystal formation | Coherence, growth, in-situ cells, microfocus, soft-lithography |
| Howes | Leicester | Solid interfaces | harder x-rays (20keV) used to penetrate thinned samples of well oriented semiconductor interfaces formed by direct bonding |
| Rennie | KCL | Liquid-solid interfaces | adsorption of surfactants, polymers and biopolymers on surfaces using diffuse, off-specular scattering to determine information about the lateral inhomogeneity. Also grafting, etching of a variety of solid interfaces |
| Vlieg | Nijmegen | Crystal growth, solid-liquid interfaces | Coherence, real-time nucleation, 2+3 diffractometer geometry, step dynamics |
| Silfhout + Prince + Derrick | UMIST | Macromolecular thin films, biomaterials | Coherence, microfocus, 'pink' beam option, 2D detector, energy scanning, stepflow growth |
| Lucas | Liverpool | Solid-liquid interfaces | High flux, microfocus, in-situ liquid cell, UHV studies weak scattering, catalysis, electroplating, crystal growth |
| Penfold | RAL | Liquid-solid interfaces | enhance and complement neutron work in the study of ordered structures at interfaces. Systems including ordering from concentrated surfactant mesophases, particularly di-chain cationic surfactant/co-surfactant mixtures. The ability to use smaller sample areas |
| Thomas | Oxford | Liquid-solid interfaces | |
| Zarbaksh | QMW | Liquid-solid interfaces | behaviour of adsorbed alkane films on silicon surfaces in the vicinity of their bulk melting points |
| Clarke | Cambridge | Liquid-solid interfaces | adsorbed monolayers from solution |
| Macdonald + Jones + Roberts | Cardiff + Sheffield + Nottingham | Polymer interfaces, semiconducting polymers, biomolecules | X-ray diffraction, high flux, microfocus, coherence |
| Hamley | Leeds | Polymer nanostructures | specular reflectivity of copolymers on solid substrates and off-specular studies of in-plane ordering |
| Steadman | Leeds | Magnetic thin films | requires the high brilliance of the latest generation synchrotron radiation sources. |
| Binns | Leicester | Magnetic nanoparticles | reconstruct the shape of the magnetic clusters formed in gas-aggregation source |

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