

XAS 2b - The Core X-ray Spectroscopy Beamline for Diamond

Background

Modern X-ray absorption spectroscopy (XAS) can only be performed using synchrotron radiation, and it is an indispensable tool in many areas of science. During the past twenty years, XAS has progressed from being a technique only suitable for specialists in x-ray physics to become a widely applicable tool for people whose principal interests lie elsewhere. XAS is now a viable tool for 'one off' or occasional use. This situation has resulted from the steady development of reliable spectrometers free of 'glitches' and new generations of software for data reduction and data analysis.

However on day one of operations at Diamond, the only station designed for x-ray spectroscopy will be for microfocus studies. There will be no facilities for x-ray spectroscopy of the type currently offered at the SRS. Therefore construction of beamline for non-specialist x-ray spectroscopy is of strategic importance for underpinning the base of UK science and technology and has to be a high priority. Such a station will play an important role in introducing new users to the power and versatility of SR techniques.

Many important samples do not require the intensity of radiation that will be provided by insertion devices on Diamond. Hence it is proposed to build the core beamline on a bending magnet source, (although it is important to note that the performance will be better in most spectral regions than the best current dedicated XAS beamline in the UK). We propose to build a station that will give results of the highest possible reliability and accuracy.

Science

This will be a core beamline that will meet the scientific requirements of the whole community and underpin experiments carried out on the two other XAS beamlines proposed for Diamond. The range of experiments will be arguably as wide as any beamline on Diamond, spanning chemistry, physics, biology, earth and environmental science, and materials science with engineering.

XAS makes a pivotal contribution to the understanding of heterogeneous and homogeneous catalysis. It is of increasing importance in electrocatalysis, corrosion and the development of fuel cells. In the development of new and complex functional materials the ability to study the local structure and electronic state of active components makes XAS particularly valuable. There is increasing interest in the preparation of materials for nanotechnology where the length scales involved fall at the boundary of diffraction and spectroscopy, which makes it important to apply both techniques often under in-situ conditions.

XAS is an important synchrotron technique for obtaining molecular-scale information in Environmental Science. It is crucial because it can provide structural data, unobtainable by other techniques, on the full range of 'environmental' materials such as fluids, crystalline and non-crystalline (amorphous phases & colloids) solids, surfaces and biomaterials. The elements-specific nature of XAS permits study of chemically complex systems, including those in which the element of interest is a minority constituent.

Likewise in the study of glasses and the amorphous state, x-ray spectroscopy gives unparalleled information on the local environment. The technique is applicable at both high temperature and pressure and contributes to our understanding of the properties of liquids and high temperature melts. Studies of crystallisation and order disorder transitions at high-pressure and temperature are of fundamental technological and geological importance.

XAS will continue to make contributions to biology, typically determining metal-ligand bond lengths to a higher accuracy than PX, while probing the (working) solution state, rather than the solid state. Current developments in structural genomics are likely to lead to a significant increase in the number of metalloproteins available for study. The station will be well suited for dilute samples (metalloproteins $\approx 1\text{mM}$) but studies of ultra-dilute samples would take place on XAS_2a.

X-ray spectroscopy is one valuable synchrotron technique of many, and experience shows that there considerable value is added by combining techniques in a single experiment. Therefore provision will be made for wide angle x-ray diffraction studies to incorporated into the beamline architecture. This will be of particular benefit to studies of complex materials to correlate changes in the short and long-range structures.

We propose to develop and apply automation techniques to data collection and analysis of XAS data. This has a dual purpose. The first is to maximise throughput and efficiency on the station for undemanding samples, but the second is more important in that quality of data may be improved by use of algorithms for rapid screening of samples and optimisation of data collection. An objective is provision of a high quality 'remote' service for standard XAS.

Strategy for x-ray spectroscopy at Diamond

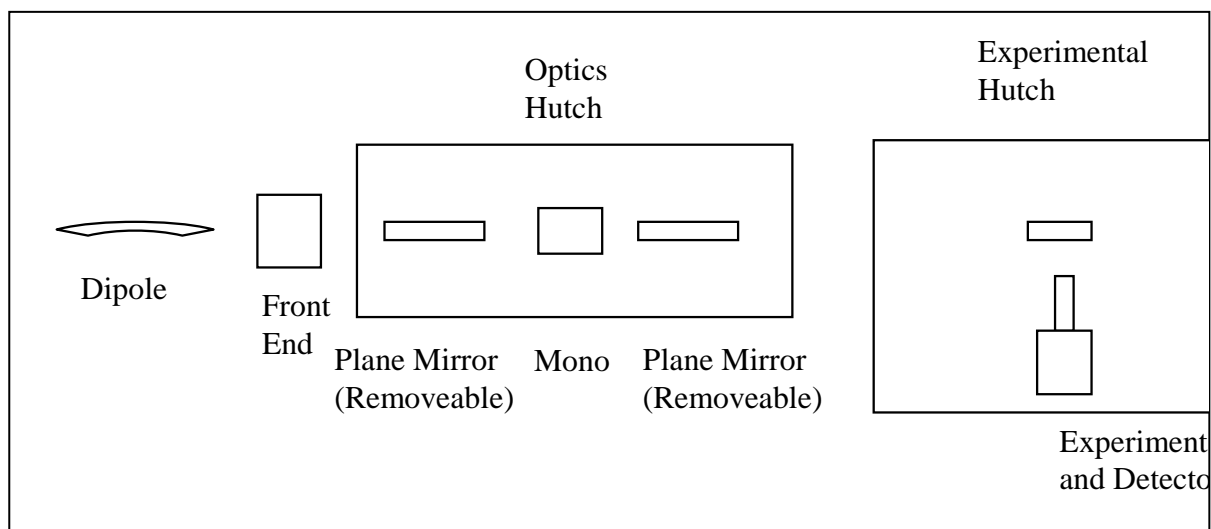
This proposal is an integral part of a coherent strategy for x-ray spectroscopy in the UK. In the medium / high-energy range the vision for the early years is centred upon facilities provided by three beamlines. These are the microfocus (BL-13), ultra-dilute / time resolved (XAS-2a) and this core (benchmark) beamline. The intention is to provide continuity and evolutionary enhancement of current XRS facilities in the UK, coupled with provision of opportunities to develop new types of spectroscopy. We envisage parallel developments in the area of soft x-ray spectroscopy, especially for both microfocus and in-situ studies. In the longer term we wish to see development of ultra high resolution XAS (eg x-ray Raman spectroscopy) on Diamond.

Provisional Beamline Specification

The goal is photon-statistics limited performance. Very careful consideration will be given to all sources of noise – mechanical, electrical, thermal, and optical, based upon many years of experience at the SRS and BM29. In order to make the maximum use of the flux, a plane first optic mirror and sagittal focusing monochromator are specified. The mirror will be set at the same angle as XAS_2a for compatibility, but a third uncoated stripe will allow soft edges down to 2keV to be explored. Beamlines such as BM29 at the ESRF have demonstrated that there is a need for a simple beamline where the K edges of lanthanides give an extended range over data collected on the softer L-edges. Whether this capability should be included is still a matter for discussion, but the beamline will be built with a large amount of flexibility in order that many different types of experiments can be carried out. The station will be designed to be easy to set up for simple experiments and for inexperienced users.

Compatibility – the sample area will share a common sample bench with XAS_2a. Provision will be made for rapid interchange of user-supplied environments for complex experiments (high pressure, in-situ catalysis, fuel cells, corrosion). Many experiments will require simultaneous XRD. The detector currently specified is a gas microstrip, but by the time of start-up silicon microstrip detectors should be available.

Source:	Dipole
Optics:	Removable plane mirror coated with 2 stripes of Pt and Rh and blank Si stripe set at 2 mrad vertically, accepting 3mrad horz. Cooled sagittal double crystal monochromator with interchangeable crystals.
	energy range 2-75 keV;
	resolution $\Delta E/E$ 1×10^{-5} to 1×10^{-3} ;
	photon flux with Si(111) at 10keV:
	mirror in, sagittal focusing $3 \times 10^{12} \text{ s}^{-1}$ (16.5×10^{12})
	mirror in, no sagittal focusing $3 \times 10^{11} \text{ s}^{-1}$ (BM29 10^{11})
	photon flux with Si(311) at 40keV:
	mirror out, sagittal focusing $1.2 \times 10^{10} \text{ s}^{-1}$ (9.3×10^9 , BM29 1.5×10^{10})
	beam size
	focused $\sim 15 \mu\text{m}$ (v) $\times 1500 \mu\text{m}$ (h) FWHM
	unfocused 1mm (v) \times 10mm (h)
Detectors:	Multielement compact solid state detector Curved gas μ -strip detector for X-ray Diffraction
Software:	Flexible environment allowing for easy integration of user equipment, automation of tasks and setting-up, control of multiple techniques and remote access for monitoring.
Sample environments:	Cryostat, furnaces – all fully integrated into the data acquisition system.



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