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An IR Beamline for Infrared Microspectroscopy (IMS) and Combined Micro X-ray Diffraction with IMS (MXD/IMS)

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Scientific Case

There are more than 30 IR beamlines at synchrotron light sources throughout the world based on storage ring bending magnets. The majority of these beamlines are designed for Infrared Microspectroscopy (IMS), in which microscopic dots of material are analysed by infrared spectroscopy, which is a powerful and versatile method of determining chemical structure. An IR microscope based on a Diamond bending magnet will produce a photon flux into a 3-5 micron spot 1000x greater than that from a conventional source and 5x greater than that achievable on the IR 11 beamline for IMS currently under construction at the SRS. Such a facility alone would attract a considerable user community (section 3) covering a wide spectrum of science from biomedicine to archaeology. **However, this phase in the development of Diamond allows us to propose a more radical beamline that would be a world first in having the capability of combining two essential techniques for the structural analysis of complex materials, Xray diffraction and IR spectroscopy, both focused on the same microscopic volume of material.**

A wide range of user groups of the SRS and other synchrotrons are employing both X-ray and IR techniques to study their samples in various environments. With a few exceptions, this has meant repeating experiments at two different laboratories while attempting to duplicate experimental conditions. The proposed combined beamline will promote advanced structural and chemical characterisation of a wide range of commercially and environmentally important materials.

Polymer and liquid crystal morphology and reaction dynamics. In studies of semicrystalline polymer systems, the combination of small angle X-ray scattering (SAXS) and IR spectroscopy on evolving microstructures would make it feasible to follow a process or reaction dynamically and obtain information not only on the backbone framework of a system but also to gain insights into the orientation of chemically and structurally important moieties. The *in-situ* evaluation of the roles of chemical bonding and structural evolution could give useful feedback to the chemical engineering community on process design. In many important polymer systems, such as PET and polyurethane, it will be necessary to be able to record wide angle X-ray scattering (WAXS) as well as SAXS to follow orientation and deformation processes in regions varying from amorphous to crystalline. Dichroic ratios in IR spectra will provide orientational information. The same combination of techniques (SAXS/IR) in time-resolved experiments on smectic liquid crystals (bulk samples as well as in devices) could give information on the switching mechanisms. In particular, the IR analysis would allow the possibility of looking at how different parts of the molecule switch at different rates.

High pressure studies across a wide range of materials problems will benefit from the powerful microscopic combination of XRD and IR for analysing crystalline microstructures. Examples include high pressure phases of Lawsonite and their role in the geological recycling of water; super-hard carbon materials; pressure dependence of drug polymorphism; and the simulation of the high pressure behaviour of proteins.

Beamline Specification

The natural opening angle of IR synchrotron radiation is relatively large and this aperture needs to be allowed for in the design of the dipole magnet areas and associated vacuum vessels. The DIAMOND Low Energy Working Party has established that relatively minor modifications in the dipole area to the current design for DIAMOND (changes to the shield wall and positioning a plane mirror in the crotch area) would allow 11 mrad vertical and 35 mrad horizontal collection angles to an IR station, compared to the optimum for IMS of 20 mrad x 20 mrad.

The infrared beamline optics will be based on the SRS IR 11 design using relatively inexpensive spherical focusing mirrors and delivering a matched beam into a commercial infrared microspectrometer. There will be a facility to switch the IR beam between a conventional IR microscope and a microscope modified for simultaneous XRD measurements so that the station can operate purely as an IMS facility so as to optimise the use of both the IR beamline and the adjacent X-ray line.

The IR microscope utilises reflecting optics for the objective and condenser, with the beam being focused onto the sample at a numerical aperture of 0.65, at a distance of 10mm from the front of the objective lens, and being collected at approximately 15mm from the focus by the condenser. These "Schwarzschild" optics each consist of a small convex primary mirror and a larger concave secondary mirror and show very high throughput in the infrared, without chromatic aberration. The paired mirrors however have a "central obscuration", that is a central portion of the incoming beam which is not reflected from the primary mirror to the secondary mirror. It would be possible to introduce a small (2 mm) aperture into the primary mirror, without significant loss of IR throughput. This would then allow a collimated or converging X-ray beam to pass through centre of the IR optics and to be diffracted by the region of the sample at the focus of the synchrotron IR beam. A "drilled" pre-mirror could be used to deflect the collimated IR beam along the axis of the incoming X-ray beam. The collection optics would depend on the geometry of collection of the diffracted X-rays. Small angle diffraction (below 1 degree) could be collected through the centre of the IR condenser in a reverse manner to the objective (Figure 1). However wide angle X-ray diffraction will require either the IR condenser to be interchangeable with the X-ray detector, or the use of beryllium IR optics to allow the X-ray beams to pass through to a wide-angle detector.

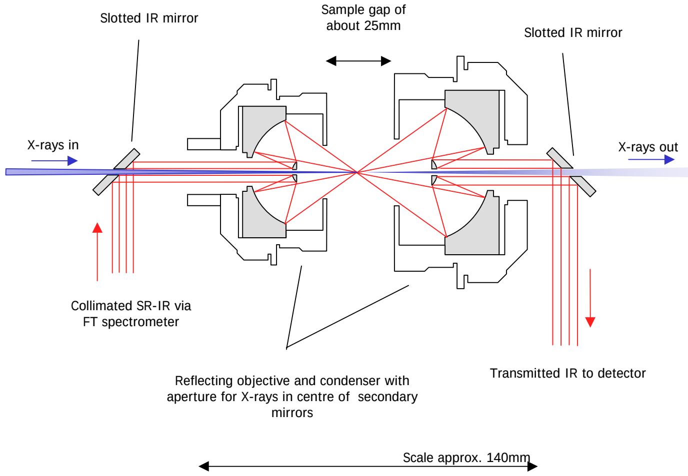

Figure 1 Combined SAXS/IR Optics

X-ray Optics

Low divergence X-rays extracted from the proposed microfocus SAXS beamline would be produced from a tuneable undulator, in the energy range 4keV to 20 keV. The focused beam size attainable would be of the order of 5 microns and below, which is complementary to the microfocus synchrotron IR beam. For optimal use of the combined facility, it is requested that the proposed IR beamline be situated on a bending magnet adjacent to the microfocus SAXS facility.

User Community

The IMS community is large and diverse, and continues to expand. The users listed below exemplify some of the existing applications of IMS, and include a number of groups who will benefit from the combined XRD/IMS facility. The user community is expected to grow even faster once we achieve higher performance levels through the IR 11 station at the SRS and eventually on Diamond. Experience worldwide also points to further growth. Five IMS beamlines are planned at the ALS in Berkeley which will bring the total in the USA to eighteen. Currently there is one station at the SRS (13.3), which is used for both RAIRS and IMS and which is not fully optimised for IMS. Another (IR 11) has been funded specifically for IMS and is under construction.

Current and Previous Users of Station 13.3at the SRS

M. ALMOND, P. HOLLINS, UNIVERSITY OF READING: *Photochemical reactions in the solid state*

S. FISHER, LEEDS GENERAL HOSPITAL, M. CHESTERS, NOTTINGHAM UNIVERSITY:*IR microscopy for post-operative analysis of oral tumours*

I. SYMONDS, DERBY CITY HOSPITAL, M. CHESTERS, NOTTINGHAM UNIVERSITY:*The role of IR spectroscopy in cervical screening.*

C. PULHAM, UNIVERSITY OF EDINBURGH:*High pressure analysis of pharmaceuticals and Structural changes in proteins and small organic molecules under pressure*

R. RAVAL, UNIVERSITY OF LIVERPOOL: *High throughput screening of novel catalysts*

J. SULE-SUSO, KEEL UNIVERSITY, *IR microscopy of radiation damage in mammalian cells.*

I. FARHAT, UNIVERSITY OF NOTTINGHAM: *IR microscopy of biopolymer blends.*

S. BAYLISS, A. SAPELKIN, DE MONTFORT UNIVERSITY*: Studies of advanced materials - fullerenes and nanotubes at high pressure*

R. LEWIS, DARESBURY LABORATORY, *IR microanalysis in the study of breast cancer development.*

H. POLLOCK, A. HAMMICH, L. BOZEC, UNIVERSITY OF LANCASTER: *Advanced photothermal imaging, beyond the diffraction limit.*

J. DWYER, PETER GARDNER, UMIST: *Zeolite development*

G. EECKHAUT, B. DAUNCH, HUNTSMAN POLYURETHANES*: Chemical imaging of industrially important polymer structures*

K. ROGERS, CRANFIELD UNIVERSITY*: Understanding the processes involved in biomineralisation and Bioprosthetic development*

R. WITHNAL, UNIVERSITY OF GREENWICH: Nucleic acid base stacking, studied by synchrotron polarised IR microscopy.

D. SIGEE, UNIVERSITY OF MANCHESTER: *Population diversity and seasonal variation in freshwater plankton*

K. SEDDON. QUEEN'S UNIVERSUTY BELFAST: *Identification of dyes in ancient documents.*

A. MARCELLI, C. ILLIESCU, INFN-LNF, FRASCATI, ITALY: *Heavy metal uptake in mammalian cells*

L. BENNING, N. YEE, University of Leeds: *Bacterial uptake of heavy atoms.*

A. PAWLEY, UNIVERSITY OF MANCHESTER: *Behaviour of the water-bearing mineral Lawsonite at high pressure.*

C. DYER, DSTL, PORTON DOWN*: Rapid identification of airborne bacterial particles.*

L. PYLE, UNIVERSITY OF READING: *IR microscopy of processed food products.*