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Diamond Beamline Proposal 034

An Infrared Beamline for Microspectroscopic Analysis on Diamond

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Acknowledgements

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An Infrared Beamline for Microspectroscopic Analysis on Diamond

1. Summary

It is proposed to build a world class beamline for Infrared Microspectroscopy using a bending magnet source. This facility will offer the highest level of sensitivity attainable at diffraction limited spatial resolution in the mid-IR range, which will support applications of analytical microspectroscopy to disciplines from medicine through biology, food science, chemistry, environmental science, geology, physics, forensic science, and archaeology to fine art. It is also intended that this facility should form a focus for activity aimed at developing non-diffraction limited IR microscopic methods using broad band synchrotron radiation, with the aim of achieving sub-100nm spatial resolution.

IR spectroscopy is a widely used and versatile method for chemical analysis, which produces a spectrum with tens of characteristic absorption bands from a single molecular species. The high information content of such spectra lends itself to the operation of data bases and the “fingerprint” approach to chemical identification. Infrared Microspectroscopy (IMS) produces this chemical identification from a microscopic volume, which is necessary to the understanding of the chemical and physical properties of the vast range of materials that are organised on a microstructural level. The use of synchrotron sources has brought IMS to new levels of sensitivity and spatial resolution, with subsequent impact across a wide range of life and physical sciences.

The first UK synchrotron beamline designed for IMS (11.1) will be commissioned at the SRS in the spring and summer of 2003, although an IMS facility has been operating successfully on the Far-IR beamline (13.3) since 1996. The beamline proposed here will provide a further order of magnitude step up in performance in terms of flux delivered to a 3 μm spot, arising from the smaller source size of the Diamond synchrotron. The design of the optical transfer system will benefit from experience gained at the SRS and in the construction of IR beamlines currently planned at the ESRF and Soleil. The beamline will be designed for optimum performance in the mid-IR, but with a suite of detectors taking advantage of competitive performance in the Near-IR and Far-IR. The IR instruments on the end station will combine the best available technology for both single point spectroscopy and imaging, which currently would involve two alternative instruments. The wide range of disciplines represented in the expressions of interest will call for a versatile suite of sample environments and sampling methods coupled with off-line facilities for sample preparation and results analysis.

The world class performance of the proposed Diamond facility will enable new studies at sub-cellular resolution of phenomena related to disease states and their diagnosis and treatment; the mapping of solid state and catalytic reactions in microcrystals; the characterisation of microscopic patches of nanoscopic thin films, important in lubrication, wear, colloid chemistry and high throughput screening; as well as enhancing many of the current IMS programmes supported by the SRS beamlines. Taken together these activities contribute significantly to health, quality of life and prosperity in the UK.

2 Scientific Case

2.1 Introduction

The potential impact of synchrotron infrared microspectroscopy on the quality of life in the UK and on the UK economy can be visualized in terms of diverse areas of activity ranging from medical diagnosis, food science and forensic science to high throughput screening in materials and catalytic chemistry, geochemistry and archaeology. The ability to acquire high quality IR spectra, in measurement times of seconds, from materials on a length scale of a few microns owes its impact to the high information content of the IR spectrum coupled with wavelength – limited spatial resolution (2 – 10 μm). The synchrotron source achieves this performance by delivering up to 10^3 x more IR light on to a 3 μm dot than does a conventional infrared source. The first demonstration of synchrotron infrared microspectroscopy (IMS), less than 10 years ago [1-3], has led rapidly to a situation where every 3rd generation synchrotron source operates at least one IMS beamline, and many operate several IMS beamlines.

The proposed beamline at the Diamond synchrotron would be the third in a line of synchrotron IMS developments in the UK, which began with the installation of an IR microscope on the Far-IR beamline, 13.3, in 1996. Although not optimized in the mid-IR, this beamline has supported a vigorous user community and has attracted new synchrotron users in a wide range of disciplines, many of which are described below. The success of beamline 13.3 provided the platform to establish a dedicated IMS beamline at the Daresbury laboratory which was funded by linked Research Council Grants to Nottingham University, Reading University and the Daresbury Laboratory. The new beamline 11.1 is due to be commissioned in the Spring/Summer of 2003. The proposed Diamond IMS beamline will offer further improvements in performance arising from the smaller source size of the Diamond synchrotron compared to the SRS at Daresbury. The advances in flux delivered to a 3 μm spot are summarized in table 1.

Promises of "word-beating" performance may not, in this instance, be directly related to the IR flux delivered at a microdot by this particular beamline design. This flux will be comparable to that achieved by leading competitors. The signal-to-noise ratio in synchrotron IMS spectra is commonly limited by noise related to beam instability, which is addressed by positional feed-back systems which are constantly being refined. Given that best practice on achieving beam stability will be incorporated into this beamline design we can expect enhancements in S/N ratio exceeding those predicted by the flux advantage. We are fortunate to be in a position to benefit from the experience of colleagues currently planning and/or designing IMS beamlines elsewhere (e.g. ESRF, Soleil).

We therefore confidently aim to provide a world-class beamline with a first rate working environment, appropriate for the very wide range of disciplines which it will serve.

	ν (cm^{-1})	**Image dimensions at microscope upper aperture (mm)**	**Number of rays passing a 100 x 100 µm upper aperture**
Beamline 13.3	650	2.34 x 1.11	232
	4000	1.91 x 0.73	87
Beamline 11.1	650	1.22 x 0.44	465
	4000	1.25 x 0.46	727
Diamond Beamline	650	0.13 x 0.30	3543
	4000	0.14 x 0.27	4304

Table 1: Results of a SHADOW simulation comparing image properties at the microscope upper aperture. These conditions relate to a spot size at the sample stage of 3-4 µm.

We have paid particular attention to the facilities supporting the beamline, which will include:

- sample environments covering high pressure (to 90 GPa), high and low temperature (800 K to 4 K), vacuum, controlled humidity, gas and liquid flow cells

- test-bed instrumentation using parts from instruments inherited from SRS 13.3 and 11.1 for experiments requiring unconventional illumination or detection arrangements, *eg* near-field experiments

- off-line remote instrument operation to facilitate decision making and adjustments to experiments from the home laboratory

- remote consultation facilities such as software and hardware for rapid display of images and spectra at remote sites, which will be important for medical applications which often require frequent consultation with pathologists and other medical specialists

- associated sample preparation and biological containment preparation rooms equipped with stereo and phase contrast microscopes, a Class II biosafety cabinet, incubator and inverted microscope.

2.2 Scientific Impact

2.2.1 Medical Applications

Cancer Screening and Diagnosis

A number of UK teams are contributing to world-wide efforts [4-7] to bring the analytical power of IR spectroscopy to bear on the screening and early diagnosis of cancer. A key role of synchrotron IMS in this field is to produce the highest quality IR spectra possible at cellular and sub-cellular level [8] (20 µm – 3 µm) with the aim of producing a gold standard set of training spectra as the basis of statistical protocols which will identify early abnormality in cells. The proposed beamline will be the only UK facility supporting this work. The ability to record such high S/N ratio spectra from within cells will support studies of the origins if the biochemical changes associated both with abnormality and with the results of intervention with drugs.

Cervical cancer diagnosis is an example of current work, based at the SRS, Daresbury, funded by the EPSRC Physics for Healthcare programme [9,10]. The incidence of cervical cancer in the UK remains among the highest in western Europe with false negative rates for cervical screening commonly reported at 10-20%. Current cervical screening programmes rely on cytological analysis of exfoliated cells, which is both labour intensive and subjective. Furthermore the infrastructure to collect samples and train staff does not exist in many developing countries where cervical cancer is now the commonest cause of death from malignancy in women. Screening methods based on IR spectroscopy offer potential advantages over screening by conventional cytology, which include faster and less subjective analysis, reduced cost and the potential for automation. The National Coordinating Centre for Health Technology Assessment (Department of Health) is currently considering providing funding for research programmes specifically in the area of infrared spectroscopy in screening for cervical pre-malignancy.

A team at the University of Leeds and the Leeds General Infirmary is attacking the problem of oral cancer (head and neck cancer). 3,500 new patients are diagnosed with head & neck cancer annually in the UK, approximately half of these will die of their disease and the majority will undergo treatment that results in disability and/or disfigurement and which represents a significant cost in terms of manpower and NHS resource. There is evidence of an increase in numbers diagnosed and of greater numbers of patients presenting at a younger age and without identifiable risk factors. While there are possibilities for screening, their emphasis is on the understanding of the cancer and the host/disease relationship. Here sub-cellular spectroscopy is crucial, *eg* in following changes resulting from therapeutic intervention using both material derived from patients and commercially available cell lines.

In a similar vein, a team at UMIST/Christie Hospital Manchester working on IR based early diagnosis of prostate cancer is studying prostate cancer cell lines.

Other biomedical areas of study which will benefit from the high performance of the Diamond IMS beamline involve structure property relationships in biomedical materials such as crystalline deposits in the aorta [11], bone [12] and breast collagen [13].

2.2.2 Geochemical and Environmental Applications

The performance of the Diamond IMS beamline at sub-5 µm spatial resolution will facilitate further developments in a number of areas of geochemical and environmental research. High pressure studies underpin much of geochemistry research, where the synchrotron source is ideally suited to matching the geometrical requirements of the cell and focusing on microcrystals with high flux [14]. Synchrotron IR microspectroscopy is essential for these experiments as the high flux will allow different areas within the diamond anvil cell to be probed. This means that different crystals may be examined separately (a likely crystal size is up to 10 by 10 µm) and a background spectrum may be collected of sample-free pressure medium (typically KBr) at the same P-T conditions as of the sample. Examples include the pressure/temperature dependence of the stability of hydrated and carbonated materials, the behaviour of which in the lower crust and the earth's mantle is believed to influence earthquake and volcanic activity.

In the environmental field, *in situ* and *in vivo* biomineralization studies carried out in the last few years at the SRS IR stations 13.3 [15] have provided an insight into exciting new research possibilities in geobiology. In addition, the increased potential of the proposed Diamond beamline has been demonstrated though a series of experiments undertaken at the infrared beamline at the Advanced Light Source, Berkley Laboratory. Novel IR methods and algorithms for the analyses of IR spectra have been developed and they were applied to determine biomineralization mechanisms and kinetic rates using the changes in the IR spectral features on cyanobacterial cells [15,16]. Future work, more demanding on instrument performance, will be directed to the quantification of the mechanisms and kinetic rates of *in situ* and real-time changes in the biogeochemical environments around microbial cells and their effects on the organic functional groups on cell surfaces.

2.2.3 Solid State Physical Organic Chemistry

Solid-state chemistry of organic crystals has attracted considerable attention in recent years – an interest which has been driven by two principal considerations. The first of these is the possibility of developing clean, solvent-free, syntheses with little deleterious impact on the environment. The second is the phenomenon that has become known as *crystal engineering*. The packing geometry of molecules within the crystal may facilitate reactions, which would not occur in the solution phase, leading to novel product distributions and, in many cases, single-step syntheses of species that would otherwise be difficult to produce.

The measurement of the kinetics of solid state reactions and the development of mechanistic models ultimately depends on the ability to spectroscopically monitor, non destructively, single crystallites in the microgram to picogram range [17,18]. Work on the SRS 13.3 beamline has produced beautiful kinetic data for the photodimerisation of substituted cinnamic acids. Wider application of this approach will need to address the smaller range of crystallite sizes typical for co-crystallised materials, for which the Diamond facility will be well suited. More detailed studies will be possible on larger crystallites, where, for example, in a photolysis experiment, it will be possible to irradiate the crystal at one end and to monitor the rate at which the reaction proceeds at different positions along the crystal: this experiment should permit a more detailed insight into the excitation and propagation mechanisms than has been available hitherto using any experimental technique.

2.2.4 Interfacial Science

IR detection and characterisation of thin (1 –100 μm) molecular films combined with spectroscopic mapping on the scale of a few microns spatial resolution, is beyond the capability of instruments using conventional IR sources. The developments in UK synchrotron IMS facilities, culminating in the Diamond beamline will enable such measurements to be achieved. Applications will include:

- the study of boundary lubricant layers (monolayers and thin films) confined in a 20 μm diameter area between hard solids in contact
- the IR imaging of phase domains in surfactant layers at the gas/water interface
- the measurement of the time dependence of surfactant distribution in high speed liquid jet surfaces
- high throughput screening in materials and catalytic chemistry, where the primary technological challenge is in reading an array using analytical techniques which non destructively characterise dots of material in the nanogram to picogramme range.

2.2.5 Food Science

Applications of synchrotron IMS to problems in Food Science have attracted interest at both the academic and popular levels [19-21]. The processing of food is central to a major component of UK industry. Future studies at the Diamond facility, of processes such as phase separation and fat penetration, will have the capability of operating in real time and measuring *in situ*, rather than having to rely on the *post factum* analysis of a previously prepared sample.

2.2.6 Near-field Infrared Microspectroscopy

There is considerable current interest in overcoming the limits set on spatial resolution by diffraction effects in IR microscopy by applying near–field conditions for either delivering or detecting radiation. The synchrotron source has the considerable advantage of broad- band operation in the IR, compared to more commonly employed laser sources, but achieving the maximum possible brightness is vital for success. The ultimately desired spatial resolution in, for example, biomedical applications is <100 nm, which tends to require an apertureless approach. Exploratory experiments at the SRS 13.3 beamline have illustrated the potential for apertureless measurements using the synchrotron microscope to illuminate a 10 μm diameter spot and using a nanoscopic thermal probe [22]. The prospects for this type of experiment will be transformed by the higher flux obtainable at the Diamond beamline.

2.2.7 Additional Areas of Synchrotron Infrared Research

The following research areas have also exploited the use of synchrotron infrared microscopy at beamline 13.3, SRS. The benefits to be gained from the improved performance expected from the proposed beamline on Diamond will allow significant new advances to be made.

Anisotropy in high-T_C superconductors

The question of the existence of charged walls that separate different antiferromagnetic domains in superconducting cuprates is one of considerable importance, since it has been proposed that the presence of charged stripes in these materials confines the remaining carriers into essentially 1-D structures, leading to abnormal transport and superconducting properties [23]. Improvements in the spatial resolution achievable for these measurements will be possible with the new beamline currently being installed at the SRS, but the ultimate sensitivity will be achieved with the proposed beamline on Diamond. This example also highlights the requirement for a cryo cooling facility on the infrared microscope.

Sorption and Diffusion in Catalysts

Diffusion plays a vital role in heterogeneous catalysis, since efficient catalysis requires rapid penetration of reactants to the internal active sites, and similarly rapid escape of products. Models of such processes in zeolite catalysts need to be compared with experimental measurements made *in situ* on single crystallites of catalyst, which tend to be in the 100 µm size range. Feasability studies at the SRS have established a capability to measure concentrations of reactants and products at precisely defined locations within a zeolite microcrystal as a function of time using synchrotron IMS. The proposed beamline on Diamond will provide the sensitivity and spatial resolution necessary to progress these experiments, which provide the quantitative kinetic parameters for the reaction on the micro scale which is crucial for building a reliable model of the catalytic process

Synchrotron IMS in Archaeology and Fine Art

Infrared and Raman microspectrometry have in recent years been increasingly used as a powerful and versatile method in the scientific investigation of works of art and archaeological finds. The brightness of the synchrotron IR radiation from Diamond will thus be useful not only for the analysis of narrow structures (paint sections, or sections of deteriorated surfaces) but also for an improved signal/noise ratio particularly in reflected light measurements. The availability of a side port reflectance accessory will be important in this field, making possible the non-destructive analysis of large artefacts such as documents and paintings

Spectroscopy of Microcrystals Embedded in Meteorites

IR spectroscopy appears to be highly suited to provide crystal chemical information on the mineralogical constituents of meteorites. In an investigation carried out at the SRS, infrared reflectance spectra have been recorded on polished sections of stony meteorites. It is expected that synchrotron IR microscopy using the proposed beamline on Diamond would allow the examination of crystallites significantly smaller than those examined so far, and would prove extremely useful in the compositional determination of many mineralogical samples in addition to the meteorites described here.

References

1. Carr S.L., Dumas P., Hirschmugl C.and Williams G.P., *Il Nuovo Cimento*, 1998, 20D, 375-395.
2. Reffner J.A., *Cellular and Molecular Biology*, 1998, 44, 1-7.
3. Wetzel, D.L., Reffner, J.A., and Williams, G.P., *Mikrochimica Acta*, S14 (1997) 353-355.
4. Neviliappan S., Kan L.F., Walter T.T.L., Arulkumaran S., Wong P.T.T., *Gynaecologic Oncology* **85** (1): 170-174 (2002).
5. Chiriboga L, Xie P, Yee H, Vigorita V, Zarou D, Zakim D, Diem M, Infrared spectroscopy of human tissue. I. Differentation and maturation of epithelial cells in the human cervix. *Biospectroscopy* (1998); 4: 47-53.
6. Rigas B., Morgello S., Goldman I.S., Wong P.T.T. *Proc. Natl. Acad. Sci. USA* **87**, 8140-8144. (1990).
7. Romeo M.J., Quinn M.A., Burden F.R., McNaughton D., *Biopolymers* **67** (4-5): 362-366 2002
8. Miller L.M., Carlson C.S., Carr G.L., Chance M.R*., Cellular and Molecular Biology* **44** (1) 117-127 (1998).
9. Chesters M.A., Tobin M.J., Griffin N.R., Fisher S.E. *Oral Oncology* **VI**, 193-194 (1999).
10. Tobin M.J., Rutten F., Chesters M.A., Chalmers J., Symonds I., Fisher S., Allibone R., Hitchcock A.. Investigating the potential for infrared microanalysis in cancer screening. *European Clinical Laboratory* **21** (4) 20-22 (2002).
11. Rogers, K. McCollum, C. Lane, D.W. Boucher, E. *Int. Conf. Nucl. Analy. Meth. In Life Sci.* Beijing (1998).
12. Rogers, K.D. Zioupos, P. *J. Mat. Sci. Let.* **18** 651-654 (1999).
13. Lewis, R.A. Rogers, K.D. Hall, C. Towns, E. Slawson, S. Evans, A. Pinder, S. Ellis, I. Boggis, C. Hufton, A. Dance, D. *J. Synch. Rad.* **7** (2000) 348-352.
14. Pawley, A.R. and Allan, D.R., Mineralogical Magazine, 65: 41-58. (2001).
15. Benning L.G., Phoenix V, Yee N, Tobin MJ, Konhauser KO and Mountain BW In: *Geochemistry of the Earth's Surface*, 6: 259-263. (2002).
16. Benning, L.G., Phoenix V. R., Yee N., Tobin M.J. Molecular characterization of cyanobacterial silicification using synchrotron infrared microspectroscopy. *Geochimica Cosmochimica Acta*. (2003). In review.
17. Atkinson S.D.M., Almond M.J., Bowmaker G.A., Drew M.G.B., Feltham E.J., Hollins P., Jenkins S.L. and Wiltshire K.S., *J. Chem. Soc., Perkin Trans.* **2** 1533(2002).
18. Atkinson S.D.M., Almond M.J., Hollins P. and Jenkins S.L., *Spectrochim Acta*, A59629 (2003).
19. Mousia Z, Farhat IA, Pearson M, Chesters MA, Mitchell JR. FTIR microspectroscopy study of composition fluctuations in extruded amylopectin-gelatin blends *Biopolymers* 62 (4): 208-218 (2001).
20. Bouchon P., Hollins P., Pearson M., Pyle D.L., Tobin M.J., *J. Food Sci.,* 66 918 (2001).
21. *Observer Food Magazine*, September 2001.

22. Bozec L., Hammiche A., Tobin M. J., Chalmers J. M., Everall N. J., Pollock H. M. *Meas. Sci. Technol.* **13** 1217-1222 (2002).
23. Bianconi A., Saini N.L., Lanzara A., Missori M., Rossetti T., Oyanagi H., Yamaguchi H., Oka K., Ito T., *Phys. Rev. Lett.* **76** (18): 3412-3415 (1996)

3 Beamline Requirements

This proposal seeks to begin to address the future requirements of the synchrotron infrared microspectroscopy community by installing a state of the art facility on Diamond. Current projections suggest that there will be demand for two modes of operation on such a beamline. Firstly a single point “confocal scanning” microspectrometer (mode 1), such as is installed on the many existing IR beamlines world-wide, would provide the highest spatial resolution for the investigation of small and complex samples. Secondly, a multi-pixel line-scanning FTIR imaging system would be included (mode 2), the first generation of which are only just becoming available. This system would allow the “imaging” of larger area samples at slightly lower spatial resolution. The confocal scanning system will deliver a beam of approximately 1000x the brightness of a conventional infrared source into a diffraction limited spot of 3-6 microns diameter. The design of the beamline optimises performance in the Mid-IR range but will give significantly enhanced performance, compared to a conventional source, in both the Far-IR and Near-IR regions. Commercial FTIR microscope and imaging systems will be installed, which conventionally operate between 600 cm^{-1} and 4,000 cm^{-1}, but this can be readily extended to 100 cm^{-1} to 10,000 cm^{-1} with a number of beamsplitter and detector options. Development of detectors in the defence and commercial sectors is very competitive and is expected to lead to continual improvements in instrument performance. Ultimately it is possible that optimised single point measurement and optimised imaging will be combined in the one instrument; at present this is not the case so it is prudent to plan for a capability to illuminate more than one instrument.

The instruments under consideration are generally fitted with motorized sample stages, autofocus, and motorized apertures. Similarly, steering mirrors in the optical transfer system delivering light to the infrared microscopes will be motorised and under remote control. The ability to control many aspects of the microscope set-up via software, including focus, aperture size and sample selection, will make the beamline amenable to remote access and control. We shall aim to develop operating protocols which allow users to control some of the simpler experiments from their home laboratory. Operation of the infrared systems will be via the dedicated software provided by the manufacturer. Our experience is that users are happy with this approach, and are often experienced in operating the software on arrival at the synchrotron, having used similar systems in their own laboratories.

A wide range of sample environments and sample handling facilities must be provided on the beamline, reflecting the diverse range of the infrared user community. Sample environments required include high pressure (to 90GPa), high and low temperature (800 K to 4 K), and controlled humidity. Gas and liquid flow cells will also be required, both for biological experiments at near ambient conditions and for in situ work on heterogeneous catalysts at moderate pressures up to 1 MPa. Provision will also be made for micro ATR (attenuated total reflection) measurements with the appropriate objectives, and a side port accessory will be included for the examination of large objects.

Off-line facilities for monitoring experiments and data analysis will need to provide computer stations or data points for use with personal lap top computers for up to six people in a comfortable environment. In addition it will be necessary to develop rapid and convenient protocols for presenting images and spectra to collaborators at remote sites. This facility is currently being explored at the SRS, and will be particularly important for allowing quick consultations with expert collaborators, such as pathologists, who are not present on the beamline.

The facilities for sample preparation associated with the endstation must include an area equipped with stereo and phase contrast microscopes, and a separate biological containment room, housing a Class II biosafety cabinet, incubator and inverted microscope.

4. Beamline Specification

4.1 Extraction of IR radiation

A number of options for achieving acceptable extraction angles for IR operation were considered by the Working Party for Low Energy Provision on Diamond. The recommendation was that a modified dipole vessel allowing collection angles of 35 mrad (horizontal) x 30 mrad (vertical) be adopted. An option which does not require modification of the dipole absorber would give a restricted view of 35 mrad (h) x 11 mrad (v). The Beamline 034 Working Group carefully considered these options.

A calculation of the flux delivered at the first focus of light, ie at the exit window from the synchrotron, employing different vertical acceptance angles was carried out by Paul Dumas and is shown in figure 1. The effect of a restricted vertical acceptance angle becomes apparent at longer wavelengths due to diffractive spreading of the beam. It was noted that the collection efficiency of the "35 x 11" option starts to drop below 10 μm (1000 cm-1) and is 2-3x less than optimum at 100 μm (100 cm^{-1}) and an order of magnitude below optimum in the Far-IR at 1000 μm (10 cm^{-1}).

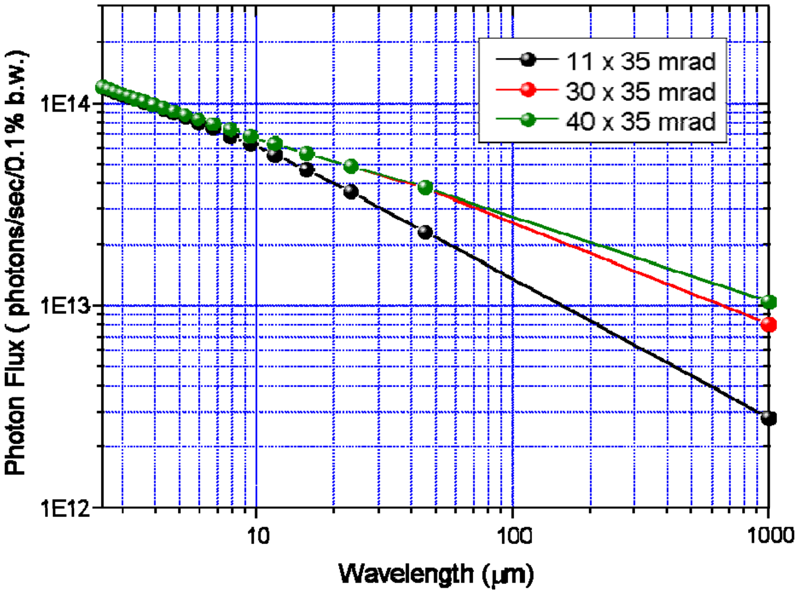

Figure 1 Collection efficiency as a function of vertical acceptance angle

In the context of the current proposal it is clear that the 11 mrad vertical acceptance angle provides acceptable collection efficiency in the range above 20 μm (500 cm^{-1}), but compromises performance on moving further towards the Far-IR. At this early stage it was felt not to be prudent to compromise future applications of Diamond in the Far-IR. Therefore the beamline 034 Working Group strongly endorses the recommendations of the Low Energy Working Group and has incorporated the 35 mrad (h) x 30 mrad (v) specification into this proposal.

Extraction of the beam from the "crotch" region immediately downstream of the dipole vessel avoids any interference with subsequent focussing magnets. Figure 2 shows the proposed extraction optics scheme. The beam is extracted upwards by a plane mirror situated directly after the dipole vessel (M1). This mirror has a central slot to avoid overheating of the element by the intense X-ray and UV beam. The slot will subtend an angle of 2 mrad. Thermal sensors close to the edges of the slot in M1 will control steering of the beam to ensure that the hard radiation passes through the slot.

An ellipsoidal focusing mirror (M2) would be situated 60cm above the height of the electron beam, and would direct the extracted radiation horizontally outwards, perpendicular to the electron beam. The beam would pass through the sidewall of the synchrotron and come to a focus at the UHV exit window.

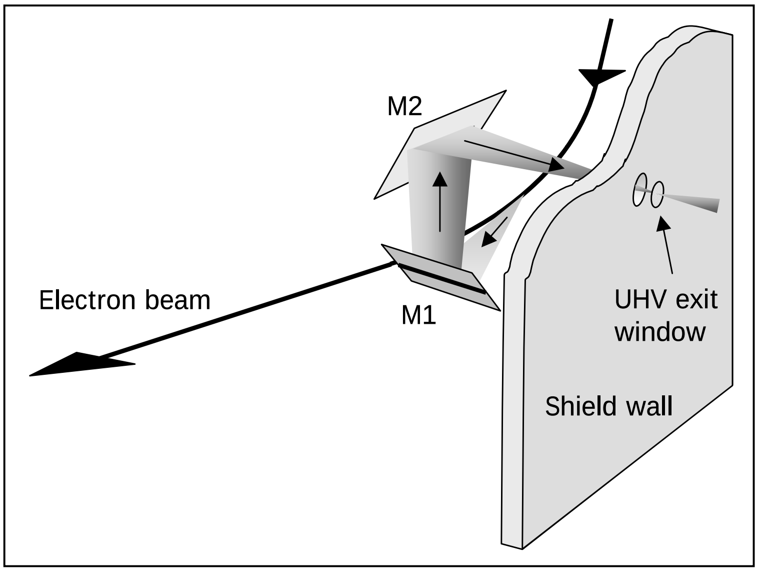

Figure 2. IR Beam Extraction Optics

Downstream of the exit window, a parabolic mirror (P1, figure 3) would be used to collimate the infrared beam, and two plane mirrors would steer the collimated beam. An optical configuration would be employed which allowed the interchange of collimating mirrors of different focal length to match the detector area of the microscope system being used. A similar system is currently being installed on beamline 11 at the SRS. Use of either mirror P1 or P2 directs the beam to alternative instruments as shown, or one of the commercial instruments could be replaced with a "test-bed" instrument for exploratory work in areas such as near-field microscopy

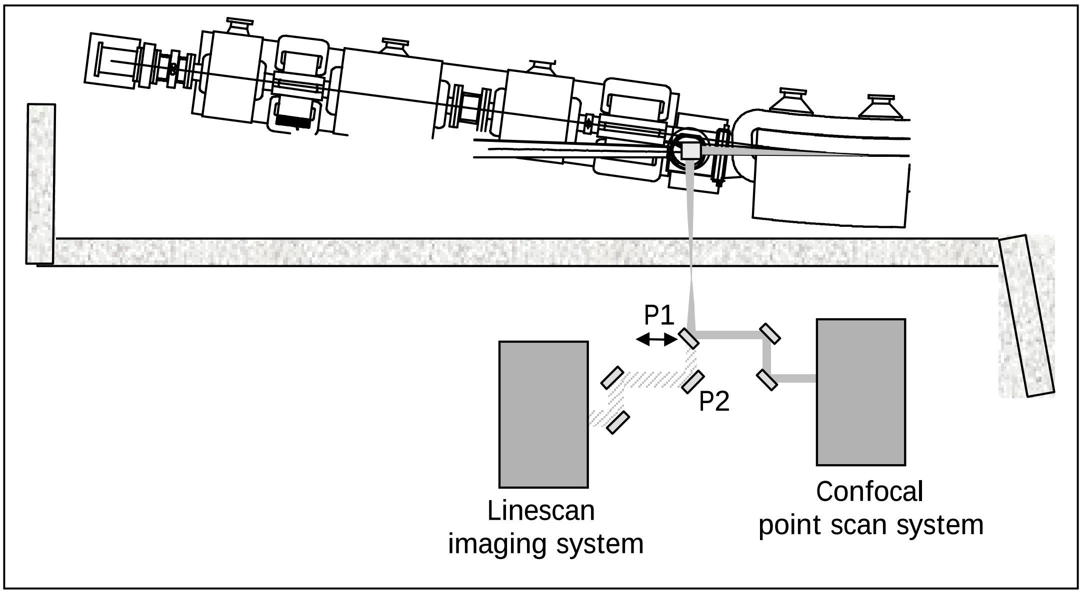

Figure 3. Interchangeable Collimating Optics for Microscope Instruments

In summary, the operational characteristics of the beamline will be

Photon energy:	10,000 cm^{-1} – 100 cm^{-1} 1 μm – 100 μm
Bandpass:	Wide band FTIR instrument resolution 1 – 16 cm^{-1}.
Photon beam at exit window:	5 x 10^{14} (photons/sec/0.1% b.w.) at 1000 cm^{-1}
Photon Beam at sample (10 μm spot)	>1 x 10^{14} (photons/sec/0.1% b.w.) at 1000 cm^{-1} (10^3 x larger than delivered by a conventional source)
Spectrometers:	FTIR spectrometer/microscope FTIR imaging microscope with small array (5x5, or 8x2) detector

4.2 Optics and Instrument Hutch

The steering and collimation optics (from P1 in figure 3), along with the FTIR microscopes can be accommodated within a single hutch, which should be a minimum of 4m x 4m and provide a clean, air conditioned environment. The steering mirrors can accommodate vertical movement of the beam to match a convenient instrument height using optical benches firmly mounted on the laboratory floor.

4.3 Further Beamlines

The Working Party also considered some aspects of the provision of further infrared beamlines on the Diamond synchrotron. Apart from the provision of further modified dipole vessels, the possibility of extracting both bending magnet radiation and edge radiation at one dipole vessel was considered. It was noted that the design of the new ESRF IR beamline utilises edge radiation, which is produced at the point the electrons enter the bending magnet field. It should therefore be possible to design extraction at the dipole vessel to produce two IR beams, separated by about 5^0, which could be collected independently and applied to different endstations.

The Working Party requests that this possibility be considered before the design of the IR dipole vessels is finalised.

5. **Costs**

Estimated costs for the construction of infrared beamline on Diamond

- Wide aperture dipole vessel
- Beamline, including optics and benches
- Endstation equipment
  - Confocal FTIR system
  - Line scanning FTIR "imager"
- Sample environments
  - Diamond anvil pressure cell
  - Flow cells
  - Liquid helium cryostat
  - Humidity cell
- Manpower for design and construction
- Off line equipment
  - Sample prep microscopes
  - Pathologists "discussion" microscope
  - Class II bio. lab. equipment including safety hood, incubator etc.
- Additional spectral analysis software

Beamline Proposal 034:
An Infrared Microspectroscopy Beamline for Diamond

Expressions of interest are invited from research groups in using an Infrared Microspectroscopy beamline on the Diamond Light Source.

Diamond http://www.diamond.ac.uk/ will be built at the Rutherford Appleton Laboratory and is due to be available to users in January 2007. With 24 cells, and at 3.0 GeV, it will be will be unique among the medium energy synchrotron radiation sources and will present great opportunities for fundamental and applied research in both the physical and the life sciences. It will provide very bright radiation from undulators up to 20keV and high flux from multipole wigglers and wavelength shifters to energies greater than 100keV. Bending magnet sources will provide intense radiation over a wide spectral region from 40 keV to the IR.

After consultation with the UK scientific community, the funding agencies and with advice from the SAC, seven beamlines for Year 1 and four beamlines for Year 2 have been identified. The details of these are to be found at http://www.diamond.ac.uk/Activity/ACTIVITY=Beamlines;. We are now proceeding with the preparation of eight further proposals for beamlines to be available for user operation after January 2009.

It is proposed that one of these beamlines should be an Infrared Microscopy beamline.

The outline specification of the beamline is set out below.

The beamline will be designed for two modes of operation

1. Point-by-point IR microspectroscopic analysis at diffraction-limited spatial resolution (3-10 μm).
2. Imaging mode using a small array detector with the synchrotron beam focus tailored to match the detector array geometry.

Mode 1 will provide the highest sensitivity IR spectra from a single point with a spatial resolution of 3 μm, suitable, for example, for analysis of sub-cellular biomaterial.

Mode 2 will be capable of generating large IR spectroscopic maps (millimetre dimensions) maintaining spatial resolution of < 10 μm.

Source:	Bending magnet. Collection angles 35 mrad Horizontal x 11 mrad Vertical
Optics:	Mirror transfer system and microscope delivers microspot to the sample of diameter 10 μm (mode 1), or 30 μm (mode 2). Flux at sample into a 10 μm spot is 10^3 x larger than that delivered by a conventional IR source
Spectrometers:	FTIR spectrometer/microscope

FTIR imaging microscope with small array (5x5) detector
Wavenumber range 600 – 10000 cm^{-1}
Resolution 1 – 8 cm^{-1}

Sample environment: High pressure anvil cell (to 90GPa)
Tissue culture flow cell, (category P2 containment available at station)
Low temperature (to 4K) cell

Expressions of interest, not longer than two sides of A4 should be sent as a word document to diamond@rl.ac.uk by **February 12th 2003**. They should be marked: **PROPOSAL 034** and include:

your name and affiliation;
the overall objectives of your proposed research;
the likely long-term impact on science and technology
and your comments on the outline beamline specifications.

A working group, chaired by Professor Mike Chesters, has been set up to prepare the case for the beamline and propose its primary aims.

Colin Norris
Science Director for Diamond

Name	Affiliation	Area of interest
M J Almond	School of Chemistry U. of Reading	Solid state photochemistry and “crystal engineering”
C D Bain	School of Chemistry U. of Reading	Characterisation of thin films in boundary lubricant layers. Phase domains in surfactant layers
N W Clarke	Christie Hospital Manchester	Diagnosis of prostate cancer and prostate metastases
J Dwyer	Department of Chemistry UMIST	Chemical diffusion in single crystal zeolite catalysts.
I Farhat	School of Biosciences U. of Nottingham	Applications of biopolymers in pharmaceutical and food science
S E Fisher	Leeds General Infirmary & U. of Leeds	Infrared spectroscopy in diagnosis in prognosis of head and neck cancers.
P Gardner	Department of Chemistry UMIST	Infrared spectroscopy of prostate cancer.
A Hammiche	Department of Physics U. of Lancaster	Applications of near field infrared spectroscopy in materials and life sciences
C M B Henderson	Department of Earth Sciences U. of Manchester	Pressure/temperature dependence of the stability of hydrated and carbonated minerals.
I Symonds	Derby City General Hospital and Nottingham University.	The applications of vibrational spectroscopy in cervical screening.
A Pawley	Department of Earth Sciences U. of Manchester	Pressure dependent phase changes in hydrated minerals
H M Pollock	Department of Physics U. of Lancaster	Development of near field nanoprobe-based microspectroscopy.
D L Pyle	School of Food Biosciences U. of Reading	Understanding the interactions between fats and food products. Development of low-fat products.
R Raval	SSRC	Development of chiral

	U. of Liverpool	catalytic surfaces.
S A T Redfern	School of Earth Sciences U. of Cambridge	Pressure/ temperature dependence of the stability of hydrated and carbonated minerals.
K D Rogers	Medicine and Biosciences Cranfield University	Understanding the process of biomineralisation in a wide range of materials, including heart valves, and breast tissue.
L G Benning	School of Earth Sciences University of Leeds	Biomineralisation and heavy metal uptake by “extremophile” bacteria.