

Australian Synchrotron Development Plan Project Submission Form

Section A: Summary and Proponent Details

Project Title

Advanced Infrared Science beamline

Spokesperson

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Executive Summary (approx. 100 words)

The proposed beamline is aimed at providing a beamline to develop brand new capabilities in the infrared spectral region based around a combination of an FTIR microscope system and an alternative dispersive detection system. Whilst the FTIR system will allow for new capabilities such as pump-probe experiments, near field spectroscopy and the installation of custom made apparatus for spectroscopy in extreme environments the novel dispersive system will allow for rapid infrared imaging and time resolved infrared experimentation. The extracted beam will also provide greater flux by combining bending magnet and edge radiation into one intense beam.

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Section B: Detailed Description

B1: Description of Proposed Beamline/Development Project A new spectrometer system for an advanced synchrotron IR beamline

This beamline is intended as a developmental beamline to provide new capabilities and allow experiments that are not currently possible. Both edge radiation and bending radiation will be extracted and combined to optimize the source brightness for delivery to a system with the following capabilities:

- Rapid imaging at high lateral resolution.
- Near field spectroscopy (thermal and scattering AFM probes)
- Dispersive detection with linear and/or 2D array detectors.
- Long working length objectives for custom made experimental apparatus (high pressure cells, high Temp cells, cryostat cells).
- Pump-probe capabilities using a UV source.
- Time resolved IR

The extraction of the infrared beam from the synchrotron bending magnet will follow the same design as the existing beamline with sideways extraction of a large horizontal aperture of edge and bending magnet radiation. However to enable this full fan of radiation to be coupled to a single instrument at a point focus, a "Magic Mirror" as employed at the UVSOR and SPring-8 infrared beamlines will be incorporated into the optics downstream of the synchrotron exit window. (New Infrared and Terahertz Beam Line BL6B at UVSOR. S. Kimura, E. Nakamura, J. Yamazaki, M. Katoh, T. Nishi, H. Okamura, M. Matsunami, L. Chen and T. Nanba. AIP Conference Proceedings 2004, VOL 705, pages 416-419).

Figure 1 outlines a new spectrometer system for synchrotron IR which will be developed in collaboration with Paul Dumas (Soleil synchrotron) and Ulrich Schade (Bessy II synchrotron). The heart of the system will be based around commercial IR microscope optics with a collimated extracted beam directed into a FTIR spectrometer in the standard fashion. This will maintain a user friendly interface and allow the beam to be alternatively directed through the FTIR interferometer for experiments that do not require the dispersive system. Additional plane flip mirrors will be located prior to the Michelson interferometer to bypass the scanner and allow the beam directly through the microscope where for imaging the sample will be raster-scanned in the microscope in a confocal arrangement. The new spectrometer system will be optically coupled to the microscope at the position where the second FTIR detector is normally mounted. The spectrometer system will consist of a dispersive spectrometer with a linear array detector and/or an Echelle spectrometer with a 2D array detector. 2D MCT (64×64, 128×128 and 256×256) photovoltaic arrays have been available for some years whilst linear arrays of typically 256 elements are now becoming available through China and Russia. Australia does have a capacity to construct high sensitivity versions of these detectors at UWA using molecular beam epitaxy (MBE) through Profs. Laurie Faraone and John Dell and we will approach them in the first case. The spectrometer module, consisting of the spectrometer (size about: $300 \times 200 \times 200$ mm³) and the detector with Dewar (size of about: $300 \times 200 \times 100$ mm³) will be mounted mechanically on



Upper focusing mirror Upper aperture (field stop) Cassegrain "objective" - x-y Sample stage Cassegrain "condenser" Lower aperture (field stop) Cassegrain "condenser" Lower aperture (field stop) Lower focusing mirror

top of the microscope housing.

Con-focal microscope Fig 1: Schematic structure of the confocal microscope arrangement with a dispersive spectrometer.

The spectrometer with linear array detector can essentially be used for time-resolved investigations, whereas the Echelle spectrometer can be used for investigations requiring a high S/N ratio and/or for spectral mapping. Here, it will be possible to take about 1000 spectra s⁻¹ with a S/N of about 1000. This would allow for imaging on the fly (no stop for spectral data acquisition) with 10 μ m spatial resolution (step size) with a speed of 0.1 mm²/s. This requires an ability to drive the xy-table with a step frequency greater than 1 kHz and also synchronization of the step sequence and the spectral acquisition. Both tasks are technically feasible.

B1.1. IR spectrometer with high time resolution

The construction of the optical spectrometer for high-time measurements corresponds to that of a conventional grating spectrometer with an entrance slit whilst the aperture of the detector element will be used as an exit slit. The optics will be placed in a cryostat at 77 K to minimise the thermal background radiation. The linear detector will consist of 256 individual MCT elements allowing a maximum spectral resolution of 4 cm⁻¹ over a 1000cm⁻¹ bandpass. For the biologically interesting spectral areas around 3000 cm⁻¹ (lipids), 1600 cm⁻¹ (proteins) and 1000 cm⁻¹ (carbohydrates) three different interchangeable gratings will be used. It is also possible to optimize the peak detectivity of MCT semiconductors through the MBE process so we will investigate procuring three optimally designed narrow band linear detectors for the three regions of interest. Photovoltaic MCT detectors (HgCdTe) allow a time resolution of better than 2 µs and the AS revolution frequency of the electron beam (1.3879 MHz) can be used as an intrinsic



detection frequency for the infrared synchrotron light. If it becomes apparent in the course of the development that photovoltaic MCT detectors can not be procured, slower photoconductive MCT detectors with a time resolution of better than 50 μ s can be used instead together with a mechanical chopper for light modulation. Although slower, the photoconductive detectors do allow for the spectrum to extend below 900cm⁻¹ to 750cm⁻¹.

B1.2. IR Echelle spectrometer

The Echelle spectrometer consists of the functional elements shown in Fig. 2



Fig 2: Principle sketch of the Echelle spectrometer

The Echelle type spectrometer works like a normal grating spectrometer with entrance and exit slits but where different orders are deliberately spatially overlapped. The overlapping orders are separated by an additional prism (crossed dispersion) placed in the beam path. At the focal plane these orders are then spatially resolved on to a 2D array detector (see the stripes in Fig 3 taken with a visible Echelle spectrometer and a CCD).



Fig 3: Two-dimensional Echelle spectrum of a fluorescent lamp in the visible spectral range (Hgline spectrum overlaid with fluorescent continuum)



In order to achieve a high S / N it is important to accumulate a large number of carriers (~ photons) in the detector because the noise scales with the root of the number of carriers. The following is an assessment of the expected S/N for the IR beamline at BESSY for 1000 cm⁻¹ and realistic parameters for the detector scheme. With a spectral resolution of 4 cm⁻¹ a synchrotron radiation flux of about 1.8×10^{19} photons s⁻¹ hits the detector pixels. A pixel covers an area of 40 μ m × 40 μ m and generated at 77 K a dark current of 5.6×10^9 e s⁻¹ m². Contributions from the background radiation can be neglected if the housing temperature is 77K and the readout noise can also be neglected. With an integration capacity of 5×10^7 electrons (full well capacity) the maximum integration time calculates to 1.4 ms and one gets a S/N of 5,900 for a single read out. Taking spectra for 1 s with a rate of 500 Hz accumulation frequency the theoretical S/N increases to 130,000.

B1.3 Near field spectroscopy

The lateral resolution of transmission or reflection IR microscopy is controlled by the diffraction limit. Consequently to reduce the lateral resolution and obtain sub micron resolution near field techniques are required and a bright synchrotron source provides the possibility of obtaining good S/N in such experiments. To date most IR based systems have been developed using pulsed laser sources or FEL sources to irradiate the tip of an Atomic Force Microscope (AFM). The AFM is used control the tip position location and distance from the sample and to raster scan to obtain images. Raschke et al (ChemPhysChem, 2005, 6, 2197-2203) have developed scattering type scanning near field optical microscopy (s-SNOM) to obtain 10nm resolution images of copolymer samples. In these experiments an IR beam is focused on to the cantilever tip at the sample and the scattered light from the tip apex gathered through the same objective used for delivery. The confocal nature of the experiment minimizes background signals and contrast for imaging is obtained from differential absorption in the C-H stretch region. An alternative method relies on a photothermal effect where when the sample is illuminated at a wavelength corresponding to an absorption, the sample in the vicinity of the beam (typically <100nm) is heated instantaneously and the object expands causing displacement and subsequent oscillation of the AFM tip (A. Dazzi et al, Ultramicroscopy, 2008, 108, 635-634). The oscillation is recorded to provide a spectral response for each laser of FEL wavelength. If a broadband source is used rather than a laser then the oscillation provides an interferogram that can be processed by Fourier transformation to provide a full spectrum.



Fig 4a. Basic setup for apertureless near field imaging (max-planck-innovation.de).





Fig 4b Photothermal AFM probe in contact with polymer sample and resulting CH stretch absorption peaks recorded on IR beamline at Daresbury Laboratory (UK).

The systems described briefly above are currently being tested at Soleil and ANKA and we intend to develop the most promising of these techniques on the IR beamline.

B1.4 Pump-Probe capability

By using a laser source or synchrotron uv pulse time resolved infrared spectroscopy can be achieved. The time resolution is determined by the laser or synchrotron uv pulse and such a system using a ps pulsed Ti-Sapphire laser as an excitation source and the synchrotron (18.9ns bunches) as the probe has been developed recently at NSLS (Lobo et al , *Rev. Sc. Instr.*, (2002), 73,). We will develop a system on this beamline to carry out such pump probe experiments, firstly by extracting a uv beam from the synchrotron to provide ns resolution and at a later date incorporating a pulsed laser source to achieve ps time resolution.

B1.5 Spectroscopy in extreme environments

Microspectroscopy in extreme environments (eg. high pressure, live cells, high temperature, low temperature) is challenging and usually requires special devices (diamond pressure cells, flow cells, temperature controlled cells) and special objectives to probe such environments (long working length objectives, high NA objectives). Many such measurements are not currently possible and we envisage this beamline having interchangeable optics to allow for a wide range of experimental extremes. The AS IR beamline team led by Dr. Mark Tobin have an International Science Linkage grant under the French Australian Science and Technology program (FAST) for 2009-2011 to develop high pressure techniques in conjunction with a team at the Soleil synchrotron lead by Dr. Paul Dumas. Another group (led by Prof. Brendan Kennedy (USyd)) have an active ARC LIEF application to provide high pressure, low temperature equipment across the IR and X-ray beamlines. Together these teams will ensure progress in this area on the new IR beamline.



B2: Applications and Potential Outcomes to Australian Scientific Community

How does the project advance synchrotron-based research in Australia/NZ? What are the likely outcomes? Include specific examples where possible.

The concept of the beamline is to make advances at the cutting edge of synchrotron infrared spectroscopy and provide a resource with unique capability. The major advantage of the current IR microscopy beamline at the AS is the ability to record single point spectra at diffraction limited resolution. To record high spatial resolution, high S/N spectral images, which is a major desire of the majority of users, the sample must be raster scanned. For even small samples this takes many hours, compared with the minutes required for IR focal plane array (FPA) imaging instruments, albeit at higher S/N. One of the major thrusts of IR spectroscopy is disease diagnosis and monitoring and an ability to rapidly identify and monitor disease before the onset of physical symptoms would be a major advance.



Fig 5. Cerebellum images showing the relative concentration of (a) lipid and (c) nucleic acid derived from FPA spectral data. (b) Contiguous section stained with H&E, blue square indicates area interrogated using the higher spatial resolution mapping using the synchrotron source. Scale Bar = 100µm. (e) Increased magnification of the areas interrogated with the synchrotron light source with immunofluorescence showing myelin organisation and cellular infiltrate following staining with an anti-MBP antibody (green) and DAPI (blue). (d) Lipid ester carbonyl concentration map and (f) nucleic acid concentration derived from synchrotron mapping data overlaid onto the immunofluorescence stained section.

An example project that would benefit immensely from rapid synchrotron imaging would be the location of early stages of Experimental Autoimmune Encephalomyelitis (EAE) an animal model

of Multiple Sclerosis (MS) (Biophotonic infrared imaging sheds new light on the pathology of autoimmune-mediated demyelination. Philip Heraud, Sally Caine, Naomi Campanale, Tara Karnezis, Mark J. Tobin, Donald McNaughton, Bayden R. Wood and Claude C.A. Bernard, accepted in Neuroimage Sep 2009). Fig 5 above shows FPA IR spectral images of *ca* 0.7mm² sections of brain tissue (20 min collection) compared with stained sections. Possible micro lesions are only just discernible from the FPA images and the synchrotron IR beamline was required to identify them as early onset EAE. Fig 6 shows synchrotron IR maps of the small

areas delineated in fig 5 where an Artificial Neural Network (ANN) trained from later disease stages successfully delineates disease on samples taken from animals prior to the onset of physical stages of the disease. With a rapid imaging system the full scale maps could be obtained at the highest possible spatial resolution and microlesions located rapidly via ANN providing diagnosis. The same advantages apply in other fields



Fig 8. Synchrotron ANN maps of suspected micro-lesions. The grey regions correspond to lesions.



that require imaging to both locate and identify/characterize micro-samples within complex matrices such as in geology, forensics, semiconductors, polymers, materials science, cultural heritage and pharmaceuticals.

As well as rapid imaging at "normal" diffraction limited lateral resolution the beamline is envisaged to be able to provide lateral resolution down to 10nm, a distance approaching intra molecular dimensions. This will lead to molecular spectral information at an unprecedented resolution and provide probes capable of flowing chemical change at sites such as inside cell organelles, at interfaces.

B3: Match to Selection Criteria

• Meet the demands of an identified group of researchers for new techniques

The infrared community in Australian and New Zealand is wide spread and committed to the present beamlines. There are many research problems that arise however that require facilities and techniques well beyond those currently available. This proposal will provide a beamline that will be driven by the community to provide cutting edge facilities.

• Take advantage of the existing third generation light source

Due to the beam extraction design and the remarkably stable synchrotron light output the AS IR beamline is already one of the most stable in the world, providing better S/N and higher spatial resolution than all others used by Australian scientists. This stable flux of the AS will provide an excellent platform on which to build an advanced beamline to provide for the Australia/New Zealand community.

• Will position Australasian scientists at the leading edge of their field.

The capabilities envisaged for the beamline and outlined above will provide facilities beyond those of comparable beamlines around the world. With the current IR beamline providing a "workhorse" for diffraction limited spectroscopy, ATR spectroscopy and far-IR spectroscopy the new beamline will provide opportunities to position the community at the forefront of developments in IR spectroscopy.

• Can be demonstrated to be feasibly constructed within a 3 year timeframe.

A second extraction port for IR already exists and we envisage using the same extraction optics that have been highly successful in providing the current IR beamline. The pump-probe techniques will take advantage of a system already active at NSLS, the near field technique systems require putting together existing technology and the high pressure, high/low temperature systems will be supported by two projects already in place. Hence the major time involved will be design and construction of the dispersive spectrometer system and its control systems. Two highly credentialed IR beamline scientists (Paul Dumas and Ulrich Schade) have checked the feasibility of the ideas and availability of components and we envisage being able to complete this well within three years.

B4: Potential Users

Does the project address a clearly identified need in the community? The need may be actual or potential.

The infrared community have been active in running workshops and conferences, most recently through sessions at the International Conference on Vibrational Spectroscopy (Melbourne, Jul 2009) where a number of international speakers attended a synchrotron workshop. The on going



development needs of the community are discussed at these venues and this proposal comes directly out of those meetings. The more local Australasian conference on vibrational spectroscopy (ACOVS) attracts around 100 delegates, and it is this scientific cohort driving this proposal. ACOVS9 has already been set for Wellington in mid 2011. IR spectroscopy is also used in many academic disciplines, industry and government research and the cohort of potential users is well beyond those attending ACOVS or presently using the IR beamline. Hence we would expect new facilities and techniques to attract further users.