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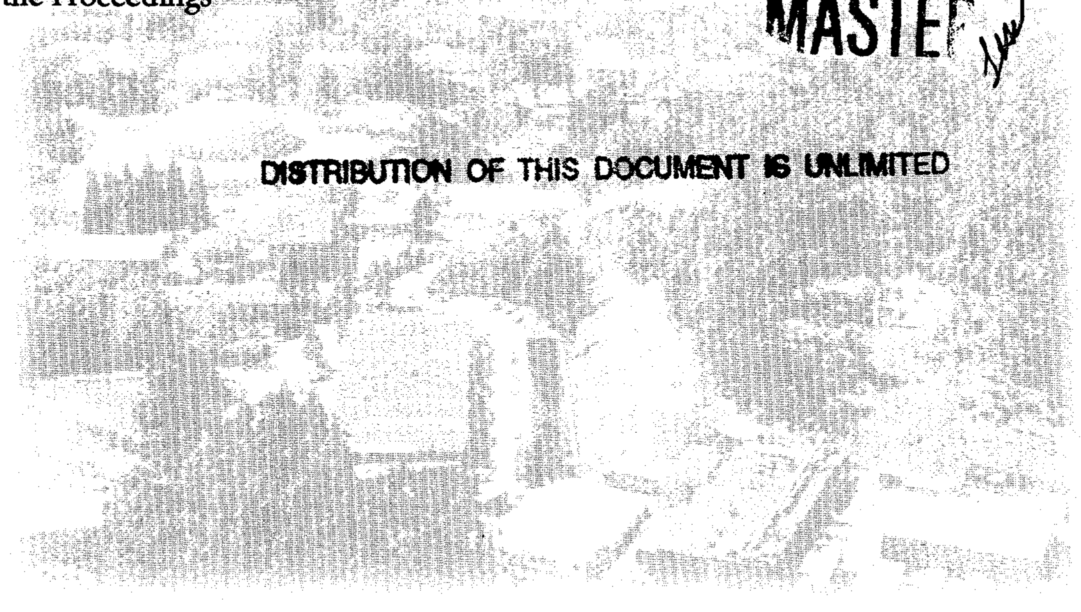

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THE FIRST SYNCHROTRON INFRARED BEAMLINES AT THE ADVANCED LIGHT SOURCE: MICROSCPECTROSCOPY AND FAST TIMING

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ABSTRACT

A set of new infrared (IR) beamlines on the 1.4 bending magnet port at the Advanced Light Source, LBNL, are described. Using a synchrotron as an IR source provides considerable brightness advantages, which manifests itself most beneficially when performing spectroscopy on a microscopic length scale. Beamline (BL) 1.4.3 is a dedicated microspectroscopy beamline, where the much smaller focused spot size using the synchrotron source is utilized. This enables an entirely new set of experiments to be performed where spectroscopy on a truly microscopic scale is now possible. BL 1.4.2 consists of a vacuum FTIR bench with a wide spectral range and step-scan capabilities. The fast timing is demonstrated by observing the synchrotron electron storage pattern at the ALS.

INTRODUCTION

Synchrotron-based infrared beamlines provide considerable brightness advantages over conventional (thermal) IR sources [1]. This brightness advantage manifests itself most beneficially when measuring very small samples. In the commissioning of the first IR beamline at the ALS, BL 1.4.3, we have experimentally measured the small spot-size obtained by our IR microspectroscopy system when using the synchrotron beam as the source and we compare it to the conventional Globar source. We demonstrate the diffraction-limited focus and the corresponding factor of ~100's improvement over conventional sources in measured signal through very small apertures. This new tool enables a host of new scientific pursuits where FTIR spectroscopy (indicating chemical species, phase changes, etc.) can be monitored on a microscopic scale.

Synchrotron-based infrared beamlines also provide a unique opportunity for doing time-resolved IR spectroscopy. Synchrotrons operate with electrons traveling in bunches around the storage ring. Each bunch emits a pulse of synchrotron radiation every time the electrons are forced to turn via a bending magnet. This creates a train of light pulses with a temporal spacing of 2 nanoseconds in the case of the ALS (see Figure 4). Pump-probe type experiments can therefore be performed at speeds up to 2nsec. We provide a demonstration of this fast-timing capability using the electron filling pattern in the ALS storage ring.

EXPERIMENTAL CONFIGURATION

The details of the construction and commission of these IR beamlines is provided in greater detail in a previous publication [2]. Briefly, the synchrotron light is collected from the 1.4 bending magnet through a 10mrad vertical and 40mrad horizontal opening, as schematically drawn in Figure 1. This light is deflected vertically by 0.5 meter, then is refocused outside the shield wall by an ellipsoidal mirror, m2. A 'switchyard' then contains a series of optics to collimate the IR beam and distribute the beam to one of three end stations.

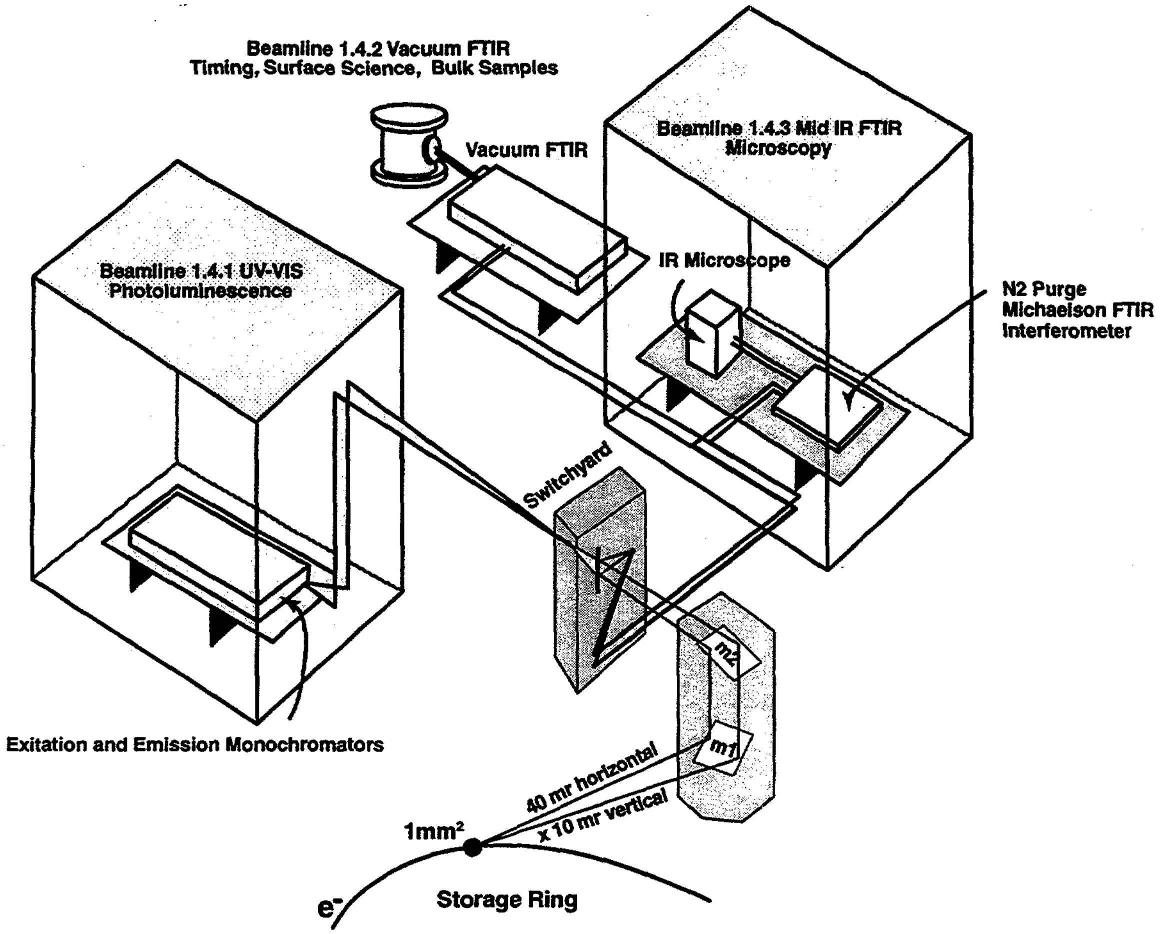

Figure 1. Schematic drawing of ALS beamlines 1.4.1, 1.4.2, and 1.4.3.

BL 1.4.3 inputs the collimated synchrotron light into a Nicolet Magna 760 FTIR bench. The modulated light is then passed through a Nic-Plan IR microscope that can perform both transmission and reflection measurements. The sample stage is computer controlled for motions in the x and y plane enabling automated spectral measurements across a sample with steps as small at 1 µm.

BL 1.4.2 uses the synchrotron light for the input of a Bruker IFS 66v/S vacuum FTIR spectrometer. The light then passes through a standard sample compartment, or out one of three external ports. One external experiment being set up is a UHV surface science chamber that will allow grazing incidence reflectivity measurements. This IFS 66v/S instrument has a wide spectral range, 50 cm^{-1} to 25,000 cm^{-1}, and it has step-scan capabilities in addition to rapid-scan to enable fast timing measurements.

SPOT SIZE

We have made measurements to determine the small spot sizes achievable with the infrared microspectroscopy equipment on beamline 1.4.3. The spectrometer software can obtain an area scan by moving the sample stage with 1 micron spatial precision under the focused IR beam and acquiring FTIR spectra at each point. To determine the actual focused spot size of the synchrotron beam and compare it to the internal Globar IR source, we used a 10 µm pin hole and measured the transmitted spectra as a function of the pin hole position. No other beam-defining apertures were used.

Integrating over the measured mid-IR range, we obtain an intensity number. The intensity as a function of pin hole position is shown in Figure 2. A small spot size readily apparent.

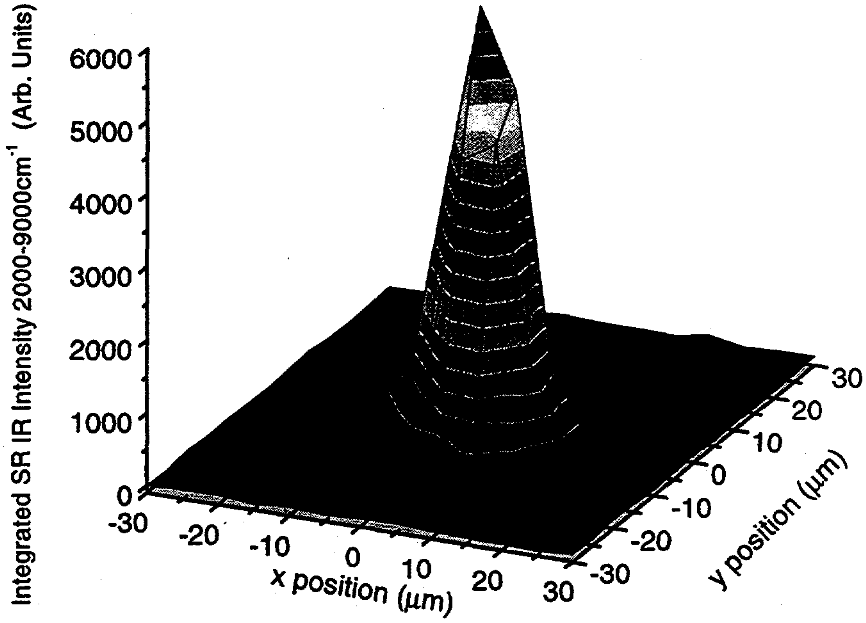

Figure 2. Integrated IR signal intensity from 2000 - 9000cm^{-1} through a 10μm pin hole being scanned on microscope stage. There are no other apertures in the optical path. This graph demonstrates the small spot size achieved using the synchrotron IR beam

In Figure 3 we plot the x and y profiles of this spot along with fits to a Gaussian line-shape. The data fits very well to a Gaussian with resultant widths of 10 μm in x and 8 μm in y. This spot size is becoming roughly diffraction limited for the low-energy end of our detection capabilities (1000 cm^{-1}= 10 μm wave-length).

When we compare the above measurements to analogous measurements made with a conventional Globar thermal IR source, the brightness advantages of the synchrotron become readily apparent. The Globar has a much broader peak profile of around 100 μm in width simply

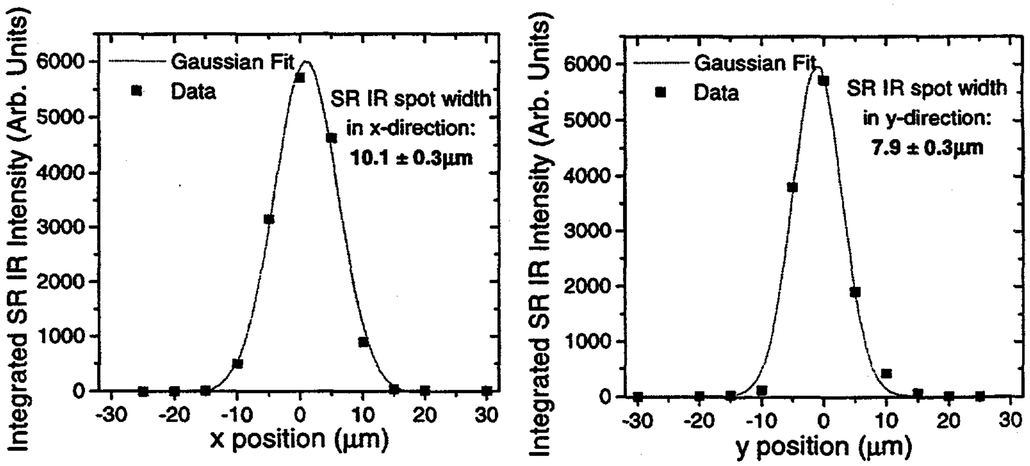

Figure 3. Peak shape profiles in the x (left panel) and y (right panel) directions. We observe a well-defined Gaussian peak shape with widths of 10μm in x and 8μm in y.

due to the large source size of this glowing filament source making a better focus impossible. Therefore, while the overall amount of light passing through the optical system from the Globar and the synchrotron sources is comparable, nearly all of the synchrotron light can be focused onto a 10 μm spot. To achieve a similar spot size using the conventional Globar source, one must simply mask the source and throw away a large factor of signal intensity. We measure improvements of several hundred in intensity through small pin-holes (2 - 10 μm diameter) for the synchrotron source compared to the Globar.

One example of the use of BL 1.4.3's microspectroscopy capabilities is the non-destructive monitoring of the *in situ* inorganic-organic interactions at biological-mineral interfaces [3]. The detailed localization of these bacteriological processes is only possible using a synchrotron based instrument due to the high signal at small spot sizes providing excellent spatial contrast.

FAST TIMING DEMONSTRATION

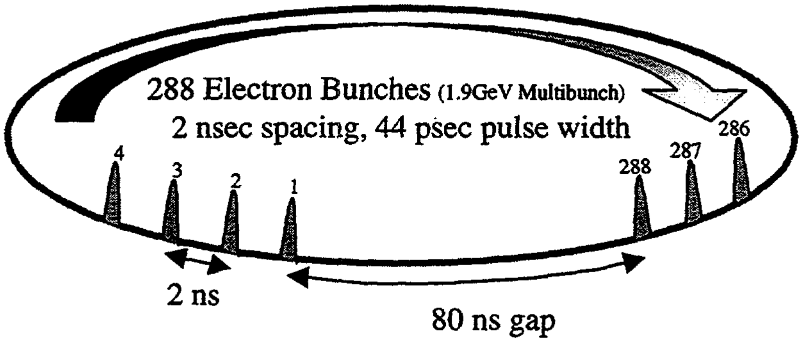

Figure 4. The ALS generates pulses at intervals of 2nsec, except for a single long gap of 80ns. We use this time structure to demonstrate the fast timing capabilities of BL1.4.2.

Standard rapid scan Fourier Transform Infrared Spectroscopy (FTIR) acquires scans on the time scale of one second, so the very fast pulses of a synchrotron source are not noticed. However, using the step-scan capabilities and fast electronics of the Bruker IFS 66v/S FTIR spectrometer on BL1.4.2, we can measure IR spectra with a time resolution as fast as 5 nsec. This means that processes that occur on the nanosecond all the way up to hour time scales can be monitored with time-resolved spectroscopy using this beamline.

To demonstrate the fast-timing capabilities of this beamline, we synchronized the detection electronics with the ALS ring timing structure (one trigger per complete revolution of the electrons). The ALS typically operates in multibunch mode with 288 bunches spaced 2 nanoseconds apart, followed by an 80 nsec gap. The individual pulse width is 44 psec (FWHM). This electron filling pattern is schematically drawn in Figure 4.

IR spectra were obtained at 5nsec time slices for a total of 650 nsec (a complete revolution of each electron bunch). We plot the measured intensity as a function of time integrated over a region in the visible wavelengths in Figure 5.

Figure 5 clearly demonstrates that we can observe the 80 nsec gap in the synchrotron light pulses. The IR detector and digitization electronics are not yet fast enough to observe the 2 nsec pulse spacing, but forthcoming enhancements should allow sub-nanosecond timing enabling the use of individual synchrotron pulses to serve as the probe in very fast time-resolved experiments.

CONCLUSIONS

BL 1.4.3 utilizes the high brightness aspects of the synchrotron light source to perform IR microspectroscopy at spatial resolutions much higher than is possible with conventional IR sources. Experimental systems where the sample size is quite small, or where features on a sample are on the micron size scale, will gain significantly by using ALS Beamline 1.4.3. A few examples include observing bacteria ingesting specific chemicals in real time [3], localization of

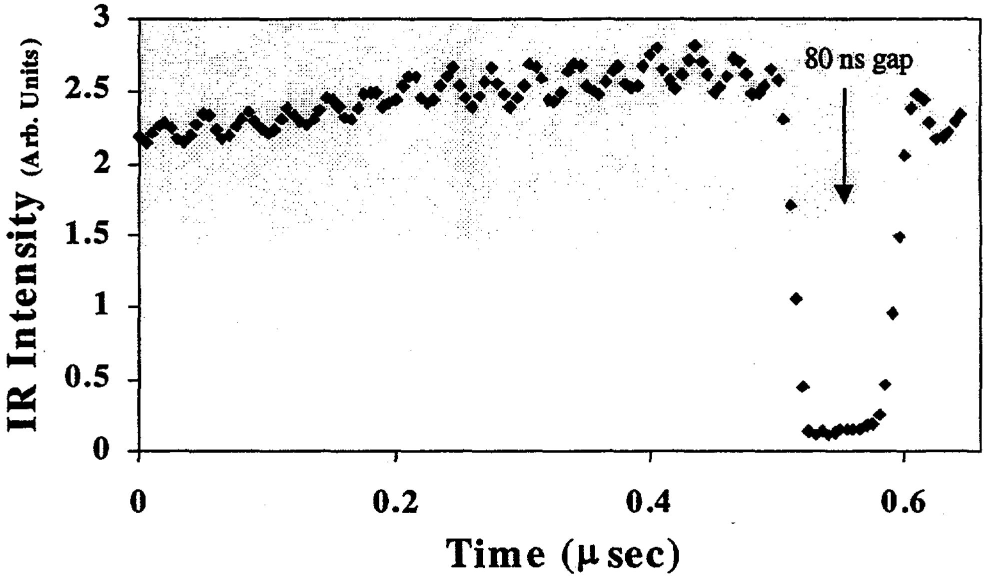

Figure 5. Time-resolved measurement of the IR intensity showing the 80 nsec gap in the ALS synchrotron filling pattern. The time resolution of the current electronics does not yet fully separate the 2 nsec spacing between pulses.

adsorbates on a variety of surfaces, particulate contamination on semiconductors, polymer laminates and composites, and analytical forensic studies of small samples.

The step-scan capabilities of the IFS 66v/S FTIR spectrometer on BL 1.4.2 allow for very fast time-resolved IR spectroscopy. This instrument, in conjunction with the ALS synchrotron's pulsed nature, makes possible a host of new time-resolved and pump-probe spectroscopic experiments. Typical applications for this beamline will include pump-probe measurements (semiconductors, metastable states), environmental science (adsorbates, bacteria, soil chemistry, bioremediation), biological materials (identification of biomolecules, time-resolved chemical reactions), high-pressure systems (materials in diamond anvil cells), and measurements at cryogenic temperatures and/or in high-magnetic fields (far-IR reflectivity from high-T_c and other correlated electronic systems).

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