The First Infrared Beamlines at the ALS:
Final Commissioning and New End Stations[+]

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ABSTRACT

The design and initial commissioning of the first IR beamline at the ALS has been described previously.[1] We report the final commissioning and first results of the mid-IR spectromicroscopy beamline 1.4.3. In addition, several improvements and two new branchlines are presented. Beamline 1.4.2 is connected to the front end under vacuum and consists of a Bruker Rapid- and Step-Scan vacuum FTIR bench. The modulated light is then coupled into a UHV surface science chamber for grazing incidence reflection studies. Several more external ports are available from the Bruker bench. Beamline 1.4.1 receives light from a separate port on the beamline 1.4 front end and connects to an optical table for photoluminescense and other experiments using photons with energies up to 6eV.[2]

Keywords: Infrared, synchrotron, microscopy, surface science, photoluminescence

1. INTRODUCTION

The infrared (IR) beamline complex at the Advanced Light Source (ALS) has expanded to include three separate end stations. The original objective of adding an IR front end to the ALS for the clearly demonstrated and well established use of mid IR microscopy[3] has been broadened to include a second IR instrument for bulk samples, surface science, and fast timing experiments; and the inclusion of an ultraviolet/visible (UV/VIS) end station for photoluminescence experiments. This paper lists the current status of the beamline complex, and some of the difficulties and results of the final commissioning.

1.1 Beamline 1.4.3—Mid IR Spectromicroscopy

Figure 1 shows the 1.4.3 Spectromicroscopy facility on the right hand side of the diagram. The bending magnet synchrotron light is reflected from a plane mirror M1, an ellipsoidal mirror M2, and a flat mirror M3 all in ultra high vacuum (UHV). The UHV ends with a wedged diamond window. The light proceeds from the diamond window in rough vacuum in the switchyard where it is collimated by two cylinders. The light enters the connecting piping as a parallel beam designed to optimally fill the objectives in the Nic-Plan IR microscope. The reader is referred to reference 1 for the details of this design. We report here final changes that were implemented during commissioning. It was necessary to modify the Nicolet 760 IR Bench so that the synchrotron beam would enter the instrument off center in the field. This allowed the beam to miss the center of the beamsplitter and the laser detector. The center of the beamsplitter has a different coating optimized for the HeNe fringe counter, which is the wavelength standard. The light was put back on center by modifying the double sliding mirror just after the interferometer in the optical train to have different end stop positions. Nicolet facilitated this process for us by providing the name of the manufacturer of the slide

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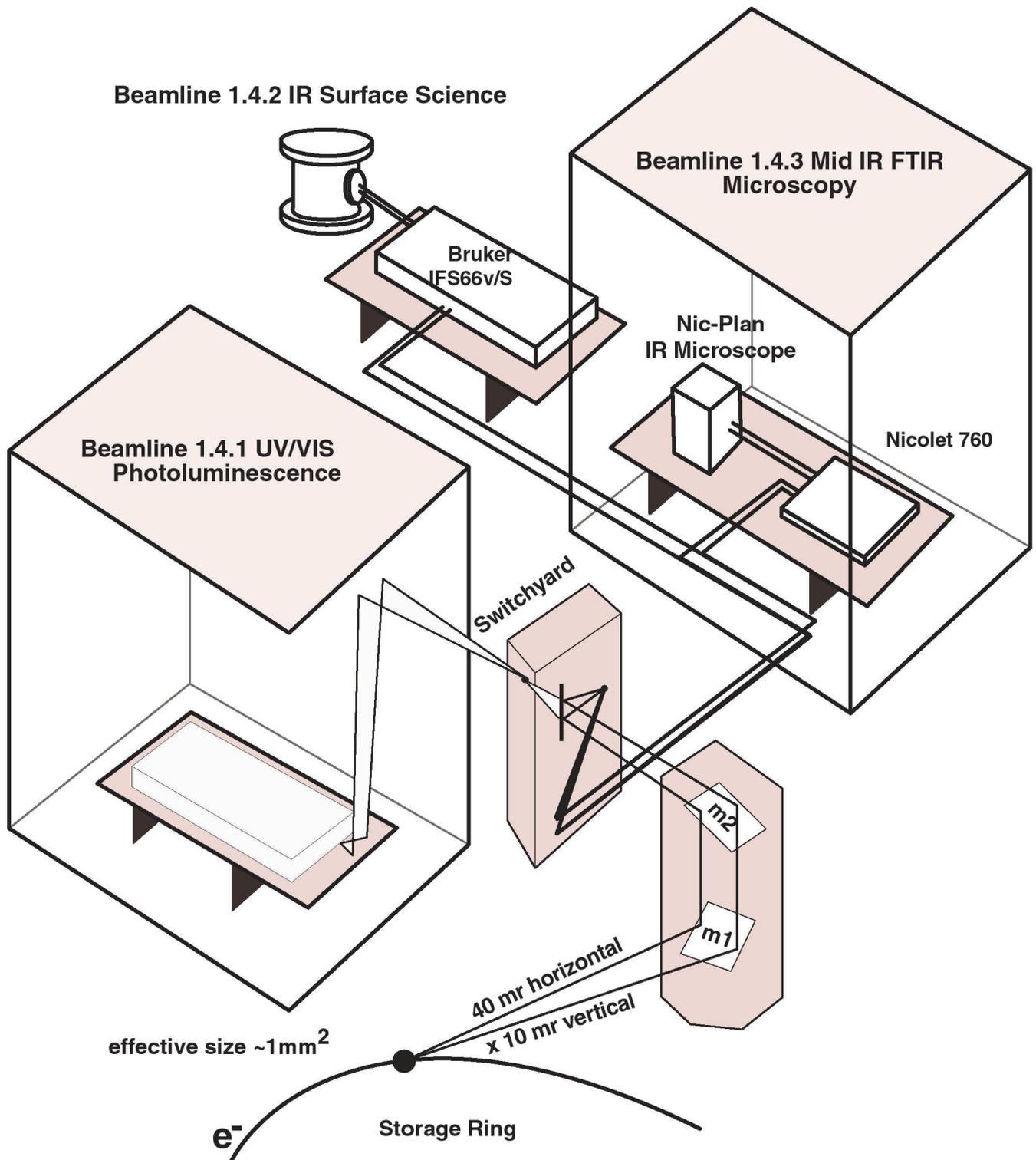
Figure 1 Schematic Layout of the Beamline 1.4.x Complex

mechanism, and selling us an extra one to modify. We also wish to thank Uwe Arp of NIST who originally pointed out the problem to us. Good signal strength has been obtained in the microscope. The overall power from the synchrotron is of the same order as the internal IR source. Typically the microscope operated in reflectance mode measuring the reflectance of a gold mirror standard shows a fully saturated signal on the MCT detector on the lowest gain setting of 1 with full ring current and VVR2 open. VVR2 is the fused silica windowed valve, which protects the storage ring if the switchyard is open or up to air (See reference 1). Figure 2 shows the essentially diffraction limited spot size obtained with the microscope. This map of intensity through a five-micron pinhole was made with no apertures in the beam other than the opening in the storage ring at the front end. The entire intensity in the mid IR region can be focused into this diffraction limited spot. This amounts to the order of 200 times more light available within a 10-micron area than with the internal Globar[TM] IR source.
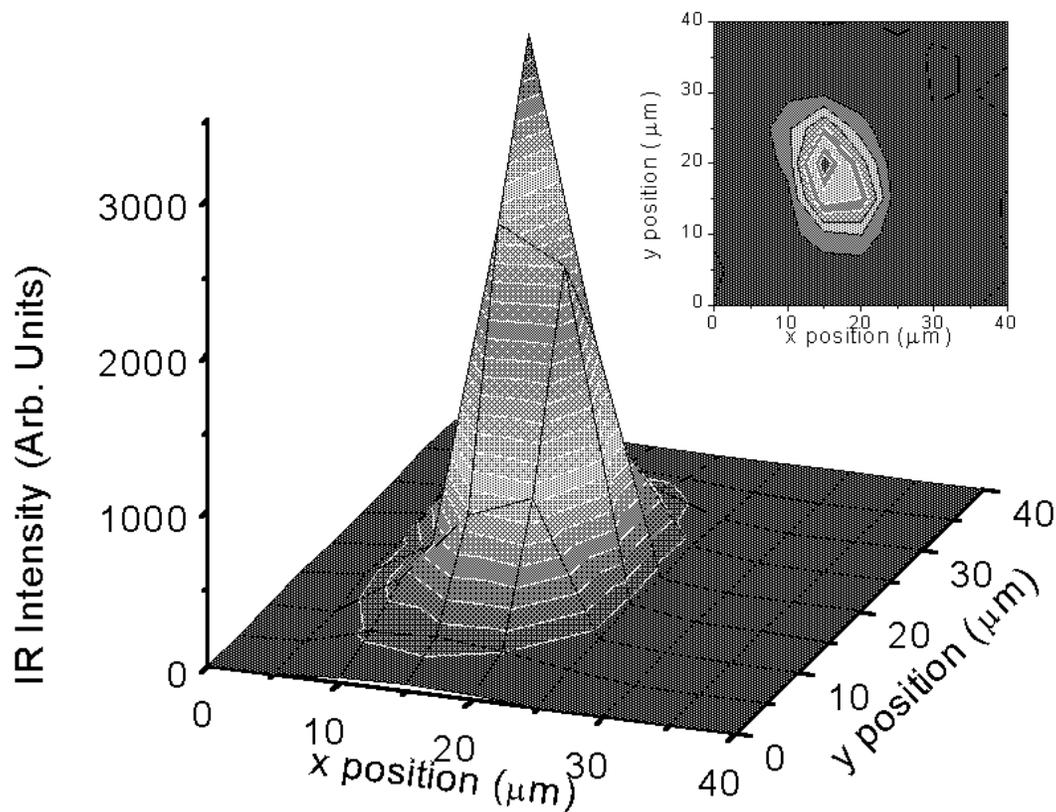
Figure 2 Measurement of the un-apertured spot size in the focal plane of the IR microscope integrated over mid IR wavelengths.

Figure 3 shows an example of what can be done with this small spot size. The work of Holman *et al.*[4] has shown that small (~20 micron) colonies of specific bacteria can reduce highly toxic Chromium in the VI oxidation state to the much less soluble, and therefore much less toxic trivalent chromium. When toluene is present as a co-contaminant the Cr (VI) reduction is observed to proceed quicker, presumably because the toluene is used as a carbon source. The figure shows three reflectance maps all taken from the same spectral data set of a bacteria colony living on rock. The colony of bacteria is identified by the presence of the well known amide II peak of protein, and the absence of both toluene and chromium VI peaks over the colony strikingly shows the potential of the synchrotron-based FTIR technique. Obtaining a comparable signal to noise with the internal source would make the local depletion of Cr (VI) and toluene impossible to observe, and would eliminate the specific identification of the chromium reduction with the colony of bacteria.
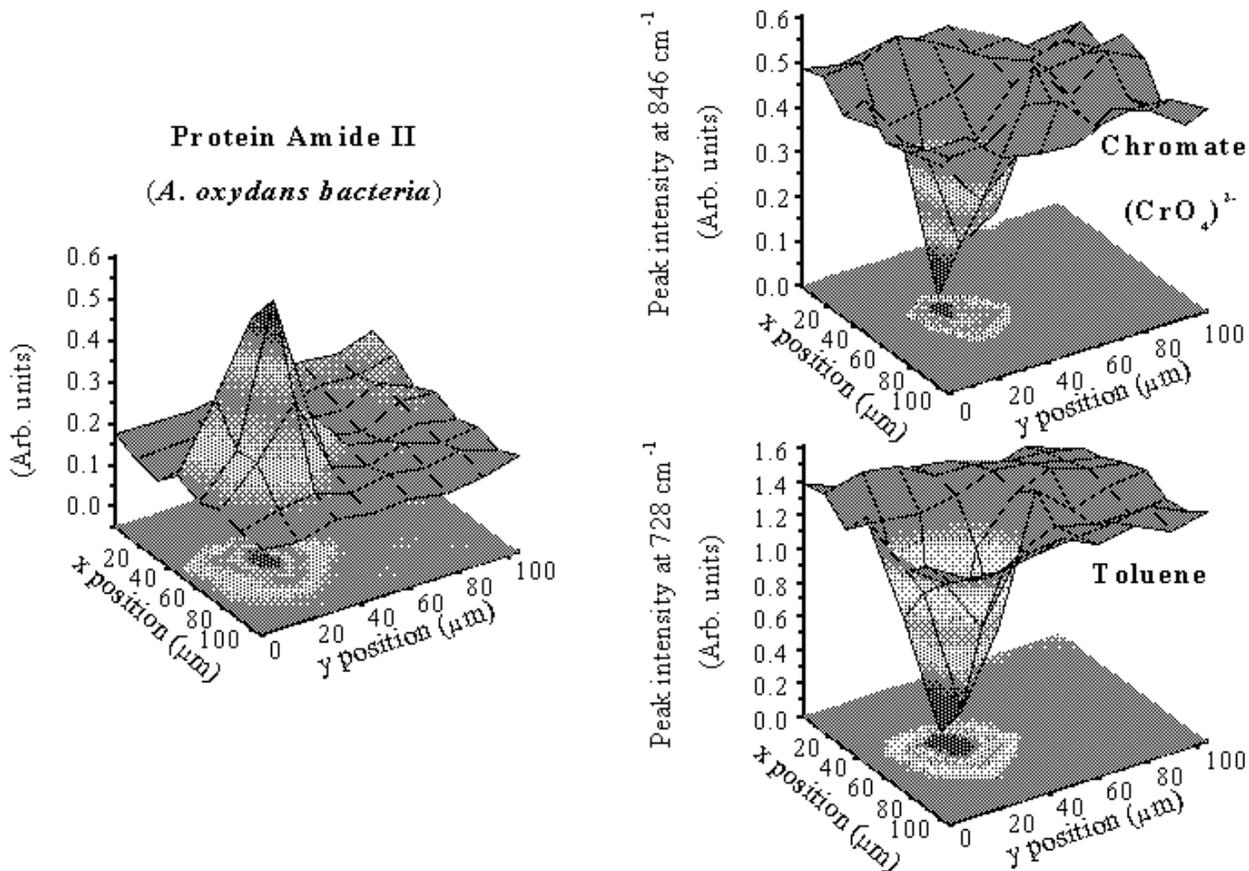
Figure 3 Anthrobacter oxydans bacteria, isolated from a contaminated DOE site in Idaho, attach themselves to magnetite mineral surfaces. We locate the bacteria via their spectral signature (above). We observe a depletion of chromate and toluene (right) by the bacteria after five days of exposure.

1.2 Beamline 1.4.2—Vacuum IR Spectroscopy

Figure 1 shows the 1.4.2 Vacuum IR Spectroscopy facility in the middle of the diagram. The next to the last flat mirror in the beam pipe between the 760 bench and the microscope is removable under vacuum allowing the parallel beam to proceed to the second rigidly mounted optical table. On this table is placed the Bruker *IFS*66v/S vacuum FTIR spectrometer with step scan capability. This instrument was chosen for its versatility and the ability to do step scan and timing, and for the capability to go to far IR wavelengths. Typically, synchrotron sources provide more power in the far IR than conventional sources, but we have yet to make a detailed comparison on 1.4.2. (In the case of microscope beamline 1.4.3 the synchrotron clearly has more output at the longer wavelengths in the mid IR when compared to the internal source in the bench.) The Bruker bench is equipped with a 5 ns A/D capability which was the fastest available at the time of purchase. We wish to acknowledge the guidance of Carol Hirschmugl[5] in the selection of this instrument. A significant factor in its selection was the commitment of the Bruker Company to push the limits of fast timing to at least 0.5 ns. Figure 4 clearly shows the break in the bunches of about 80 ns when the ALS is completely filled, and we expect to resolve individual bunches when the faster electronics and detectors are added.
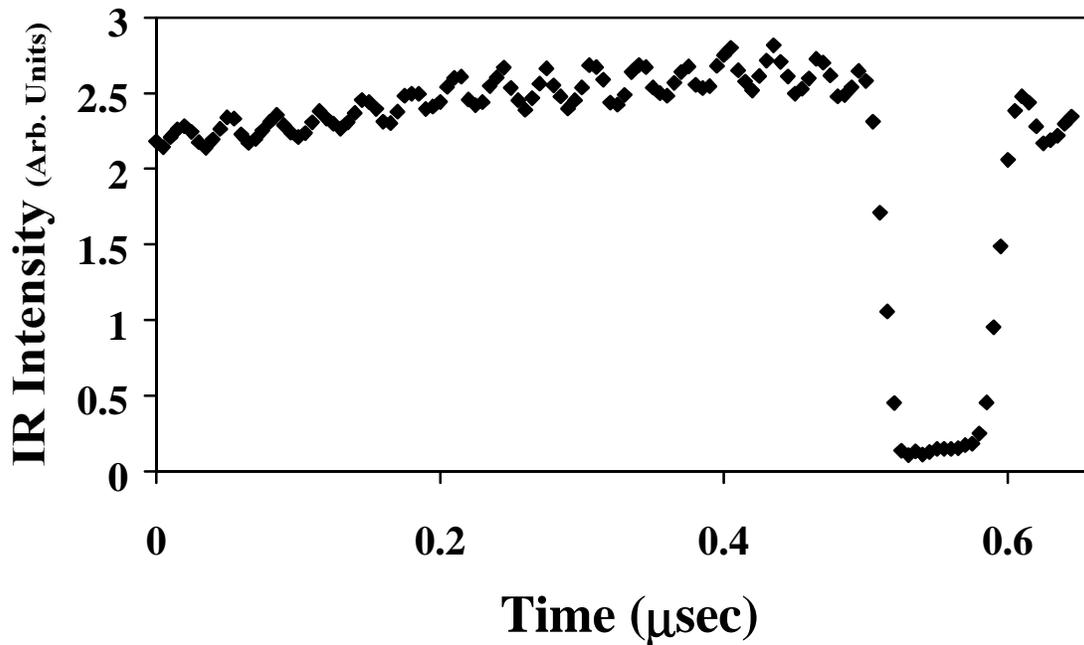
Figure 4 Total integrated IR intensity from the Bruker bench using the ALS timing signal showing the normal gap in bunches. The periodic pattern along the top of the plot may be an effect of the 2ns pulse time interval (500 MHz oscillator) with respect to the 5 ns digitization rate

We have purchased several beamsplitter/detector combinations including a liquid He-cooled silicon bolometer. A low temperature cryrostat is in the process of installation and debugging this summer for use in measuring the far IR reflectance of many novel strongly correlated electron materials. A mercury lamp is installed which will allow a good comparison of the far IR output of the beamline with respect to the more conventional source. The vertical aperture limit of 10 mr. (full angle) at the storage ring aperture should be the limiting factor in how far the long wavelength range will extend. The edge radiation from the sector zero injection straight may be a much better way to extract far IR from the ALS given the vertical opening angle constraints. See, for example, the paper and cited references in Bosch.[6]

1.3 Beamline 1.4.1—UV/Vis Photoluminescence

The left side of Figure 1 shows the UV/VIS photoluminescence area. M1 and M2 are coated with bare aluminum. This coating with the natural oxide layer helps absorb extreme UV which would normally pass gold IR mirrors, (see reference 1) but preserves reflectivity to about 6 volts (see reference 2). For use of 1.4.1 M3 is lowered, allowing the light to pass into an extra low vacuum box welded onto the switchyard where the light is collimated with an off-axis paraboloid. This mirror was supplied by the Bruker company, and is used as one of the mirrors in the *IFS*66v/S. We have had considerable problems in keeping the UV flux up near predicted levels due to contamination of the mirrors. Contamination of the switchyard chamber is suspected, and a compromise has been worked out where the switchyard is partially back-filled with nitrogen gas, leaving VVR2 closed during UV/VIS operation. Standard UV/VIS components, for example, quartz lenses, monochromators and spectrographs are used on the optical table. Flux approaching calculated values (including lens absorption, etc). are obtained under ideal conditions.

2. Commissioning

2.1 Front End and Switchyard

At the end of reference 1 we showed the photocurrent from the finger mask as a function of the height of the M1/M2 chamber. The finger mask has proven to be a good concept, with none of the potentially serious problems of fatigue or water leakage showing up thus far. The incoming and outgoing temperatures of the cooling water are monitored with thermocouples as a precaution.

The chemically vapor deposited diamond window was thought at first to be a potential problem because of the intense scattering of visible light in the switchyard by the window. BRDF measurements[7] at 10.6 microns showed, however, that there was essentially no scattering in the mid IR region. The average crystal size in the window must be of the order of a micron which would explain the excellent imaging of the beam in the mid IR (Figure 2) even while the visible light patterns on the mirrors in the beamline are quite rough.

2.2 Noise

Reduction of noise in the FTIR spectra of the microscope has been the major challenge in the commissioning of the beamlines. Initial mid IR RMS noise measurements taken by dividing two sets of 64 spectra at a resolution of 4 cm^{-1} from reflection off a gold coated microscope slide in October, 1997 showed the considerable, and intolerable value 0.5 %. Even though a several day study of vibration in the area was done prior to the construction of the beamline, two 20-hp pumps, which circulated water for the RF cavities in the storage ring, were not operating at the time of the testing. All storage ring systems were turned on for the testing except this one. The pumps drove the shield walls and floor of the ALS at their fundamental rotation (58 Hz) frequency and at the fourth multiple of that frequency. In addition the low conductivity water (LCW) supplied by the ALS was plumbed near the lines for the RF chiller water, and picked up the same frequencies. The systematic reduction of vibration problems is characterized in Byrd *et al.* in this volume. Table one briefly lists some of the vibration problems and the solutions applied so far. We have succeeded in reducing the typical noise a factor of ten to twenty to the range of <0.05% RMS which we believe is typical of the day to day noise level at synchrotron-based IR facilities. We are using the noise level obtained in the IR surface science work of Hirschmugl *et al.*[8] as the "gold standard" for synchrotron-based IR noise of 0.005% It is at least our goal in noise reduction, if not the attainment of equivalent noise to the internal source. It is our feeling that the final resolution of noise questions for synchrotron-based microscopy beamlines will require a complete systems analysis including the entire optical train including the MCT detector.

Vibration Sources	Solutions		
20 hp RF water pumps	Variable drive frequency to ~44 Hz from ~60 Hz	Smaller diameter impellers	Less constriction in valving
Coupling to front end	Mount from subflooring	Saw loose from top floor	
Vibrating M1/finger mask	Lessen flow rate	Use separate chiller, separate hoses from ALS LCW	Set chiller temp at 60F
Vibrating Switchyard	Mount from subflooring	Saw loose from top floor	Stiffen & fill mount with water
Vibrating optical mounts	Replace all small mirror mounts with Bruker mounts	Eliminate mechanical slides wherever possible	
Vibrating beam pipe betw. front end and shield wall	Remount off the wall, Freeing the bellows	Eliminate ~22 Hz resonance	
RF 500 MHz master oscillator, 2-8 kHz noise	Realize sidebands drove Robinson oscillation (Byrd)	Buy new master oscillator for storage ring	
Remaining noise below 200 Hz	Build active feedback systems (in process)	Two loops necessary for angular correction	

Table 1 A brief catalogue of vibration sources and remedies. See Byrd *et al.*, this volume for details.

In order to approach this level of noise we are implementing an active feedback system based on Hamamatsu position sensitive detectors, Physik Instrumente two-axis tip/tilt piezo mirror mounts and custom feedback circuitry. We wish to thank Ed Melczer of the Atomic Vapor Laser Isotope Separation Program (AVLIS) at Lawrence Livermore Laboratory (LLNL) for suggesting the LLNL approach, and access to the technology. This work will be described in a later publication.

3. Summary and Conclusion

Even given the significant noise problems catalogued above, the tremendous signal to noise advantage provided by the brightness of the synchrotron source provides a real, significant advantage for spectromicroscopy studies in the mid IR wavelength range at spatial resolutions of <=10 microns. Absorption spectra from single human cells and bacteria are now routinely possible. We wish to strongly emphasize that the magnitude of angular motion, which we are attempting to eliminate, is, at most, a few microradians, which would not, in general, affect normal dispersive beamline instruments. Due to the nature of the interferometers used in the IR, and the Fourier transform method of processing the data, minute changes in the direction of the beam map into both specific places in the IR spectrum and also produce broad band noise throughout the spectrum. However, this problem is minor when compared to the signal to noise advantage for microscopy at spatial resolutions smaller than achievable with laboratory systems. Even better performance will be available once all sources of noise are identified and minimized at all synchrotron-based IR facilities.

The ALS IR beamlines have been expanded for uses in the far IR for the investigation of High-T_c and related oxide systems, where, in principle, the synchrotron will provide more far IR signal, and step scan and timing abilities have been added. It is expected that one or two IR surface science chambers will be routinely connected to the Bruker bench. The synchrotron IR brightness advantage also gives more signal in the grazing incidence geometry of IR surface science. Good coupling to the surface dipole moment of adsorbed molecules requires very grazing angle reflectance geometry[9] and hence a small solid angle of acceptance for the radiation. The UV end of the spectrum from the 1.4. front end is now also used for photoluminescence and other related measurements by a third end station.

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