Mid-Infrared Beamline

Infrared spectroscopy has been a major analytical tool in industrial application and basic research for decades, by virtue of the ability to identify molecular constituents of complex material by their "fingerprints" in the vibrational spectra. In the last decade, advancement in optics and detectors have made spatially resolved infrared spectroscopy (microspectroscopy) a popular technique for the analysis of inhomogeneous samples and small particles. Furthermore, the IR microscope based on Fourier-transform spectrometers became commercially available. Generally these instruments use a black-body source that radiates into a large solid angle. In contrast, synchrotron radiation provides a very bright source and a highly collimated beam that enable high spatial and spectral resolution measurements of small samples. While the total flux from synchrotron radiation is less than a typical laboratory source, the highly collimated nature becomes advantageous at pinhole sizes below ~30 μm for infrared microscopy.

Research using infrared radiation from synchrotron sources has seen steady growth over the past few years. Although the use of synchrotron radiation in this part of the spectrum started in the late eighties and is relatively new, it appears as one of the most promising applications. Several infrared synchrotron radiation facilities at NSLS and ALS are already available and the success of the infrared program at the NSLS has motivated the construction of an infrared beamline at TLS. In this report we present the design of our infrared beamline and its performance. As of this writing, the infrared beamline at NSRRC is open to outside users.

Our beamline is designed such that the infrared radiation from the bending magnet is coupled to a commercial Fourier-transform infrared spectrometer (FTIR) outfitted with an infrared microscope. The primary wavelength of the beamline will be in the mid-infrared region from 2 to 20 μm, where many materials have unique absorption features. This is also an important region for semiconductor defect identification.

This beamline collects 70 $\times$ 35 mrad of synchrotron radiation in the horizontal and vertical direction, respectively. The optical design consists of one water-cooled plane mirror, two high-order corrected polynomial bendable mirrors and a set of steering and collimating mirrors. For the consideration of vibration, the first mirror is 45 degree face-downward and its support is fixed to massive dipole magnet. In order to focus effectively the extended arc source of bending magnet, a special Kirkpatrick-Baez mirror system which uses two high-order polynomial mirrors has been designed and fabricated. The average brightness of this beamline is greater than 10^{16} photons/sec/0.1%bw/mm^2/str 200 mA. The synchrotron infrared emission at BL14A is compared to standard thermal source under different sizes of pinhole diameter and becomes advantageous for use of synchrotron radiation at pinhole sizes of approximately below 30 μm. In addition, the signal to noise ratio (SNR) of the synchrotron radiation in the mid infrared can be 300 to 500 times greater than that from a blackbody when small pinhole sizes below 10 um are measured.

The beamline optical layout has been designed by SHADOW ray tracing simulation. Two polynominal mirrors focuses the radiation on a wedged CVD diamond film window, and the beam is then collimated and steered on the entrance of an FTIR spectrometer and microscope experimental end station. The first mirror (M1) is a water-cooled extraction mirror of explosion bonded Glidcop/sst located at 1.22 meters from the source. This plane mirror with horizontal "slot" allows hard UV and X-rays to pass through without heating the mirror. The first plane mirror M1 was assembled with the mirror manipulator and then installed into the bending magnet chamber. The second and the third mirrors, M2 and M3, are proposed as bent cylinders, correcting for the extended/arc source characteristics. Only a few material are known to be capable of bending from plane shape approaching 50% of yield limit. M2 and M3 are made by superpolished 17-4 PH stainless steel. As compared with using only one elliptical focusing mirror, the focusing property is greatly improved from higher-order corrections, which directly benifits the performance of microscopy. The diamond window for this beamline is CVD diamond with 1 degree wedge to minimize interference fringes that result from multiple internal reflections. The diamond window was mounted into a double sided 35CF flange using UHV compatible diffusion-bonded technique. Our window vary from 750 μm to 1 mm (due to the wedge) in thickness such that a pressure difference of 3 atm can be safely sustained over 10 mm diameter. Immediately following the diamond window is a vacuum chamber for installing collimating and steering mirrors. The chamber is long enough to accommodate collimating mirrors with up to 500 mm focal lengths. Off-axis paraboloids having 25.45 mm and 500 mm focal length are available for matching two different collimated beam diameters to particular experimental setup when using various IR objectives.

As for the end-stations, the microscope system consists of a Nicolet Magna 860 FTIR spectrometer and a Continuum IR microscope. This beamline will primarily support microspectroscopy in the mid-infrared with samples of only a few micrometers in size, which should find a wide range of applications for the industries. The FTIR bench is capable of both rapid- and step-scan measurements. The IR microscope has several new features not found on normal IR microscope instrument. This IR microscope has a dichroic element which allows visualization of the sample even while acquiring data. This is especially convenient for a synchrotron source because the visible light spot indicates exactly where the beam is located on the sample. The optics in this microscope are infinity corrected which allows easy addition of several types of optics for assisting visualization of the sample, including visual and IR polarizers, Nomarski differential interference contrast (DIC) optics, and optional UV fluorescence with three difference filter cubes. Except for the UV fluorescence accessory, all of these optics are available at NSRRC IR Beamline 14A. This instrument is pictured in Fig. 1. This state-of-the-art IR microscope has been permanently installed at BL14A where we carried out commissioning of performance tests comparing the internal thermal IR source with the synchrotron source.

Since the signal-to-noise ratio is a crucial parameter for good measurements, we measured a

Fig. 1: Photograph of the Nicolet Continuum IR microscope (left) and Nicolet FTIR spectrometer of Magana 860 FTIR bench (right).

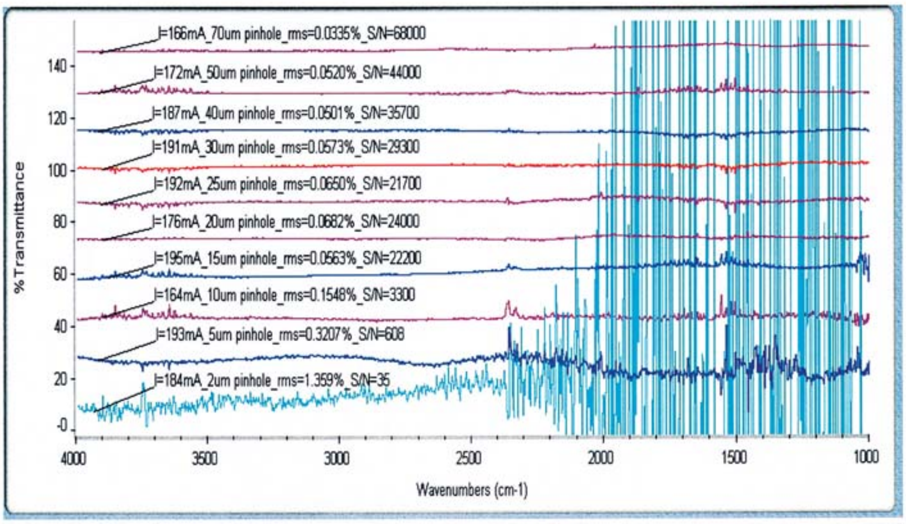

Fig. 2: 100% transmission lines showing the noise level of the NSRRC synchrotron IR source when using smaller and smaller pinhole diameters. Noted above each curve are the pinhole dimensions and the RMS noise determined between 2650 and 2750 cm^{-1}. The diffraction limited synchrotron spot size is not clipped until the pinhole diameter is below 15 microns, and even then it is primarily cutting off the longer wavelengths.

series of 100% transmission lines through various pinholes for both sources. We used an 250 μm^2 MCT-A mid-IR detector, co-added 128 scans for background and sample measurements, at a spectral resolution of 4 cm^{-1} and a scanning mirror velocity of 1.8988 cm/sec. Figure 2 shows the measured 100% lines for the synchrotron source. Each measured line is annotated with the pinhole size used and the resultant RMS noise value determined between 2650 and 2750 cm^{-1}.

Since the focused size of the thermal IR source is approximately 40 by 40 microns (from the mapping beam profile of thermal Globar IR light source), reducing the pinhole size simply decreases the total IR signal proportionally. The noise level becomes significantly worse as the pinhole size is decreased, to essentially unusable at aperture sizes below 20 by 20 microns. The focused spot size of the synchrotron source, however, is diffraction limited (3 to 10 microns in diameter), so its S/N ratio is only affected at pinhole sizes smaller than 15 microns (see Fig. 3 for the mapping beam profile of the NSRRC synchrotron IR light source). The synchrotron is observed to have a better S/N than the thermal IR source at the pinhole sizes below 30 $\times$30 microns. The noise doesn't start to increase until the aperture size reaches 10 $\times$10 microns, and a usable signal is maintained even at the smallest aperture sizes available, 5 $\times$5 microns. Note that the longer wavelengths (lower wavenumbers) are cut off by the smallest pinhole settings, as expected by the diffraction limited spot size (approximately the wavelength). The S/N value at 2650 cm^{-1} was obtained for each source and pinhole diameters by dividing the single beam intensity by the corresponding RMS noise value. The results are plotted in Fig. 4. The S/N ratio for the thermal EverGlo^{TM} source drops rapidly as the pinhole diameter size decreases, whereas for the synchrotron source the S/N ratio remains essentially unchanged until the pinhole size finally reaches the synchrotron beam spot size of 15 microns, as shown in the comparison in Fig. 4.

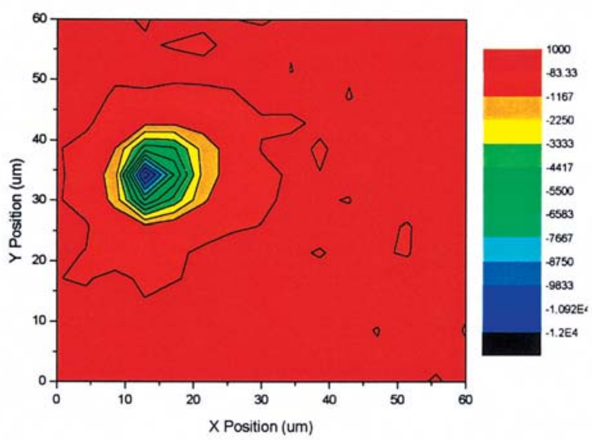

Fig. 3: Contour map for IR microscope spot size of the NSRRC synchrotron IR light source of 32X objective by mapping the beam profile with the 5 micron meter pinhole diameter in transmission. The beam size of the FWHM is around 10 by 13 microns .The step size of the mapping is set at 4 micron.

The synchrotron source's S/N ratio is 100 to 300 times better than the thermal source for pinhole sizes of 10 microns and smaller, which validates the calculated brightness advantage of a synchrotron IR beamline over a conventional thermal IR source for mid-infrared spectromicroscopy.

Infrared beamlines are known to be significantly more sensitive than X-ray beamlines to both photon beam motion and/or intensity modulations that leads to unwanted noise, and this IR beamline is no exception. In the following we presents some studies of the IR beamline noise. First we looked for noise modulations on a BPM signal. The spectrum of the horizontal beam signal in the control room near the 500 MHz harmonic shows sidebands similar to ones observed on the IR signal. These signals did not vary with quadrupole variations and were not observed on the sum or vertical BPM signals. This indicates the primary noise was on the electron beam rather than from beamline components. We then searched for noise sources in the RF and the magnet power supplies. The RF forward power spectrum has a central line at the RF frequency (499.998 MHz) and sidebands at 360 and 720 Hz, probably due to klystron power supply with continuous tails, and phase noise of the master oscillator. There was no apparent modulation sideband at the critical frequencies of 2.8 and 3.5 kHz. We also measured the shunt voltage monitor of almost all of the magnet power supplies. However, it was not possible to measure the IR noise with these supplies turned off since this badly distorted the electron beam orbit and would have required re-alignment of the beamline. Finally, we identify the noise source for the main 3.5 kHz and 4.1 kHz noise peaks arising from the power supplies of magnet corrector at U9 beamline, thus eliminating these two main noise peaks by the compensation circuit by Instrumentation Control group at NSRRC. We have made significant progress in understanding the noise at the IR beamline and we presently reach an S/N ratio in the range of 0.02% to 0.03% covering the mid-IR 1000 - 4000 cm^{-1}.

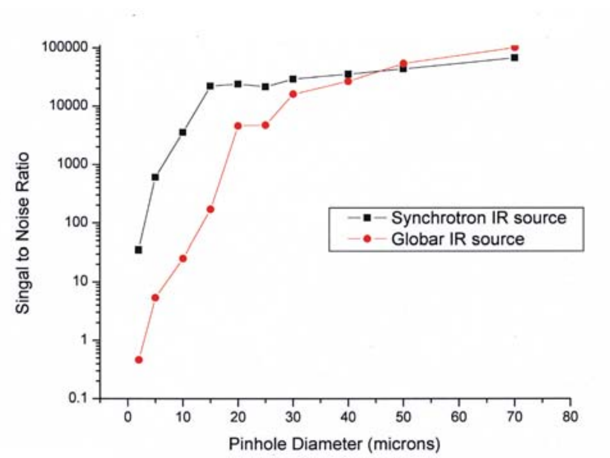

Fig. 4: Comparison of the measured signal to noise ratio (on a logarithmic scale) for the synchrotron and thermal EverGlo(tm) IR sources as a function of pinhole diameter size. We observe a greater than 100-300 times better signal to noise ratio for the synchrotron source compared to the thermal source for pinhole diameters 15 microns or smaller.

Authors

Y C. Lo, C. I. Chen, and C. H. Chang

National Synchrotron Radiation Research Center, Hsinchu, Taiwan

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Contact:

yclo@nsrrc.org.tw