Performance of the infrared microspectroscopy station at SSRF

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HIGHLIGHTS

• The first infrared beamline BL01B at SSRF has been constructed.
• A brief introduction of the infrared beamline design has been given.
• Synchrotron IR radiation provides better SNR than the internal globar source.
• The focused spot size reaches diffraction limit.
• This station has the ability of analysis samples in a small area with diffraction limited spatial resolution.

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ABSTRACT

At the third generation synchrotron light source Shanghai Synchrotron Radiation Facility (SSRF), the first infrared beamline BL01B has been successfully constructed. The infrared beamline collects both bending magnet and edge radiation. A brief introduction of the infrared beamline design has been given in this article. The infrared microspectroscopy station is equipped with a Nicolet 6700 FTIR spectrometer and a Nicolet Continuum Microscope. The flux at the entrance of the FTIR spectrometer, the intensity profile, the signal to noise ratio (SNR) with different apertures, and the focused spot size of the infrared microspectroscopy station have been measured. The performances with synchrotron radiation infrared source and internal globar source have been compared. The results indicate that the infrared microspectroscopy station at SSRF has the ability of analysis samples in a small area with diffraction limited spatial resolution.

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1. Introduction

Synchrotron infrared microspectroscopy (SIRM) is a powerful analytical technology. The synchrotron IR has 100–1000 times higher brightness than a conventional thermal globar source [1]. Due to its high brilliance and small divergence angle, Infrared Synchrotron radiation is easy to achieve the diffraction-limited spatial resolution with high SNR which is beyond the capability of conventional IR source. These characteristics are especially suitable for microspectroscopic investigations of small size samples and large samples with heterogeneous regions in a wide variety of fields. In the past twenty years, SIRM has been greatly developed. Most of the synchrotron light sources have infrared microspectroscopy stations, such as NSLS, ALS, CLS, SOLEIL, Diamond, ESRF, Australian Synchrotron, and Spring-8. Some sources even have more than one microspectroscopy stations, such as NSLS and ALS [2–9].

The vibration rotation bands of large molecules, such as protein, nucleic acid, carbohydrate, lipid, and biological membrane, the phonon spectra and electron energy spectra of crystalline and amorphous solids are all in the mid and far infrared domain. Based on the analysis of these spectra we can achieve the composition, structure and properties of the material, and can further study the relevant physical phenomena, catalysis process, biochemical reaction and mechanism of life. The SIRM technology plays an important role in the studies of physics [10], chemistry [11], materials [12], earth science [13], and biology [14] in the past twenty years for it is easy to achieve the diffraction-limited spatial resolution with high SNR. Taking biology for example, combining the microns spatial resolution with high SNR, SIRM technology is very useful in investigating individual biological cells, even living cells [14–16].

2. The beamline design and infrared microspectroscopy station at SSRF

Infrared beamline BL01B at SSRF has been successfully constructed. SSRF is a 3.5 GeV 3rd generation light source, the bending
magnet field is 1.272 T, and the bend radius is 9.16 m. This beamline is the first infrared beamline at 3rd generation light source in the Mainland China. The infrared beamline collects both bending magnet and edge radiation. The corrector constricts the horizontal angle to 40 mrad (~15 mrad to 25 mrad), and the dipole magnet vacuum chamber constricts the vertical angle to 20 mrad (~10 mrad to 10 mrad). The optical schematic diagram of BL01B is shown in Fig. 1. The extraction mirror M1 is placed at 1815 mm downstream to source point. It reflects the light horizontally with the incidence angle 45°. This mirror has a 2.6 mm central slot to avoid the damage of heat load from the intense X-ray and UV beam, and also to separate the Visible/Infrared from X-rays/UV. A second flat mirror M2 is situated 650 mm downstream to M1. It deflects the light vertically with the incidence angle 45° to the focusing mirror T1. It is adjustable in two rotations (pitch and roll) and one translation (z), which can partly compensate the extraction mirror’s unadjustability. T1 is a toroidal mirror and it is placed 1000 mm downstream to M2. It focuses the light out of the shield wall. The magnification of T1 is 1:1, which can achieve the smaller aberration for this toroidal mirror. The focus is near the CVD diamond window, which is out of shield wall. The diamond window with 1° wedged angle isolates the ultra high vacuum (UHV) and high vacuum (HV). Behind the diamond window, there is an adjustable slit. T2 is also a toroidal mirror used to collimate the light. The active feedback system includes two plane mirrors (M3, M4), three beam splitters (BS1, BS2, BS3) and two position sensitive detectors (PSD1, PSD2). Nowadays, the feedback system is not inevitable to the 3rd generation light source, especially for IR microspectroscopy. The BS2 also acts as an endstation switcher, switching the IR beam between two timesharing endstations, Time-Resolved Spectroscopy Station and Microspectroscopy Station. It can be easily changed to the beam sharing mode. More detailed beamline design of BL01B can be seen from our previous article [17].

The infrared microspectroscopy station is equipped with a Nicolet 6700 FTIR spectrometer and a Nicolet Continuum Microscope. Specifications of this station are shown in Table 1.

### 3. The performance of infrared microspectroscopy station

The photon flux of the different wavelength at the entrance of the spectrometer has been simulated using SRW. The size of various optical elements and vacuum tube, slotted mirror and the source widening caused by diffraction in optical elements are taken into account. The reflectivities of these mirrors are both ~98%. The transmission efficiency of CVD diamond window is ~65%. Two flat mirrors are used instead of BS1 and BS3 in the calculation and measurement. The calculated value of photon flux is 2.0 × 10^{13} (photons/sec/0.1% b.w.) at 1 μm wavelength for 230 mA current in the synchrotron radiation ring. Using the method similar to SOLEIL’s [18], the absolute flux at the entrance of the FTIR spectrometer has been measured using a calibrated Si-diode (FD5010-10 from Thorlabs, Inc.) combined with a 1.0 μm optical filter (100FS10-50 from Andover Corporation). The set-up is shown in Fig. 2, the collimated beam passes the filter and has been focused on the photodiode by the parabolic mirror, the photocurrent data has been measured by the picometers. The total output current of photodiode is:

\[
I_{\text{sum}} = \int_{-\Delta f/2}^{\Delta f/2} n(\lambda) T_f(\lambda) R(\lambda) \frac{hc}{\lambda} d\lambda + I_{\text{DK}}
\]

where \(n(\lambda)\) is the acquired photons per second per nanometer, \(T_f(\lambda)\) is the transmittance against wavelength of the filter, \(R(\lambda)\) is spectrum response against wavelength of the photodiode, \(I_{\text{DK}}\) is dark current of photodiode, \(BW_{\text{filter}}\) is the bandwidth of the filter. The photon flux is defined as the acquired photons in 0.1% bandwidth per second, which can be expressed as:

\[
N = \int_{-\Delta f/2}^{\Delta f/2} n(\lambda) d\lambda
\]

The response curve for \(T_f(\lambda)\) and \(R(\lambda)\) are calibration parameters of the instrument. Assuming \(n(\lambda)\) is a constant in the integral range, the photon flux can be obtained in case of measuring the values of \(I_{\text{sum}}\) and \(I_{\text{DK}}\).

With this set-up, the flux at the entrance of the FTIR spectrometer has been achieved to be about 1.5 × 10^{13} (photons/sec/0.1% b.w.) at 1 μm wavelength for 230 mA current. It is lower than the calculated value.

The intensity profile of synchrotron source and the internal source (globar) have been measured. The measurements are performed by reflection on a gold mirror using Nicolet Continuum Microscope. The MCT-A detector and KBr beam splitter are used in the measurement. Fig. 3 shows the single beam signal obtained from the internal source (230 mA) and the internal source respectively. The scale for internal source is magnified by a factor of 35. The synchrotron radiation provides about 35 times higher intensity than the globar source.

The 100% reflectance lines have been acquired by microscope on the same position have been measured consecutively. For aperture size below 10 μm, synchrotron radiation provides one order of magnitude better RMS noise value than the globar source. Taking aperture size 5 × 5 μm² for

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Fig. 1. The optical schematic diagram of the BL01B beamline (the distribution profiles on M1 and T2 have been given at 10 μm wavelength by SRW simulation).
example, the RMS noise value of synchrotron IR source is 0.06%, and that of internal globar source is 1.04%. The synchrotron IR source is about 17 times better than the internal globar source in terms of RMS noise value.

The SNR at 2500 cm\(^{-1}\) is calculated by dividing the single beam intensity at 2500 cm\(^{-1}\) by the previous RMS noise value. The SNR is strongly affected by the aperture size when using the globar source, and it is quite stable for the synchrotron source with aperture sizes above 10 \(\times\) 10 \(\mu\)m\(^2\). Just as Fig. 5 shows, the SNR of synchrotron source is much better than the globar source. For the aperture size smaller than 15 \(\times\) 15 \(\mu\)m\(^2\), synchrotron radiation provides a two orders of magnitude better SNR than the internal globar source. Just as Fig. 5 shows, the SNR of synchrotron source is much better than the globar source. For the aperture size smaller than 15 \(\times\) 15 \(\mu\)m\(^2\), synchrotron radiation provides a two orders of magnitude better SNR than the internal globar source.

The focused spot size of synchrotron IR beam has been measured with infrared microspectroscopy imaging. The microscope can achieve sample mapping by moving the sample stage with 2 \(\mu\)m per step under the focused IR beam with 32 \(\times\), NA = 0.65 infrared objective 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 source, a 5 \(\mu\)m pin hole is used and the transmitted spectra as a function of the pin hole position has been measured. There are no other beam-defining apertures in the optical path.

The intensity at 1000 cm\(^{-1}\) as a function of pin hole position is shown in Fig. 6. A small spot is produced using the synchrotron IR source. The x and y profiles of this spot along with intensity is plotted in Fig. 7 (at 1000 cm\(^{-1}\)). The focused spot diameter 17.7 \(\mu\)m can be achieved (for a 32 \(\times\), NA = 0.65 objective). Theoretically, the diffraction limit will be smaller than the Airy disk when there is central obscuration [19]. Carr researched this and proved it [20]. For our station the measured value is also smaller than the Airy disk, but the difference is not so big. And the actual measured values by ALS and CLS are all close to the calculated value by the formula “diffraction limit = 1.22\(\lambda/NA\) [21–23]. So we take Airy disk to estimate the diffraction limit.

The diffraction limit of this system at 1000 cm\(^{-1}\) (\(\lambda = 10 \mu\)m) is about 18.8 \(\mu\)m as the numerical aperture of continuum 32 \(\times\) infrared objective is 0.65. The focused spot size reaches diffraction-limited. As a comparison, Fig. 6(a) also shows the result using internal source. It can be seen that the focused spot size using SR source are much smaller than using internal source. This performance allows the infrared microspectroscopy station at SSRF possessing the
ability of analysis samples in a small area with a diffraction limited spatial resolution. It is especially suitable for analysis small samples and large samples with heterogeneous regions in a wide variety of fields.

4. Summary

The first infrared beamline BL01B at the third generation synchrotron light source Shanghai Synchrotron Radiation Facility (SSRF) has been successfully constructed. The infrared beamline collects both bending magnet and edge radiation, and a brief introduction of the infrared beamline design has been given in this article. The beamline has two end stations, one is applied to time-resolved infrared spectroscopy, the other is applied to infrared microspectroscopy. The performances of the infrared microspectroscopy station at SSRF have been measured. The flux at the entrance of the FTIR spectrometer has been achieved to be about $1.5 \times 10^{13}$ (photons/sec/0.1% b.w.) at 1 μm wavelength for a 230 mA current. The intensity profile, the SNR with different apertures, and the focused spot size of the infrared microspectroscopy station have been measured. The performances with synchrotron radiation infrared source and internal globar source have been compared. Synchrotron radiation provides about 35 times better intensity value than the globar source for $8 \times 8 \mu m^2$ aperture size. And for aperture size below $15 \times 15 \mu m^2$, synchrotron radiation provides a two orders of magnitude better SNR than the internal globar source. The focused spot diameter at sample position of microscope is measured to be 17.7 μm at 1000 cm$^{-1}$ with synchrotron radiation infrared source. These performances allow the infrared microspectroscopy station at SSRF has the ability of analysis samples in a small area with a diffraction limited spatial resolution. It is especially suitable for analysis small samples and large samples with heterogeneous regions from a wide variety of fields.

Conflict of interest

There is no conflict of interest.

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