Design, Construction and Performance of the Infrared Microscopy Beamline (BL-15) in the SR center

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Abstract

The JST project to construct a new infrared microscopy beamline at BL-15 in the SR center was started at the 2007 fiscal year as a three-year program and ended in March, 2010. The basic idea of the beamline is to install several mirrors in the chamber of the storage ring to accept the infrared part of synchrotron radiation with 250 mrad ^{H} x 50 mrad ^{V} angles, then provide it to the commercial FT-IR microscopy system. The system was completed and several performance tests have been done in comparison with the laboratory light source. Although the total IR power of the synchrotron radiation is inferior to the laboratory light source, it is superior in the brilliance and more appropriate for the microscopy. Several tests demonstrate the advantages of the synchrotron IR microscopy.

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１．Introduction

Infrared absorption spectroscopy has been widely used as a powerful analytical tool to characterize unknown materials. Now, due to rapid development of nanoscience and nanotechnology, a local analysis is strongly demanded, which accelerates the development of the infrared microscopy. For the infrared microscopy, a brilliant light source is necessary. However, conventional infrared light source, such as a ceramic heater has an intrinsic limitation. Synchrotron radiation is known to be a very powerful light source covering a wide energy range, especially promising in the X-ray region. It is also promising in a very low energy region, terahertz region, where there is no good light source.

In the infrared region, SR is not as powerful as conventional light sources, but is highly brilliant and is a promising light source for the infrared microscopy.

Use of synchrotron radiation for the light source of infrared spectroscopy, especially of infrared microscopy started in 1980's by G. P. Williams[1] at NSLS in Brookhaven National Laboratory. Stimulated with great success in its application to biological materials, several synchrotron facilities in the world started constructing the infrared beamlines. In Japan, Namba and Kimura constructed an IR beamline in UVSOR in 1990 (upgraded in 2006), and then in Spring-8 in 2002[2,3].

AURORA is the smallest synchrotron storage ring in the world, in which the electron is running circularly with a diameter of 1 m and the energy of 0.57 GeV. This was designed for X-ray lithography, and soft X-ray spectroscopy, but it can be used as a light source in the infrared region.

We designed an optical system to collect as much as IR photons from the ring, making use of the special feature of AURORA, completely circular orbit. The proposal to construct an infrared microscopy beamline for nanoscience and bioscience was approved as one of the JST innovation program for local district. The project started in the fiscal year 2007 as the three year program and ended in March, 2010. This is the report of this project which describes the conceptual design, construction, and performance tests.

2. Conceptual design

The performance of an infrared beamline is not dependent on the ring energy or ring size of a storage ring, but on the beam current times acceptance angle of the beam. In case of AURORA, the acceptance angle of the SR beam port is limited to 30 mrad^H x 30 mrad^V, However, insertion of mirrors to focus the light source point makes us possible to increase the acceptance angle substantially, as schematically shown in Fig. 1. This is the idea similar to Prof. Yamada for MIRRORCLE. Since the reflectance in the infrared region is almost 100 %, multiple mirror system will provide us a very intense infrared beamline. This is the original design in the project.

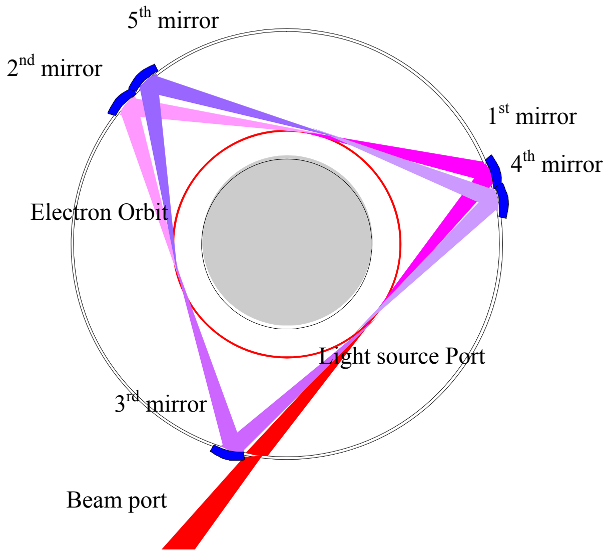


Fig.1 Schematic drawing of the multi-mirror system. By installing the 1sr mirror, the beam intensity becomes double and further installation of mirrors at appropriate positions increase by the number of mirrors.

In April, 2007, we had a chance to open the vacuum chamber of the storage ring and found it difficult to insert many mirrors surrounding the electron orbit because there are so many obstacles inside the chamber to hamper the beam path. As a result of it, we have to change the optical design completely. Final design is shown in Fig.2, in which the optical system consists of one toroidal mirror and two plane mirrors.

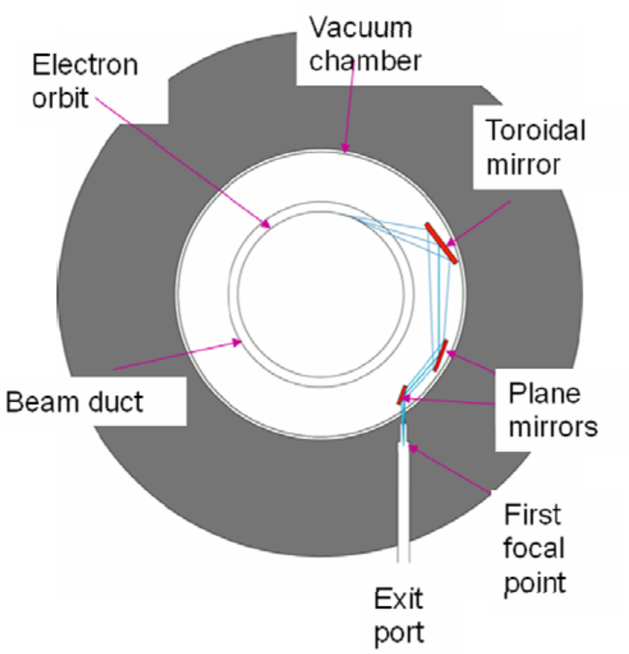


Fig.2 Optical system inside the ring chamber, consisting of one toroidal mirror (Au coated Si, 260 mm ^{L} x 60 mm ^{W}) and two plane mirrors.(Au coated aluminum, 200 mm ^{L}x 23 mm^{W} and 100 mm^{L} x 12 mm^{W})

Different from conventional light sources, the light source of synchrotron radiation is electrons running in the ring. To focus the radiation from the light in a circular motion to one point, so-called 'magic mirror' is used [4]. But, the cost and delivery time of a magic mirror were serious problem in our case. By the ray-tracing simulation, we found that drastic improvement of the optics cannot be expected by using a magic mirror and decided to use a toroidal mirror. This comes from the high emittance of AURORA, different from Spring-8. In fact, the ray tracing predicts a similar beam size at the focal point. Figure 3 shows the ray tracing results of beam images at the focal point at 0.01 eV(ca. 100 cm^{-1}) and 0.1 eV (1000 cm^{-1}).

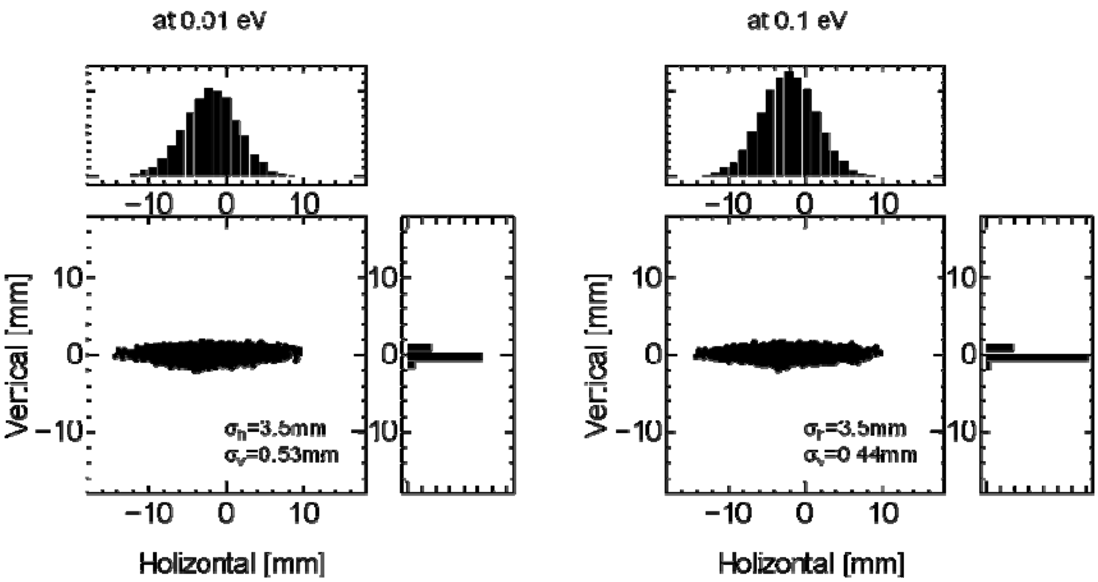


Fig. 3 Beam shape at the first focal point at 0.01 and 0.1 eV.

3. Experimental set-up

3.1 Installation of the mirrors in the chamber

Installing these three mirrors in the ring chamber was really a tough work, described as follows.

(1) Since there are many parts indispensable for the storage ring in the chamber, allowed space for the mirrors were limited.

(2) Cooling is necessary, or protection of irradiation of the X-ray beam (central parts emitted from the electron orbit plane).

(3) Remote control system for fine adjustment mechanism is necessary for the toroidal mirror.

(4) Since this is inside the UHV chamber, degassing from the components installed is strictly prohibited.

(5) All the components installed are non-magnetic, since the chamber is in the superconducting magnet.

(6) Once the optical system is installed, we cannot change or modify the system since reopening the chamber costs fortune and takes at least one week.

As for (2), we adopted the protection of irradiation by using a thin water cooling SUS pipe (6 mm in diameter) in front of the mirrors, since water cooling from the back side causes serious vibration of mirrors. This does not cause the IR photon intensity loss because the radiation of a longer wavelength is more dispersed vertically as shown in Fig. 4.

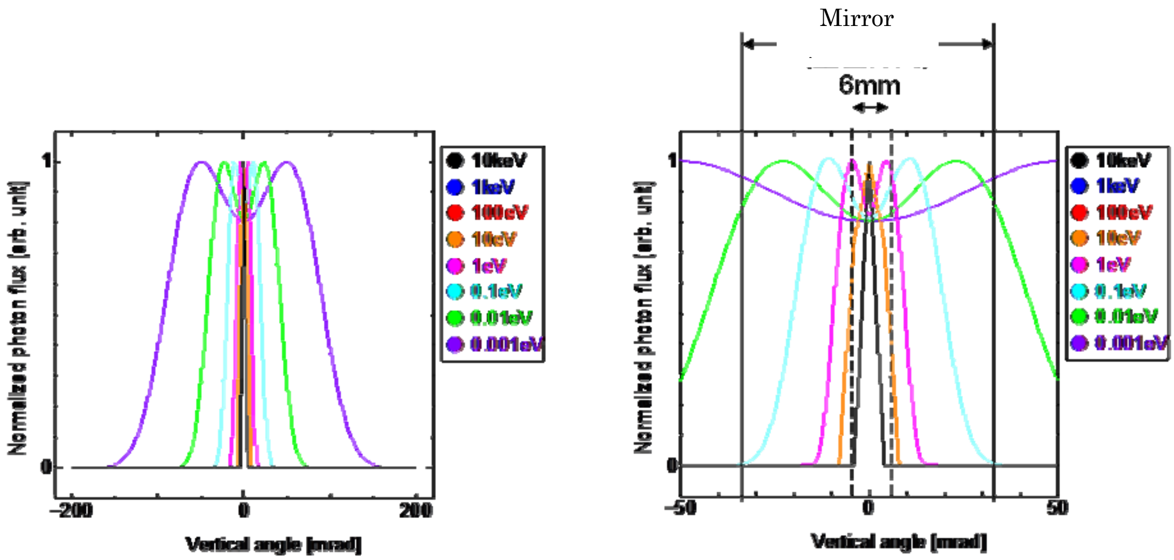


Fig.4 (a) Vertical photon flux distributions of several photon energies, and (b) Effect of blocking pipe on the photon flux.

Figure 5 shows a schematic figure of the fine adjustment mechanism of the toroidal mirror with a cooling pipe. Two non magnetic picomotors are used to adjust yoke and pitch adjustments.

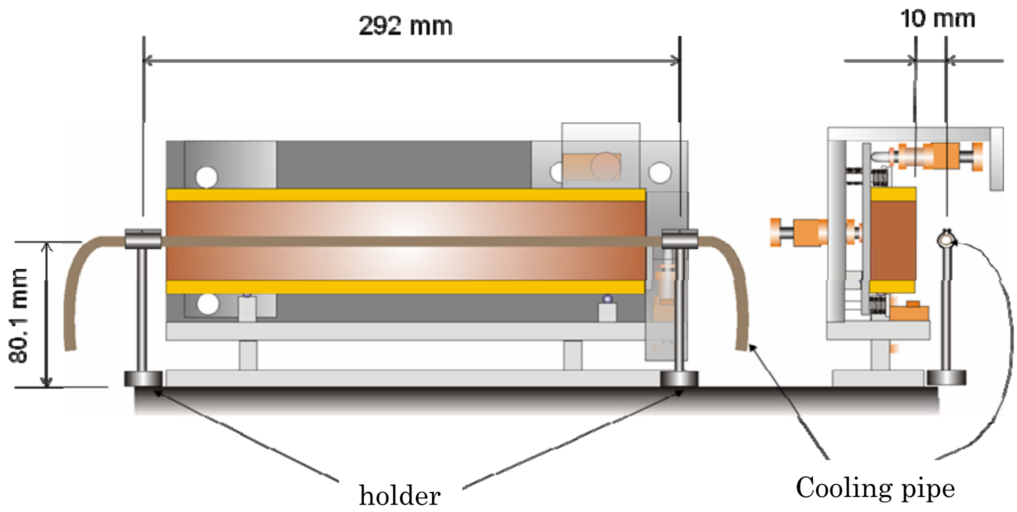


Fig. 5 Schematic drawing of the fine adjustment mechanism for the toroidal mirror.

Cooling pipe is just one pipe from one port, setting to block three mirrors in the precision of ±0.1 mm, deflecting at other parts so as not to hamper the SR beam to other beamlines. Figure 6 shows the installed mirrors and cooling pipe.

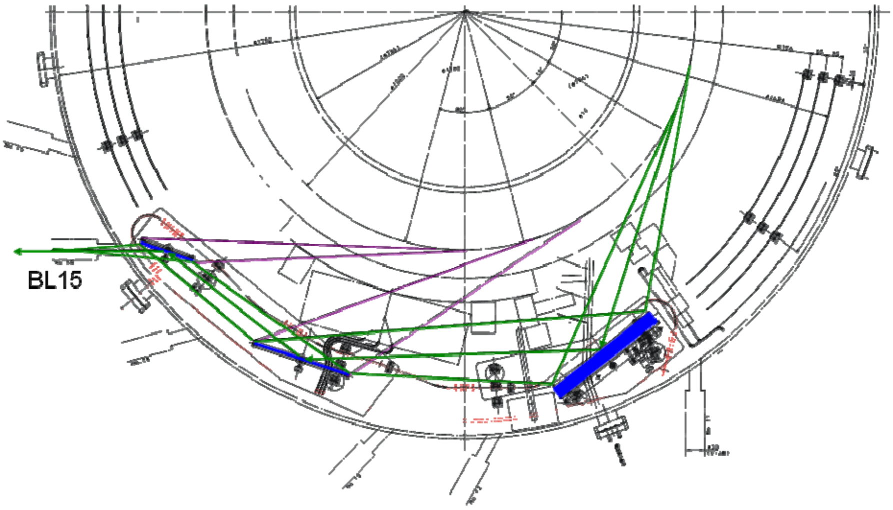


Fig. 6 Three mirrors (blue color) and one cooling pipe(pink color) installed in the ring chamber

3.2 Front-end of the IR beamline

The optical system and the front end of the IR beamline is shown in Fig. 7, where the first focal point is still in a pipe of the ring. Another toroidal mirror is set in the frontend chamber

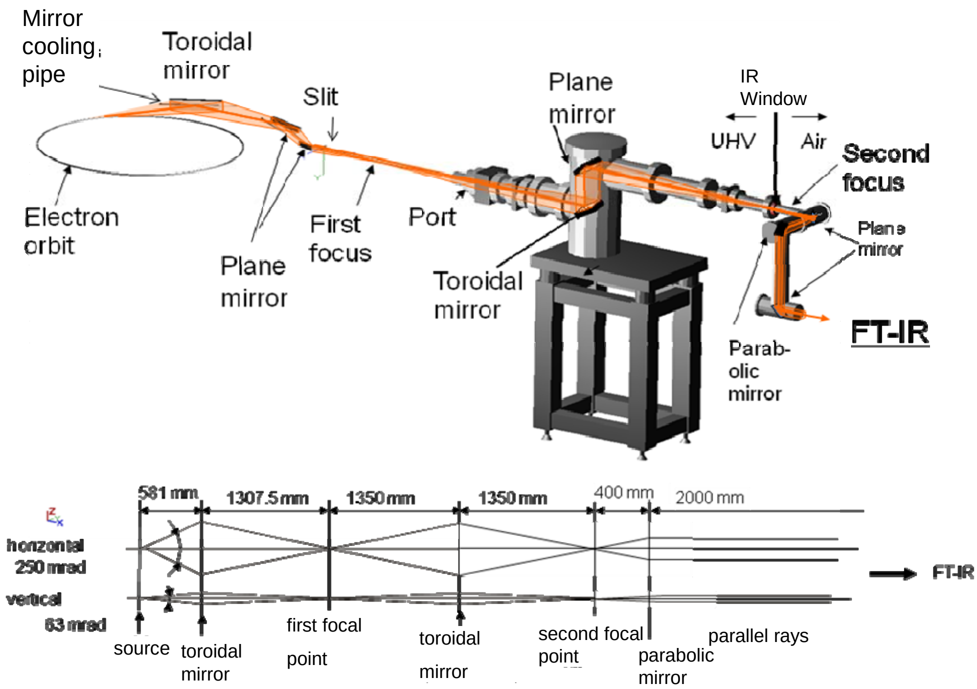


Fig. 7 The optics of the IR beamline and the schematic view of the frontend of the beamline.

so as to focus at the second focal point in air through a KRS5 window. Dispersed beam is converted to a parallel bam with a parabolic mirror and then introduced in a commercial FT-IR apparatus, Nicolet6700+Continuum (Thermo Ltd.), as shown in Fig. 8.

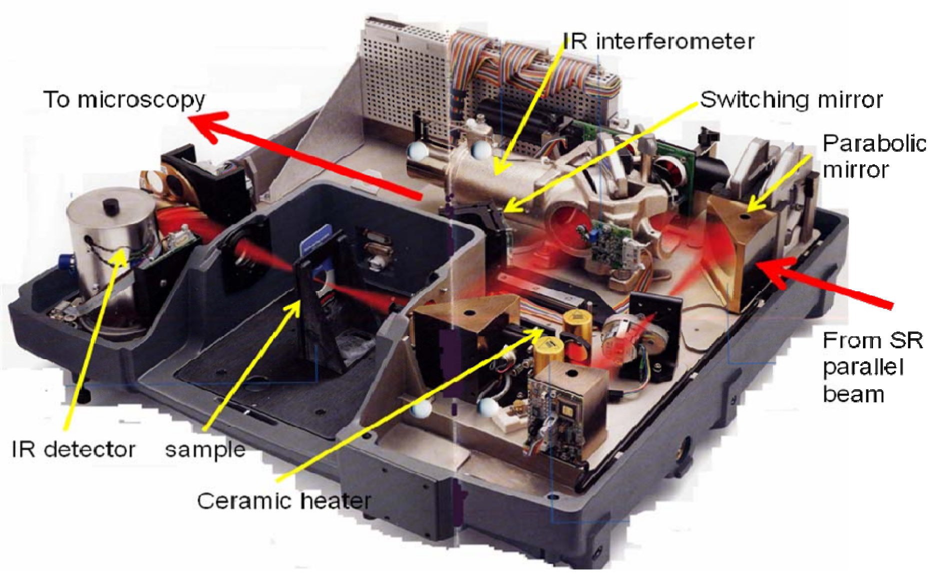
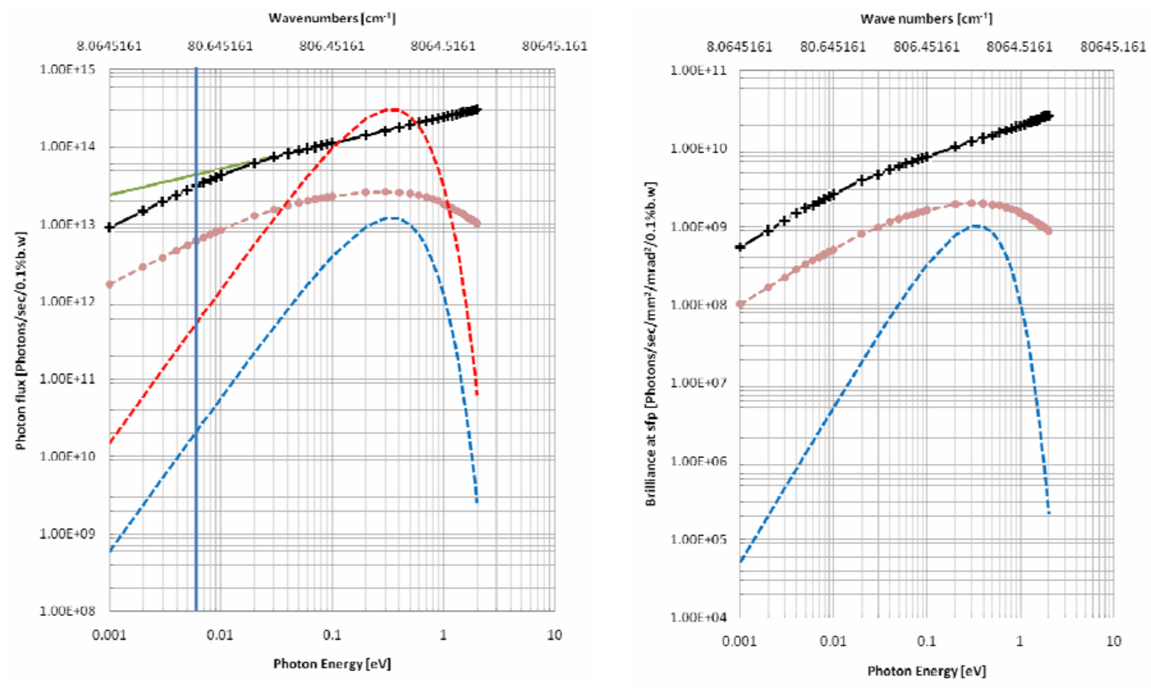


Fig. 8. Photo of Nicolet 6700. By making a hole in the side panel and removing the parabolic mirror, the SR parallel beam can be introduced in the FT-IR apparatus.

4. Evaluation of the SR IR microscopy

Maximum photon flux and brilliance of the SR infrared beam can be calculated theoretically from the electron energy, ring current and accepted horizontal and vertical angles. The results are shown in Fig. 9 (a) and (b). There are also several causes to reduce the photon flux and brilliance, such as some obstacles in the ring chamber and a water cooling pipe. Thus, the effective maximum available photon flux and brilliance are also shown in the figure, together with those of the black body source.

As shown in Fig. 9, the intensity is reduced by one order of magnitude by several obstacles. Compared with the black body source, the photon flux of the SR is lower in the mid-IR region, although it is higher in the far infrared region. In contrast, the brilliance if the SR is superior to the black body source all over the region. This suggests that the infrared microscopy with the SR is promising even though there is serious loss of intensity by the obstacles in the ring chamber. However, it should be noted that this is the maximum value with an ideal optical system. In reality, the SR electron beam size is fairly large (σ_H=1.4 mm, and σ_V=0.14mm) and the use of toroidal mirrors is not perfect. Fig. 10 shows the interferogram from the SR infrared beam, compared with that of the laboratory source. The intensity of the SR is slightly higher at the mid infrared region, suggesting further improvement of optical alignment is necessary.



(a) Photon flux (b) Brilliance

Fig.9 (a) Photon energy distribution of the photon fluxes. Black: SR infrared beam from the accepted angles at 300 mA, purple: effective photon flux of the SR infrared beam, red: photon flux from a black body source of 5 mmϕ at 1400 K, 0.02 sr, blue: that from a black body source of 1 mmϕ. (b) Photon energy distribution of the brilliance. Black, purple and blue lines are same as fig.(a).

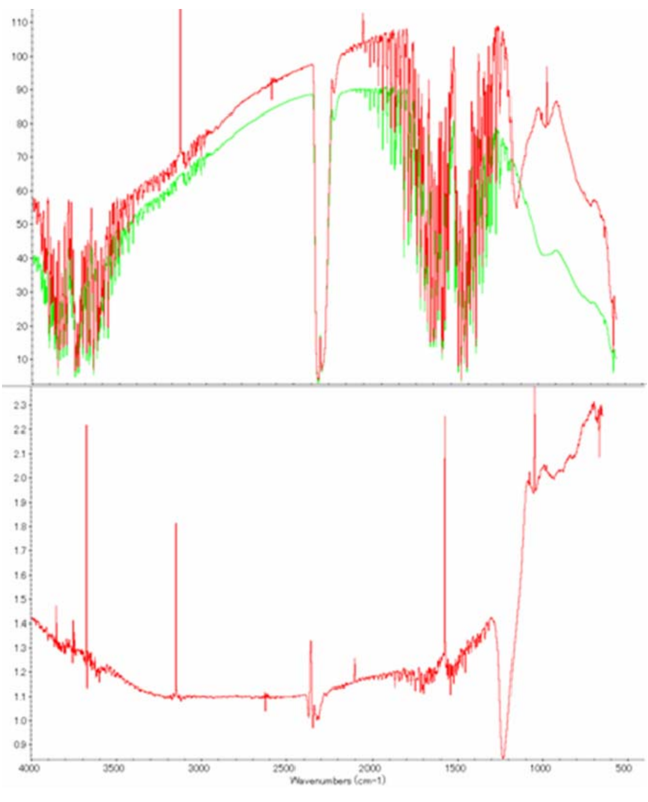


Fig. 10. Upper figure: Interferogram of the SR infrared beam (red line), compared with that of the black body source (green line)

Lower figure: the intensity ratio of the SR infrared beam by the black body source. Absorption peaks at 3200 and 1600 cm^{-1} are from water. Spike noises appear in the SR infrared beam.

To confirm the advantage of brilliance of the SR, we observed images of a 5 μmϕ pinhole at several wave numbers, and compared with those by the laboratory source. The results are shown in Fig. 11.

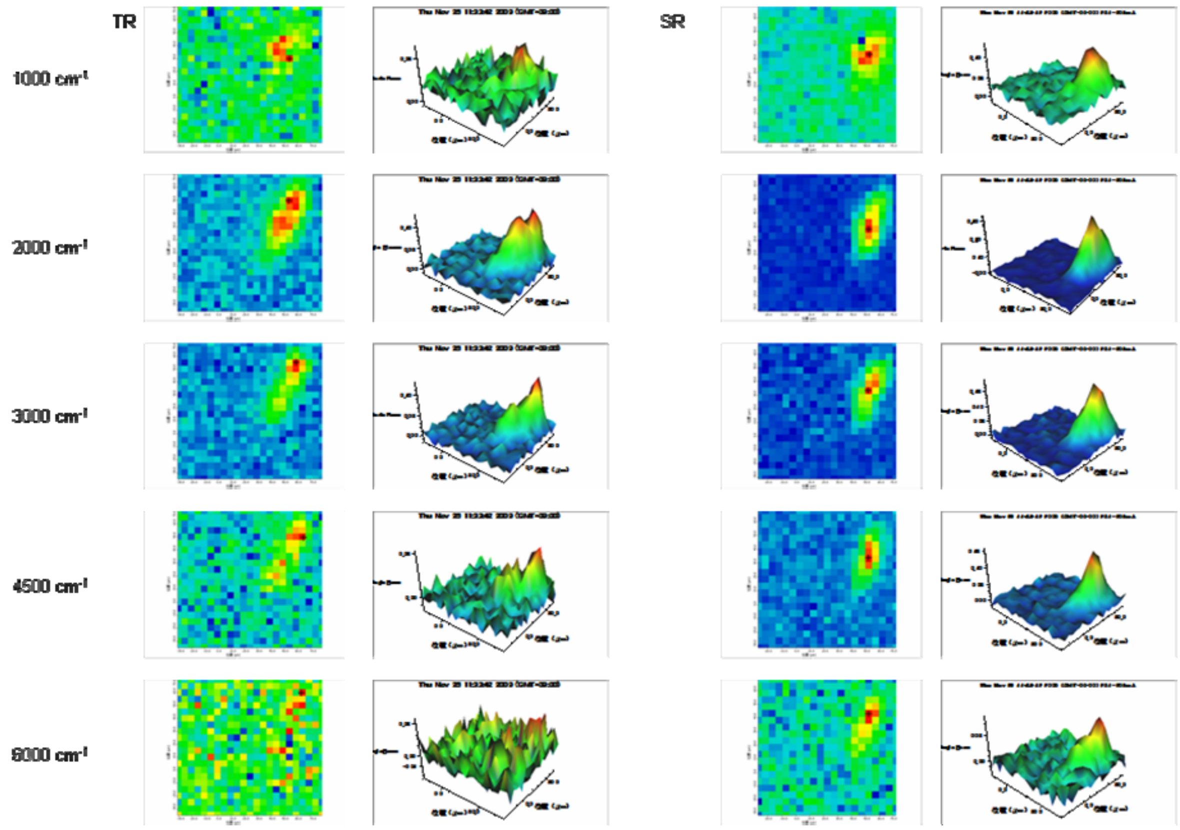


Fig. 11 Images of a 5 μmϕ pinhole at several wave numbers. Left figures are those by the TR (thermal radiation) and right figures are those by the SR IR.

It clearly demonstrates the superiority of the SR IR to the laboratory source. At 2000 cm^{-1}, double peaks appear in the laboratory source, while only single peak appears in the SR IR with more suppressed background. Table 1 indicates the comparison of IR intensity of the SR and black body thermal radiation (TR) source at several wave numbers.

Table 1 Comparison of the IR intensity of the SR and TR for a 5 μmϕ pinhole

Wave number	SR	TR	SR/TR
1000	0.14	0.064	2.18 (1.8)
2000	0.32	0.130	2.46 (1.6)
3000	0.19	0.128	1.58 (2.1)
4500	0.16	0.053	3.01 (4.6)
6000	0.08	0.034	2.35 (10.7)

Values in parentheses are from a 10 μmϕ pinhole.

We measured the IR microscopic spectra of 2μmϕ polystyrene(PS) latex dispersed on mica, with the transmission mode, using ×15 cassegrain optics. Obtained spectra are shown in Fig. 12, where the spectra were accumulated by 256 times. With the SR IR, two characteristic peaks at 2920 and 3020cm^{-1} are clearly observed, while they are hidden in the noise with TR. This experiment also demonstrates the usefulness of the SR IR microscopy.

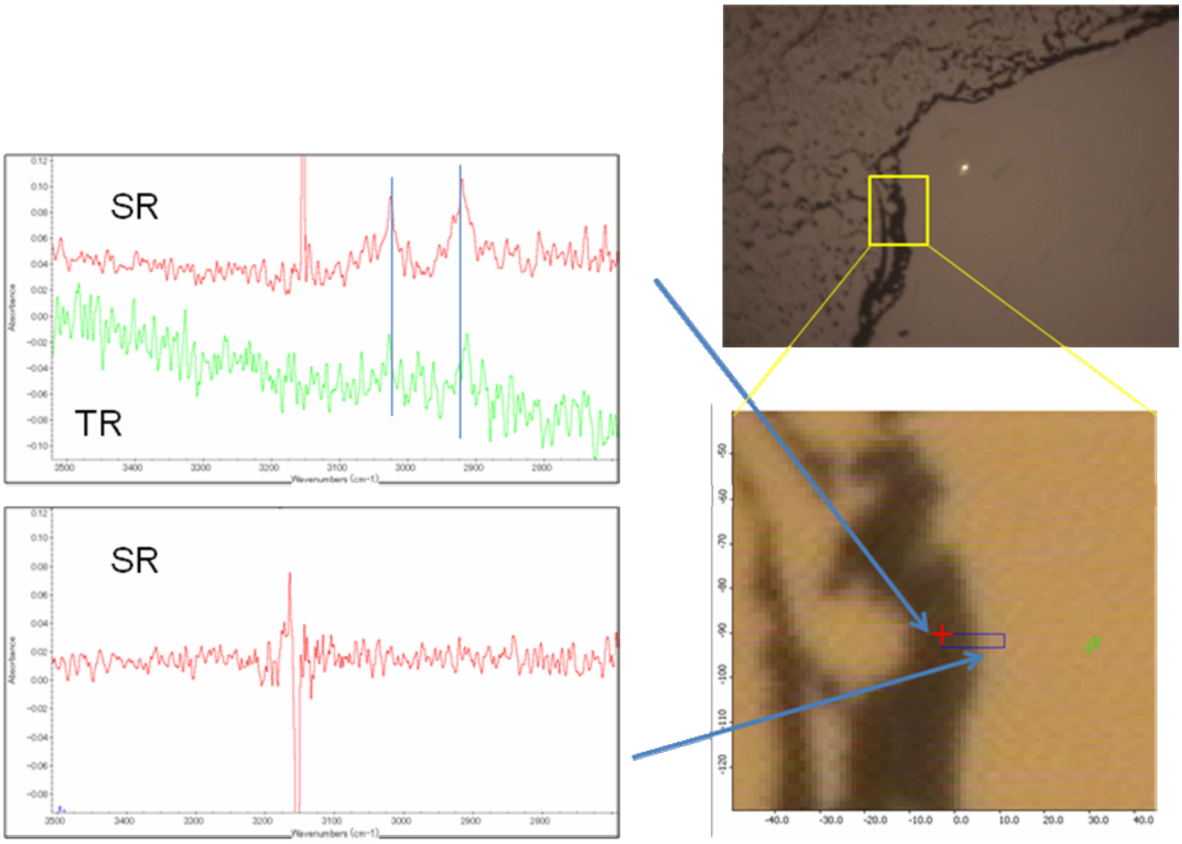


Fig. 12 IR microscopic spectra from 2μmϕ polystyrene(PS) latex dispersed on mica. Right upper figure is a photo of optical microscopy. IR spectra from PS and from mica were shown in the left figures. Two peaks at 2920 and 3020cm^{-1} are characteristic of PS.

5. Conclusion

An IR microscopy beamline was designed and constructed as a three years project of the JST innovation program. Although the project did not go well as expected in the designing stage, we could demonstrate the usefulness of the synchrotron radiation based IR microscopy, compared with the laboratory light sources. Performance tests are still in a preliminary stage and further applications, especially in the near infrared and far infrared regions are necessary to reveal the characteristics of the IR beamline. We are also trying further optical improvements, aiming to obtain the full performance of the present microscopy beamline.

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