

Application Note AN M92

Vacuum FT-IR Beamline Installations at e-Synchrotrons

In the 1970's attempts to demonstrate the advantages of the brilliant and highly collimated e-synchrotron radiation (SR) not only in the short wavelength (UV, X-Ray) but also in the long wavelength ranges (typically 4000 to 10cm⁻¹) of the electromagnetic spectrum were unsuccessful. The theoretically expected advantage of the synchrotron radiation as a bright but expensive IR source could not be demonstrated.

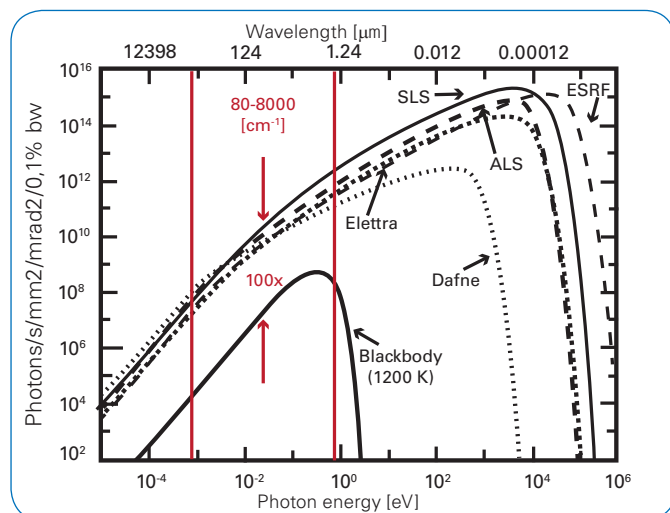


Fig. 1: Advantages of the e-Synchrotron radiation source

Tab. 1 Synchrotron Radiation Features

- Small measurement spot: ca. 500 μm diameter
- Low divergence of less than 50mrad
- Highest brilliance broad band radiation source
- Almost constant intensity versus wavelength
- Diffraction limited in the far IR/THz
- Highly linear polarized light

Tab.1: Dedicated advantages of the synchrotron radiation compared to a conventional thermal radiation sources

Adaptation and Installations

Today the system parameters for generating radiation using e-synchrotrons, as well as for the optics design for guiding the synchrotron radiation via an IR beamline to an infrared spectrometer are known (see fig.2). Successful experiments providing new insights have led to the adoption of synchrotron radiation as a brilliant radiation source (see fig.1 and tab.1) for spectrometers and consequently an increase in the number of IR beamlines with spectrometers attached. Fig. 3 shows the currently installed Bruker FT-IR spectrometers and in the publications Ref. 1 and Ref. 2 detailed explanation of recent synchrotron advantages in particular for the coherent synchrotron radiation (CSR) are described.

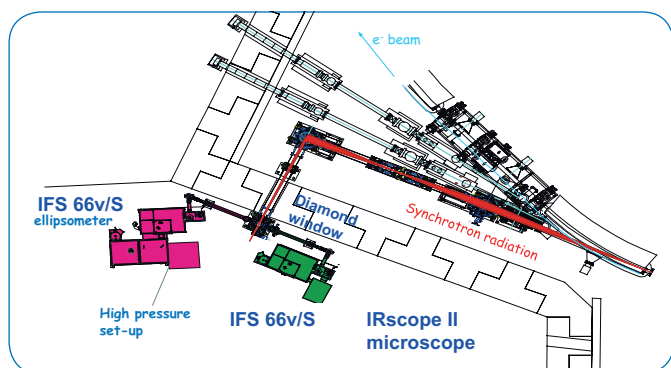


Fig. 2: SR IR beamline transfer optics for two FT-IR spectrometers (ANKA, KIT, Karlsruhe, Germany)

Dedicated Instrumentation Options

For demanding SR applications dedicated FT-IR spectrometers, options and accessory modifications are available. Preferably instruments with a vacuum optics bench are being used, which allow extremely sensitive measurements due to the absence of traces of absorptions caused by the constituents of dried air or nitrogen. Desirable accessories and options include the option to exchange beamsplitters via an automatic beamsplitter exchange unit (BMS-c), which allows switching of the spectral ranges without breaking the vacuum of the spectrometer and dedicated SR input beam vacuum chambers with additional external automatic aperture changer. Additional functionality is achieved by synchronizing the data acquisition of the spectrometer with the status of the beam line (see tab. 2). For IR-spectroscopy with an IR microscope a specially designed condenser with precise X-Y-Z adjustment and a cold LED sample illumination for excellent long term stability are used.

Tab. 2 Options for IR Beamlines

- Automatic beamsplitter exchange BMS-c unit which allows the extension of the spectral ranges without a break of spectrometer optics vacuum
- Dedicated SR input beam vacuum chamber with additional external aperture wheel
- Synchrotron real time “Stop-Start” trigger functionality synchronized with machine TTL signal
- Dedicated high stability IR microscope condenser with precise X-Y-Z adjustment
- Cold LED sample illumination and modified power supply for the HYPERION IR microscope for improved long term stability

Tab. 2: VERTEX vacuum FT-IR spectrometer options for optimum use at IR beamlines



Fig. 3: World-Wide Bruker e-Synchrotron IR beamline installations

A permanently sealed liquid N₂ cooled MCT detector (“PermaVac”) is available which provides spectra free of ice absorbance bands at 3500 cm⁻¹.

Tab. 3 Significant FT-IR Vacuum Advantages

- Free of moisture absorptions which may mask weak spectral features
- High spectral resolution features do not interfere with residual CO₂ and H₂O absorptions
- Very stable optics bench due to cast aluminum housing (weight > 100 kp)
- Rapid optics bench evacuation by high pumping speed
- No sample contamination due to use of oil-free vacuum pump
- No problems caused by dry air/nitrogen supply instabilities
- No uncompensated H₂O vapour lines caused by purge gas temperature variations

Tab. 3: Vacuum advantages of the VERTEX 80v FT-IR spectrometer

FT-IR Vacuum Advantages

For the typically very demanding research applications using synchrotron radiation a spectrometer with a vacuum optics bench is of high benefit. The IR spectra are free of moisture absorptions from the room air which may mask weak spectral features, and high spectrally resolved absorption bands do not interfere with residual CO₂ and H₂O absorptions. The solid aluminum housing with a weight of more than 100kp is a very stable optics bench which leads to superior instrument stability. The optics bench is quickly evacuated by the high pumping speed of the oil-free vacuum pump, which avoids effectively sample contamination (which might happen when using oil lubricated pumps). Instabilities caused occasionally by variations of the temperature and residual water vapor are effectively avoided (see tab. 3).

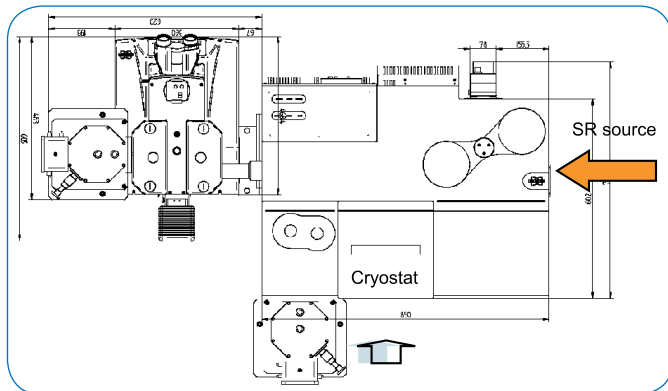


Fig. 5a: IR beamline configuration example: VERTEX 80v, HYPERION 2000 & bolometer detector.

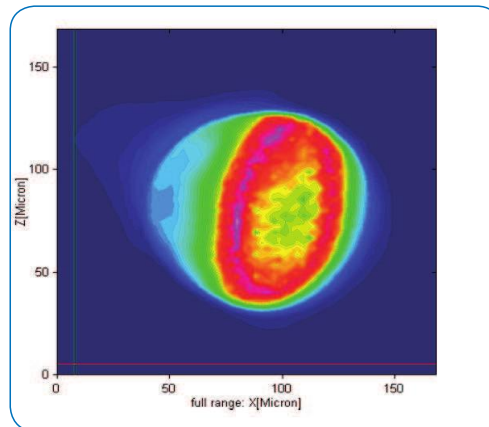


Fig. 6: XYZ (upper) and XZ (lower) contour plots using FPA detector

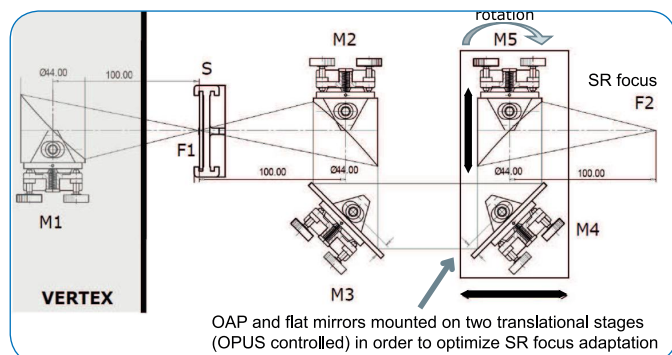
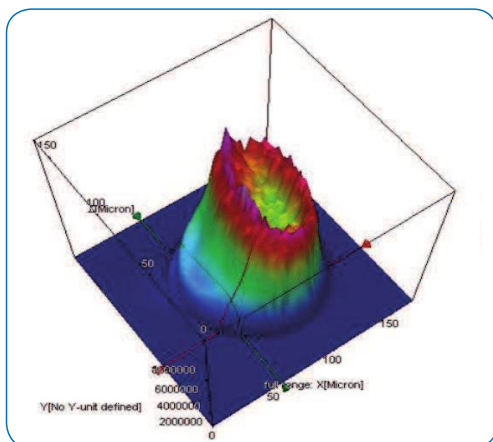


Fig 5b: IR beamline configuration example: Adjustable SR focus coupling optics layout.

IR Beamline Research Application

Vacuum spectrometers are preferred at IR beamlines as they allow the SR to be kept completely under vacuum conditions without the negative influences of atmospheric disturbances on spectral quality as described above. The high flexibility of the optics bench makes them ideal for use at IR beamlines. A typical layout for a FT-IR spectrometer for IR microscopy imaging application with optical SR coupling is shown together with an example measurement recorded with a Focal Plane Array (FPA) detector in fig. 5 and fig. 6



Selection of Published Applications

Outstanding data have been published using such beamline spectrometer combinations in the field of IR microscopy and imaging [1], mid IR ellipsometry [2], low temperature far IR [3] and high pressure physics [4] studies. In recent years the long wavelength IR spectral range (THz, typically $<100\text{cm}^{-1}$), became of high interest due to new technologies and challenging applications which led, for example, to combined measurements techniques of e.g. IR and THz wavelengths [5] as well as IR and X-ray [6].

Fig 7a - d show the set-up of various research FT-IR spectrometer combinations configured for dedicated and demanding applications in the IR and THz wavelength range.



Fig. 7a: VERTEX 80v and HYPERION 3000 Imaging Microscope (Melbourne, Australia)



Fig. 7b: Ultra High Resolution and IFS 125HR
(PSI, Villigen, Switzerland)

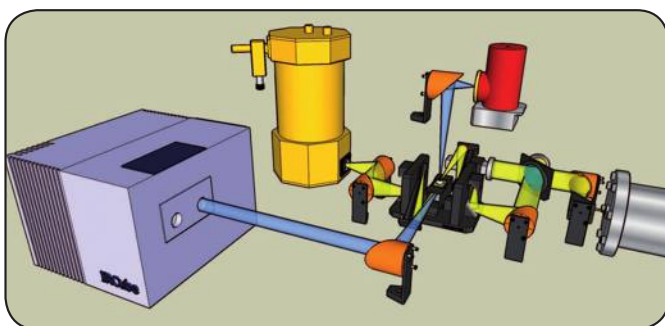


Fig. 7c: FT-IR spectrometers Matrix-M and IFS66v/S:
Parallel measurements of THz and IR spectra [5]

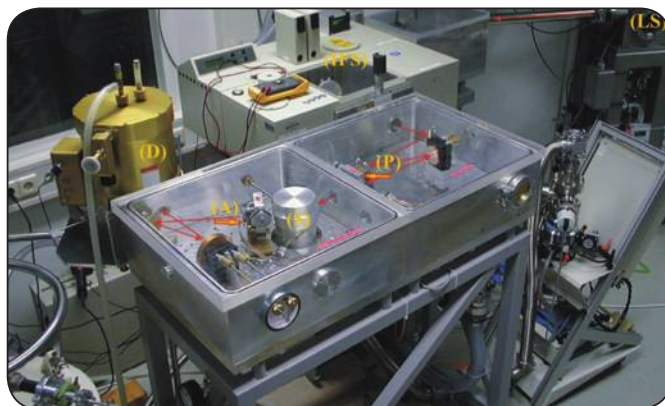


Fig. 7d: Far IR Ellipsometry and IFS 66v
(ANKA, KIT, Karlsruhe, Germany)

Summary

Vacuum FT-IR spectrometers with high flexibility using synchrotron radiation proved to be an ideal combination for the study of the characteristics of IR synchrotron radiation itself as well as for new and advanced applications requiring IR and THz radiation sources.

References:

- [1] D. Moss, B. Gasharova, Y.-L. Mathis, *Infrared Phys. Techn.* 49 (2006) 53–56.
- [2] K. Hinrichs, D. Tsankov, E.H. Korte, A. Röseler, K. Sahre, K.-J. Eichhorn, *Appl. Spectrosc.* 56 (6) (2002) 737.
- [3] C. Bernhard, J. Humlicek, B. Keimer, *Thin Solid Films*, 455–456 (2004), 143–149.
- [4] M. Matsunami, H. Okamura, A. Ochiai, T. Nanba, *Phys. Rev. Lett.* 103 (2009) 237202.
- [5] P. Innocenzi, L. Malfatti, M. Piccinini, D. Sali, U. Schade, A. Marcelli, *J. Phys. Chem. A* 113 (34) (2009) 9418–9423.
- [6] P. Innocenzi, L. Malfatti, T. Kidchob, S. Costacurta, P. Falcaro, M. Piccinini, A. Marcelli, P. Morini, D. Sali, H. Amenitsch, *J. Phys. Chem. C* 111 (2007) 5345-5350.