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Upgraded IR beamline at UVSOR

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BL6A1, a far-infrared (FIR) beamline at UVSOR, originally built in 1986, has been recently upgraded. The upgrade included the introduction of a second FT-IR spectrometer, making it possible to cover the entire FIR–IR range (3–10000 cm^{-1}) in one sequence of measurements, without having to open the sample chamber; the beamline has become a more convenient and powerful experimental station than before. The upgrade is also expected to enable such experiments as IR studies of molecules adsorbed on solid surfaces, and time-resolved IR spectroscopies. This paper describes the characteristics of the upgraded BL6A1.

Keywords: IR applications; UVSOR.

1. Introduction

As a high-brilliance light source, synchrotron radiation has been utilized over a very wide energy range. Although much of the synchrotron radiation research is concerned with VUV and X-ray applications, synchrotron radiation in the far-infrared (FIR) region also has several advantages over conventional light sources (*e.g.* Hg lamp, globar), such as a high brilliance, a flat and continuous spectral distribution, and a pulsed nature with precise temporal structure (see, for example, Nanba, 1986). Beamlines for the utilization of synchrotron radiation in FIR–IR regions have been constructed at several synchrotron radiation facilities around the world (Hirschmugl & Williams, 1995, and references therein; Marcelli *et al.*, 1998; Nelander, 1995, and references therein; Roy *et al.*, 1995, and references therein; Williams, 1995).

An FIR spectroscopic system was constructed at beamline BL6A1 of the UVSOR facility at the Institute for Molecular Science, Okazaki, Japan, in 1986 (Nanba, 1989). The system used a Martin–Puplett interferometer and covered the range from millimetre waves to the FIR region (3–250 cm^{-1}). The beamline has been utilized for transmission and reflection measurements of various materials, and has demonstrated the advantages of synchrotron radiation in the FIR region. However, optical experiments, such as the determination of optical constants, require measurements over a wide energy range from the FIR to VUV regions. Furthermore, the energies of surface vibrations probed by IR reflection–absorption spectroscopy reside in the FIR–IR region. Thus it is useful to extend the energy range of the beamline from the millimetre-wave region to the entire IR region. Below we describe the upgraded beamline in detail.

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2. Optical system

The upgrade of the beamline was planned to meet the following requirements.

(i) The energy range should be extended to 3–10000 cm^{-1}.

(ii) It should be possible to perform time-resolved spectroscopy, which takes advantage of the pulsed nature of synchrotron radiation.

In order to fulfil the above requirements, we have newly installed another spectrometer (Bruker IFS 66v), which covers an energy range 50–10000 cm^{-1}. This is a rapid-scan FT-IR spectrometer which in principle is a Michelson interferometer operated in a vacuum. Available beam splitters are quartz, KBr and Mylar films of thicknesses 6 and 12 μm.

Fig. 1 shows a schematic diagram of the upgraded beamline BL6A1. A new optical channel for the new spectrometer was inserted between the collecting pre-mirrors and the sample chamber. Diamond windows are used to separate the UHV and high-vacuum regions from the low-vacuum region. The diamond plates, 20 mm in diameter and 0.2 mm in thickness, were synthesized by chemical vapour deposition. Their measured transmittance was 0.6–0.8 in the energy range 30–4000 cm^{-1}. The features of the entire optical system after the modification can be summarized as follows.

(i) The beamline now has two spectrometers, namely the Martin–Puplett interferometer originally installed in 1986, and the newly installed one described above.

(ii) There are two parallel optical transport channels for the two spectrometers. Either of the two can be chosen with a pair of switching mirrors, and their output beams can be introduced to the same sample chamber. It is also possible to extract the beam towards another experimental system, such as a superconducting magnet for magneto-optical studies (not shown in Fig. 1).

3. Performances

Fig. 2 shows the output spectra of synchrotron radiation over the energy regions of the two spectrometers, taken with various combinations of the available beam splitters and detectors. The figure shows that the available energy range of the upgraded system is from 3 cm^{-1} to above 10000 cm^{-1}.

In order to check the advantage of high brilliance of synchrotron radiation, a collimated beam was focused onto an aperture of 1 mm diameter placed at the sample position, and the

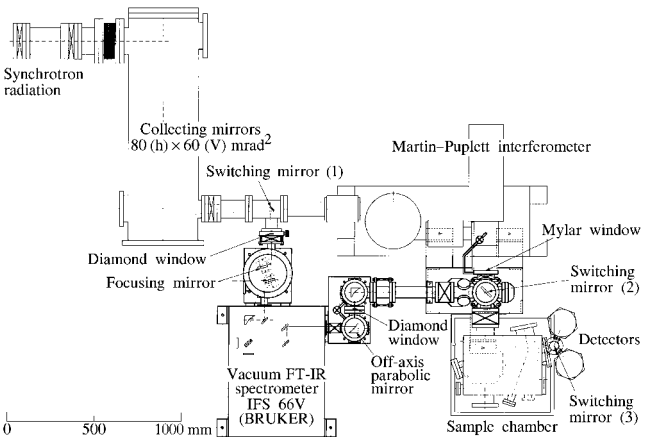

Figure 1
Schematic diagram of beamline BL6A1 after upgrading (top view).

transmitted spectral intensities were measured for both synchrotron radiation and globar sources. It was seen that synchrotron radiation is stronger than globar by an order of magnitude in the FIR region when the sample size is of the order of 1 mm.

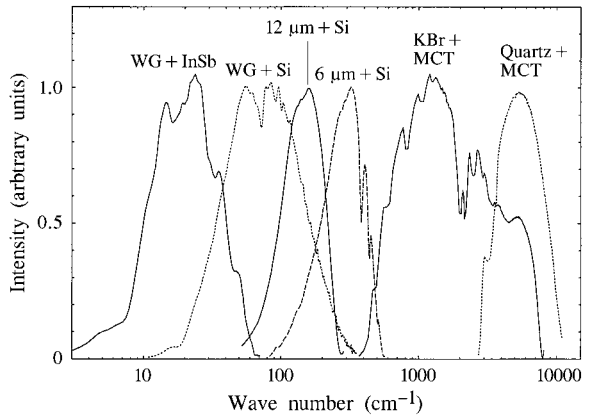

Figure 2
Spectral distribution of synchrotron radiation measured by the two spectrometers with various combinations of beam splitters and detectors. The spectra are normalized to their peak intensities and their relative intensities are not scaled. WG: wire grid beam splitter; 12 μm and 6 μm: Mylar beam splitters of thickness 12 μm and 6 μm; InSb: InSb hot electron bolometer; Si: Si bolometer; MCT: an HgCdTe detector.

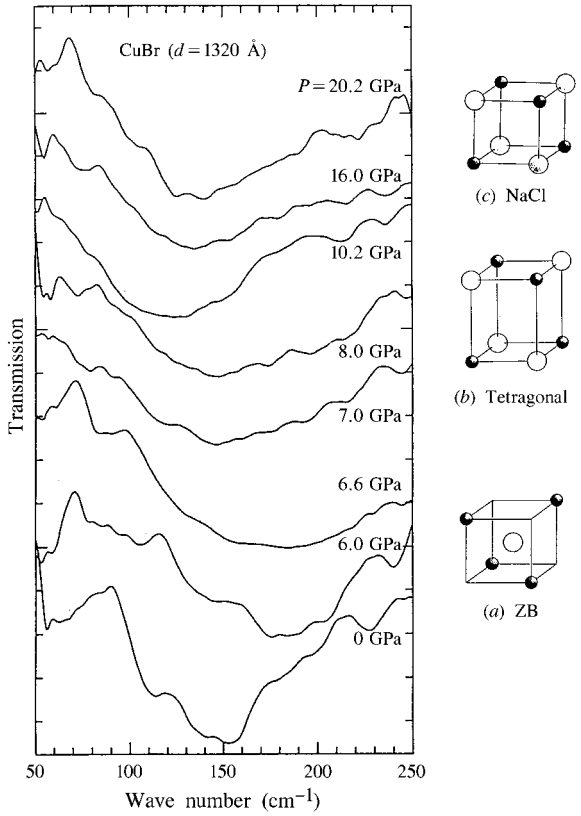

Figure 3
Evolution of the transmission spectra of CuBr microcrystals with an average diameter of 1320 Å under high pressures at room temperature. The structure at each phase, (*a*) zinc blende (ZB), (*b*) tetragonal and (*c*) NaCl structure, and the corresponding spectra are shown together. The pressures were monitored by the energy shift of the *R*1 fluorescence line of ruby chips in the cell.

Polarization distribution of the synchrotron radiation beam was also measured. A wire grid polarizer was placed in front of the detector, and spectra were measured with the direction of the wire grids parallel and perpendicular to the plane of polarization. Measured polarization is about 0.6 for almost all the energy range, which is consistent with the values calculated from the integration of original polarization distribution over the acceptance angle.

4. High-pressure measurements

FIR studies of materials under high pressure using a diamond anvil cell are examples which take advantage of synchrotron radiation in the FIR region, since the effective area available through the diamond anvils is very small, typically less than 0.5 mm in diameter. We have made FIR transmission measurements of semiconductor microcrystals under high pressure with a diamond anvil cell specifically designed for FIR experiments. Details of the high-pressure apparatus are described elsewhere (Nanba, 1989). Fig. 3 shows the FIR transmittance spectra of CuBr. As the pressure is increased, the microcrystals undergo a structural phase transition from a zinc blende to a tetragonal structure, and finally to an NaCl-type structure. The measured FIR spectra clearly show an abrupt shift of the absorption peak associated with the phase transition.

5. Time-resolved measurements

Time-resolved spectroscopy in the FIR–IR region is also a unique application of synchrotron radiation (Carr *et al.*, 1992; Carr, 1998). With a pump-probe technique, a temporal resolution as short as the width of synchrotron radiation (sub-ns) is obtained.

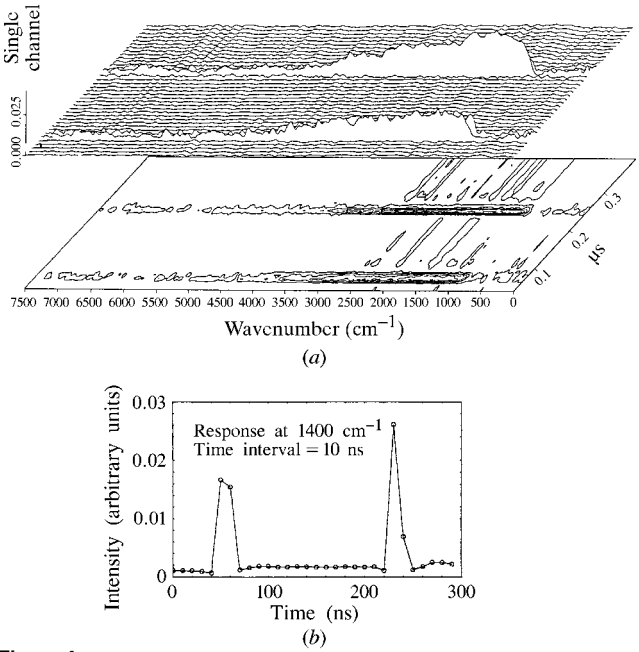

Figure 4
(*a*) Time-resolved IR spectra of synchrotron radiation measured at BL6A1 under a single-bunch operation at a beam current of 25 mA. The time interval between two consecutive spectra is 10 ns. A KBr beam splitter was used in the spectrometer, and a photovoltaic MCT detector was used to record the signal. (*b*) Temporal evolution of synchrotron radiation at 1400 cm^{-1}, extracted from (*a*).

Although there are various pulsed IR lasers with much shorter pulse widths, the very broad spectral distribution of synchrotron radiation still makes it an attractive pulsed source, especially in the FIR. We are currently preparing for a pump-probe study of photogenerated carriers in semiconductor quantum wells.

The newly installed FT-IR spectrometer can also record a time-resolved spectrum with a time resolution of 5 ns. Hence it is also possible to perform time-resolved IR experiments without synchrotron radiation (for example, using a pulsed laser as excitation source and a globar as probe source). The temporal resolution in this case is much longer, since it is limited by the detector. Therefore, this non-synchrotron radiation experiment complements the pump-probe experiment with synchrotron radiation, since the latter cannot be used for slower phenomena ($\geq$200 ns); a wider temporal range can be obtained by combining the two methods. As a test for the time-resolved operation, we measured the time-resolved spectra of synchrotron radiation. Fig. 4 shows an example of the obtained spectra. (For experimental details, see Okamura *et al.*, 1997.) It is seen that the temporal resolution of the entire system is about 20 ns, which is limited by the response of the detector. This experiment, namely using synchrotron radiation as a pulsed source and time-resolving it with the FT-IR spectrometer, may be used, for example, to evaluate the temporal response of fast IR detectors over the entire IR range.

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