Photoacoustic spectroscopy using a synchrotron light source

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Abstract: We have investigated the use of a synchrotron as a source for infrared photoacoustic spectroscopy. A synchrotron has an intrinsically high radiance, which is beneficial when photoacoustic spectroscopy is applied to small samples, especially at long wavelengths.

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OCIS codes: (999.9999) Synchrotron Radiation; (300.6430) Spectroscopy, optoacoustic and thermo-optic; (300.6270) Spectroscopy, far infrared; (300.6340) Spectroscopy, infrared

Introduction

Synchrotron radiation has been used by several research groups as an alternative to thermal radiation in both mid- and far-infrared spectroscopy [1-5]. In the mid-infrared, the major advantage of synchrotron radiation is its intrinsically high radiance (radiant power per unit area per unit solid angle). This has led to its use in infrared microspectroscopy, because all the light emitted from the synchrotron can be focused into a diffraction limited spot [1-3]. This means that, for microspectroscopy, synchrotron radiation can give a significantly higher signal-to-noise ratio than thermal radiation. Synchrotron radiation has also been used in far-infrared spectroscopy, because at long wavelengths it is more intense than either globar or Hg arc blackbody sources [4,5].

We have compared the use of synchrotron and thermal radiation in the context of mid- and far-infrared photoacoustic spectroscopy. This is typically a macroscopic technique used in the mid-infrared, but it can also be applied to small samples and longer wavelengths. The technique is of interest because it permits measurement of otherwise intractable samples, and also affords the possibility of depth profiling of layered or inhomogeneous samples.

Results and Discussion

Mid- and far-infrared spectra were acquired using a Bruker IFS 66v vacuum spectrometer on the U10A beamline at the National Light Source, located at Brookhaven National Lab. Spectra were measured at 6 cm^{-1} resolution, with a scan velocity of 2.2 kHz. Mid-infrared spectra were measured using a Ge on KBr beamsplitter, while far-infrared spectra were measured using a Ge on PET beamsplitter. For the spectra measured using synchrotron radiation, the magnitude of the photoacoustic response depends linearly on the current in the ring, which varies between 400 and 1000 mA. All of these spectra were therefore normalized to a beam current of 700 mA.

Photoacoustic spectra of samples of carbon-black filled rubber, carbon-black powder, and coke were measured using both the internal globar and the synchrotron as a source. To simulate different sample sizes, brass apertures with diameters between 1 and 3 mm were placed on top of the samples. The small photoacoustic signal from the brass aperture itself was subtracted from all the spectra, to allow quantitative comparison between the synchrotron and the globar. In the absence of an aperture, the beam size at the sample was 5 mm. Figure 1 shows the far-infrared photoacoustic spectra of carbon-black filled rubber, with no aperture to restrict the beam size. It can be seen from the figure that below approximately 200 cm^{-1} , the synchrotron has more energy than the globar source.

When the beam size at the sample is restricted by using apertures, the high radiance of the synchrotron means that it has more energy at shorter wavelengths. By comparing data taken with a series of different apertures, it is possible to obtain a good estimate, for any given sample size, of the wavenumber region for which the synchrotron will give a higher signal-to-noise ratio than the globar. Figure 2 shows the location of the crossover point for the synchrotron vs. the globar as a function of aperture size. It is clear from the figure that synchrotron radiation can improve the signal-to-noise ratio, when compared to a globar source, over all or part of the far- and mid-infrared regions of the spectrum.

Fig. 1. Far-infrared photoacoustic (PA) spectra of carbon black, obtained with different sources of infrared radiation. Dashed line, synchrotron radiation (SR); solid line, globalar.

Fig. 2. Dependence of crossover frequency on aperture size. The crossing frequency is the highest wavenumber for which SR PA intensity exceeds globalar PA intensity. The diamonds are experimental points; the solid line is a power-law fit to the data.

Conclusions

Synchrotron radiation can provide an increase in signal-to-noise ratio for photoacoustic spectroscopy in the mid- and far-infrared. This improvement is greater at long wavelengths, and depends on sample size. A quantitative relationship between sample size, and the wavenumber below which the synchrotron has more energy, has been calculated from the experimental data.

Acknowledgements

One of us (RSJ) would also like to thank Bruker Optics Inc., for the time and some of the equipment needed to perform this work.

This work was supported in part by Natural Resources Canada, and by the U.S. Department of Energy under contract number DE-AC02-98CH10886.

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