Nanoscale infrared spectroscopy

In the early days of scanning probe microscopy, researchers and instrumentation developers were often postulating about the future and, perhaps one day, the advent of the "lab-on-a-tip." While the technology has seen the development of highly spatially resolved topography imaging coupled to a series of different physical measurements, it is only recently that is has been possible to perform chemical characterization measurements with infrared spectroscopy on the nanoscale. The enabling technique is known as nanoIR™.

Craig Prater*, Kevin Kjoller and Roshan Shetty,
Anasys Instruments, Santa Barbara, USA
**E-mail: craig@anasysinstruments.com*

For many years, infrared spectroscopy has provided the ability to characterize and identify chemical species. However, it has always been restricted to resolution in the order of 5 – 10 microns and then only when applying attenuated total reflection spectroscopy, ATR. Now, when combined with the nanoscale spatial resolution of an AFM tip, it is possible to measure and map local chemical composition below the diffraction limit of light. The technology described here as nanoIR will also perform nanoscale topographic, mechanical and thermal analyses.

This exciting and unique technology is provided through a new platform called nanoIR (Anasys Instruments, Santa Barbara, CA). The nanoIR is a probe-based measurement tool that reveals the chemical composition of samples at the nanoscale (Fig. 1). This laboratory solution combines key elements of both infrared spectroscopy and atomic force microscopy (AFM) to enable the acquisition of infrared spectra at spatial resolutions of 50 - 200 nanometers, well beyond the optical diffraction limit. Potential application areas span polymer

Fig. 1 NanoIR system.

ISSN:1369 7021

science, materials science, and life science, including detailed studies of structure-property correlations.

The science behind the system applies the patent-pending technology of infrared nanospectroscopy, a technique pioneered by award-winning researcher Dr. Alexandre Dazzi of the Laboratoire de Chimie Physique ,CLIO, Université Paris-Sud, Orsay, France. This is a photothermal method which follows the absorption of the infrared and the subsequent generation of heat.

Instrumentation

The nanoIR system uses a pulsed, tunable IR source to excite molecular absorption in a sample that has been mounted on a ZnSe prism (Fig. 2). Samples are prepared in one of two ways. For many samples, ultramicrotomy is used to cut sections with thicknesses between 100 nm and 1000 nm. These are then transferred to the prism surface. In other sample preparations, it may be possible to cast thin films from solvent directly on the prism surface.

The IR beam illuminates the sample by total internal reflection similar to conventional ATR spectroscopy (Fig. 3). As the sample absorbs radiation, it heats up, leading to rapid thermal expansion that excites resonant oscillations of the cantilever which is detected using the standard AFM photodiode measurement system. These induced oscillations decay in a characteristic ringdown which can be analyzed via Fourier techniques to extract the amplitudes and frequencies of the oscillations. Then, measuring the amplitudes of the cantilever oscillation as a function of the source wavelength, local absorption spectra are created.

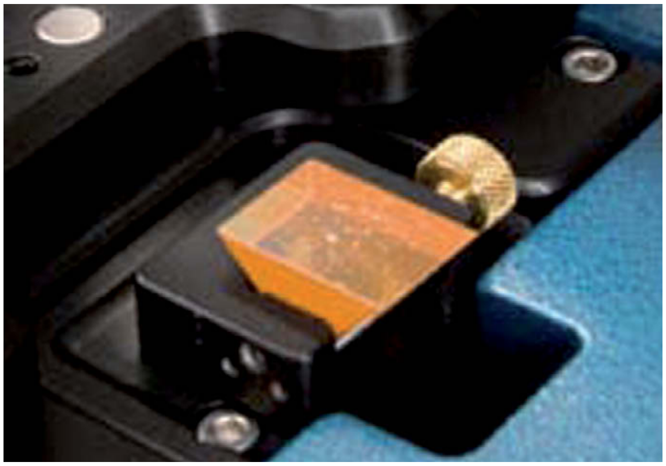

Fig. 2 Samples are mounted on a zinc selenide prism.

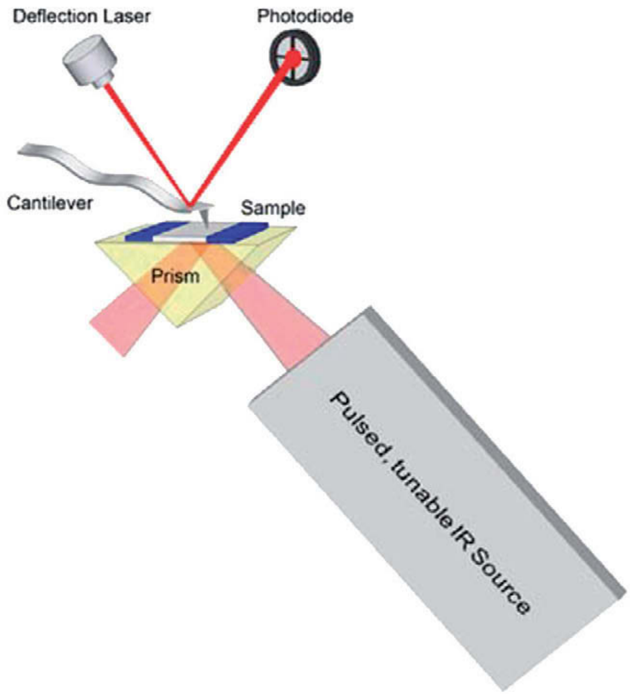

Fig. 3. Schematic shows the illumination of the sample by the IR laser.

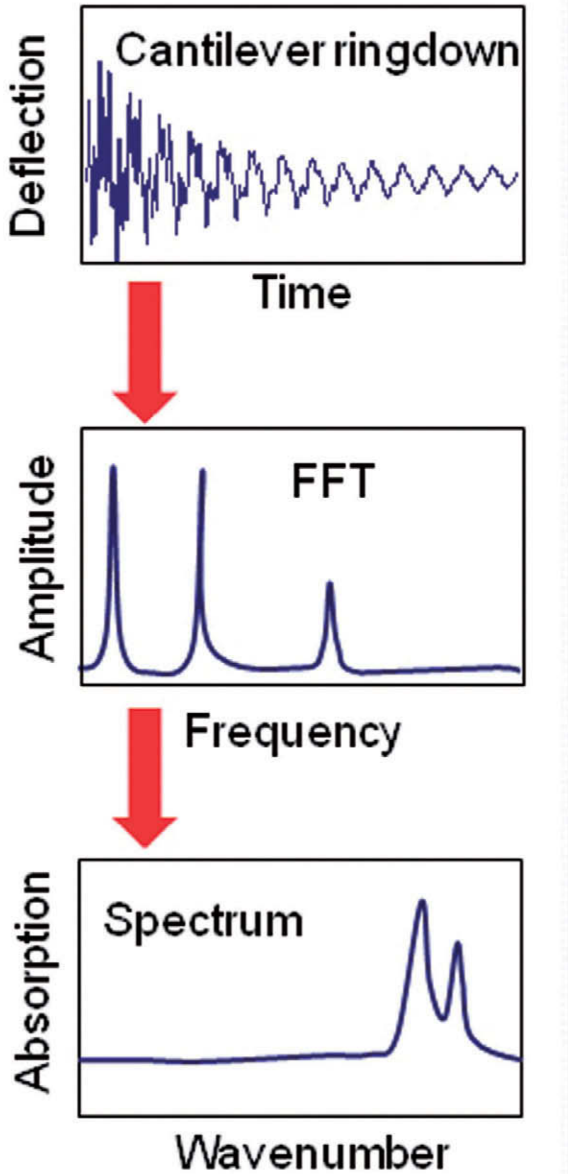

Fig. 4. Schematic to illustrate the process of analyzing the ringdown of a cantilever to generate a familiar IR spectrum.