Next-generation atomic force microscopy

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In partnership with the Center for Sustainable Polymers, the CharFac is thrilled to describe two new AFM systems!

One system is the first large-sample/motorized platter AFM in the CharFac’s 3-decade history – the **Bruker Dimension Icon** (room 22 Shepherd Labs, top photo). In addition to handling up to a 6” wafer or many smaller samples at digitally defined locations, the Icon features a low-noise, fully closed-loop scanner with a 12-micron Z range. Importantly this scanner removes the artifacts of nonlinearity and cross coupling generated by its piezo-elements. (Traditional scanners require nonlinear post-processing of topographic data to remove false curvature, for example to quantify small changes in surface height that occur over large lateral distances, e.g., when a film-deposition mask is far from substrate; moreover, the measurement of large vertical changes over small lateral distances can be erroneous due to Z scanner nonlinearity.) The Icon is also especially well-suited to **quantitative nanomechanical analysis** (providing repeated access to tip-shape-imaging and modulus standards).

The second system is truly novel: an “AFM-IR”, the **Bruker/Anasys NanoIR3** (room 1-206 Nils Hasselmo Hall, bottom photo). This system uses pulsed infrared laser light focused near the tip-sample interface, along with photoinduced sample expansion sensed at the tip-sample interface, to provide IR absorption images that in some cases can approach ~10-nm lateral resolution. (Compare to the ~10-micron resolution of conventional IR microscopes.) It includes two laser types (at left on optical table) spanning wave number ranges of 900-1800 + 2000-2300 cm⁻¹ (via an assembly of four QCL chips, black box) and 2700-3600 cm⁻¹ (OPO laser with red sticker). The laser light is pulsed at 100’s of kHz so as to take advantage of frequency-mixing concepts in order to separate IR response from the usual topographic and nanomechanical information collected in AFM scanning. As many as 8 data acquisition channels can be utilized such that a multitude of information (e.g., both chemical and mechanical) is simultaneously imaged.

Via IR absorption the mapped information can relate to chemical identity, deuteration, crystalline vs amorphous content and other stereochemical features (e.g., polymer side chains). These maps can be assembled (a) in a data-rich **hyperspectral** way – meaning a full spectrum (absorption vs wave number) per measurement site over a 2D grid, along a line, or in a set of arbitrary locations (as designated on conventional AFM images); or (b) as a high-pixel-density image at a particular wave number.
This page illustrates some of these capabilities on polymeric materials, using the tapping-mode scheme wherein the IR response is detected via the amplitude of the excited higher eigenmode of the cantilever probe. (Another operating scheme, which provides even higher sensitivity but potentially with damaging sliding friction force, presses the tip continuously to the surface while pulsing the IR laser at the cantilever’s contact resonance.)

A good example of the sensitivity and resolution of the NanoIR3 is the case of a polymer blend or block copolymer, with chemical composition varying over lateral scales of tens of nanometers. Figure 1a is an IR absorption image of a thin film of a 50:50 blend of poly ethyl:methyl methacrylate, using 1026 cm$^{-1}$ laser irradiation (denoted with vertical green line in Figure 1b) that is more strongly absorbed (brighter) by PEMA compared to PMMA. (This results from differences in side chain on otherwise very similar polymers).

Figure 1. (a) IR absorption (brighter being greater) at a wave number of 1026 cm$^{-1}$ on a PEMA-PMMA blend; (b) IR absorption spectra (1780–910 cm$^{-1}$) collected at two characteristic locations on PEMA (thin plot) and PMMA (thick plot).

A second example is a core-shell polymer fiber 40 microns in total diameter (sectioned in epoxy). The polyamide nature of the 3-µm thick nylon shell is obvious in a spectrum acquired local to this region, as shown in Figure 2a (boldest plot). Figure 2b contains IR absorption images acquired at laser excitations of 1652 cm$^{-1}$ (left, an amide I absorption line) and 1512 cm$^{-1}$ (right). In the right image the epoxy matrix absorbs the strongest (brightest), followed by weak absorption by the polyamide shell, with little absorption by the polyester core (darkest).

There are a number of tip-sample interaction details that affect the nature, quality and usefulness of the IR spectroscopy and imaging. (E.g., microtomy cutting marks produce variable geometry at the tip-sample interface, resulting in variable sensitivity to IR absorption as seen in Figure 2b.) We are eager to conduct feasibility work on a variety of sample types to determine if the method can be successful. In some cases we may need to explore both tapping mode and contact resonance schemes of operation. There also may be issues of sample thickness that beg careful consideration. (Thinner is often needed for better lateral resolution, thicker for greater sensitivity, but not so thick as to impede the transport of IR heating away from the measurement site from one pulse to the next.) Please contact Greg at cfac-spm@umn.edu with questions or to schedule a discussion of capabilities in the context of your research problem.