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New Microscopies for Polymer Analysis… What's in YOUR toolkit?

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As microscopy continues to evolve and spectroscopy continues to collide with imaging, new instruments are becoming available, offering expanded solutions for polymer analysis. This paper considers new hybrid instrumentation, centering specifically on FT-IR, Raman, and AFM.

FT-IR

For polymer chemists, the advantages of FT-IR are clear: the technology provides a chemical fingerprint which often clarifies the difference between polymorphs as well as identifying contamination. When wedded to imaging, the combined technique also provides critical context. The introduction of ATR (Attenuated total reflection) objectives has reduced the need for elaborate sample preparation and dramatically decreased analysis time. Today, integrated FT-IR microscopes are available, as well as retrofit systems to convert existing microscopes to FT-IR capability, offering considerable savings in time and investment in equipment [1]. For microscopists new to spectroscopy, manufacturers now offer a wide range of support tools for spectral characterization.

Combining FT-IR with microscopy requires a shift in sample preparation strategies: FT-IR typically requires large, flat areas while microscopy requires that the sample be in an undisturbed native state to evaluate heterogeneity, context, and morphology. Image analysis adds another layer of complexity, often requiring a contrast technique or special processing to enhance phases. Creating a brief analytical scheme prior to starting the actual analysis avoids pitfalls and maximizes results [2, 3].

Atomic Force/Scanning Probe Microscopies (AFM/SPM)

The big question most commonly asked is "what can AFM do for me?" Originally, this technique was designed to characterize surfaces, either in terms of their topography or via physical and chemical properties such as phase, electrical characteristics and electrochemistry, tacticity, and magnetism, an excellent complement to light and electron microscopy. The past two years have seen a number of exciting AFM/SPM advances. Atomic Force Acoustical Microscopy (AFAM) now illuminates differences in phases, using differing elasticity as the contrast mechanism (Figure 1) [4]. High Throughput Combinatorial Microscopy (HTCM) conducts batch analyses of dozens of samples, automatically, making it ideal for defining properties of new compositions of polymers, paints, pigments, and pharmaceuticals. NTegra TOMO, a unique AFM/ultramicrotome hybrid, opens the world of true 3D ultrastructure, imaging from the bulk, at the nanoscale (Figure 2) [5].

Raman Confocal and beyond

Advances in Raman spectroscopy have simplified this technique considerably, making it available to the mainstream. An excellent complement to FT-IR, Raman permits chemical characterization in materials with strongly IR absorbing N-H and O-H bonds. Combining Raman with confocal adds an imaging component, but, more importantly, enables the system to go below the surface of multi-layered polymers, to collect spectra from a specific location [6].

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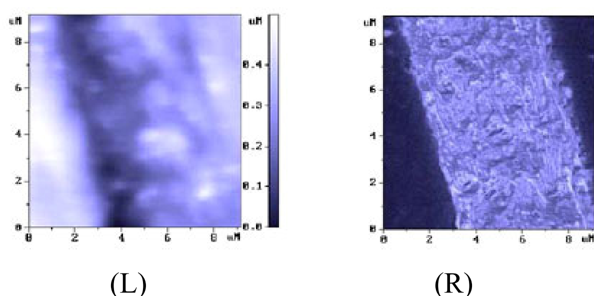
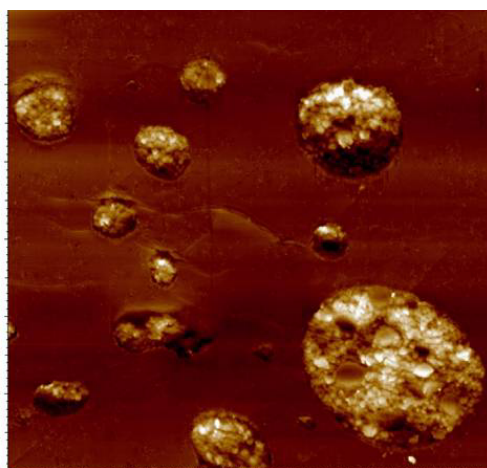
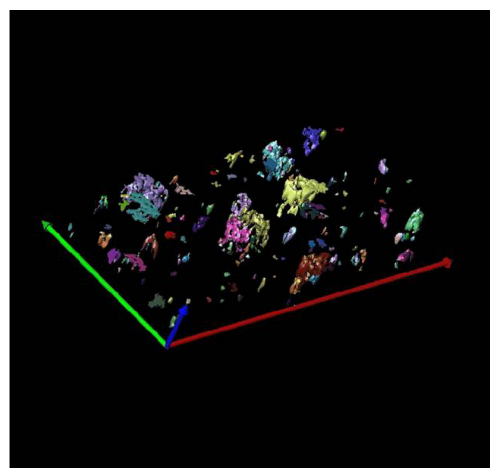
The current trend in microscopy development is hybrid instrumentation. Today, it is not unusual to find light microscopy coupled with FT-IR and Raman on one stand or to find AFM coupled with light microscopy and Raman. The ultimate example of this new trend is an integrated workstation, which runs under one software package and integrates five imaging modalities (light, fluorescence, confocal, NSOM and 40 different AFM techniques) with dual channel fluorescence and Raman spectroscopy. Using this new hybrid, a researcher can image at the atomic scale then take the Raman spectrum of a single molecule, pushing polymer analysis to the next generation.



(L) (R)

Figure 1. (L) AFM Topography Image. (R) AFAM image. Scan size: 10µm x 10µm. Sample: sandwiched layers of high density (bright) and low density (dark) polyethylene (HDPE/LDPE). Sample courtesy of J. Loos, Dutch Polymer Institute, TU/Eindhoven. Image courtesy of NT-MDT, Zelenograd, Russia.





(L) (R)

Figure 2. Comparison of traditional AFM topography image to 3D image. (L) Conventional AFM Topography image, scan size 12µm x 12µm. (R) 3D reconstruction made from 15 sequential AFM images cut at 200nm spacing, scan size 25µm x 25µm. Sample: Polystyrene/High impact PS, blended with silica. Sample courtesy of Dr. Aliza Tzur, Technion, Israel. NTegra TOMO data courtesy of Dr. Anton Efimov, NT-MDT, Zelenograd, Russia. 3D reconstruction courtesy of Media Cybernetics, Inc., Silver Spring, MD.

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