Force Modulation Imaging with Atomic Force Microscopy

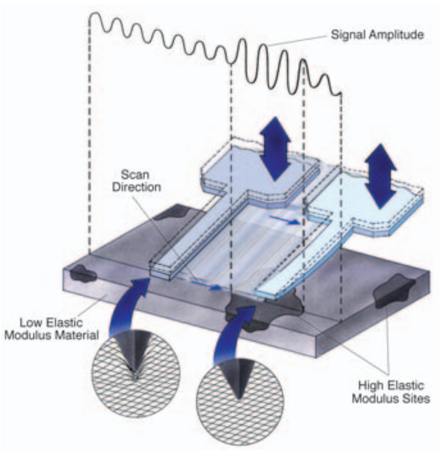

Figure 1. Diagram of force modulation principle showing probe scanning a low elastic modulus material from left to right. Signal amplitude increases when tip encounters higher elastic modulus (stiffer) sites.

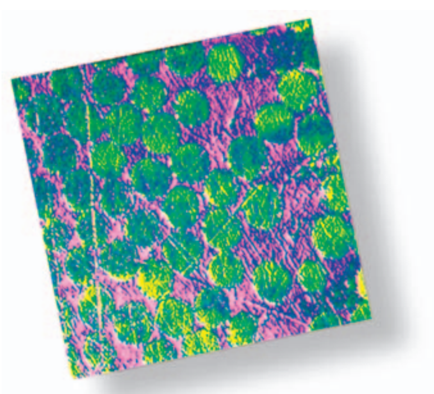

Figure 2. Carbon fibers in epoxy matrix. Sample courtesy Virginia Polytechnic Institute. 40μm scan.

Force modulation imaging is an atomic force microscopy (AFM) technique that identifies and maps differences in surface stiffness or elasticity[1]. It is one of several techniques developed as extensions of AFM. These techniques probe various surface properties to differentiate between the different materials that make up heterogeneous surfaces.

Force modulation imaging has many applications, including locating transitions between different components in composites, rubber, and polymer blends; imaging organic materials on hard substrates; detecting residual photoresist on integrated circuits; and identifying contaminants on various surfaces.

The Technique

Force modulation imaging is a secondary imaging technique based on contact mode AFM. In contact mode AFM, the probe (cantilever and tip) is scanned over the surface (or the sample is scanned under the probe) in an x-y raster pattern. The feedback loop maintains a constant cantilever deflection, and consequently a substantial, constant force on the sample.

In force modulation, the probe also moves with a small vertical (z) oscillation (modulation) which is significantly faster than the raster scan rate (Figure 1). This means the force on the sample is modulated such that the average force on the sample is equal to that in contact mode.

When the probe is modulated with the tip in contact with a sample, the sample surface resists the oscillation and the cantilever bends. Under the same force, a stiff area on the sample deforms less than a soft area; i.e., stiffer areas put up greater resistance to the cantilever's vertical oscillation, and, consequently cause greater bending of the cantilever. The variation in cantilever deflection amplitude at the frequency of modulation is a measure of the relative stiffness of the surface. Topographical information (DC, or non-oscillatory deflection) is collected simultaneously with the force modulation data (AC, or oscillatory deflection).

Advanced Design

The force modulation imaging technique was pioneered by Veeco Instruments in 1989[1]. Early designs applied a modulation signal to the z section of the piezoelectric scanner to induce the vertical cantilever oscillation. This technique had some success and was widely duplicated, but it has some drawbacks. The high-frequency modulation signal can excite the scanner's mechanical resonances. This can reduce the quality of both the topographic and the force modulation images, and is still being used today in other AFM instruments.

But, Veeco Instruments has since implemented a improved, second generation, force modulation imaging technique that contains an additional

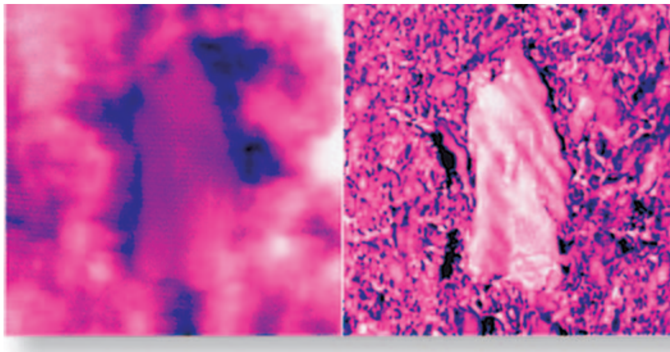

Figure 3. Contact mode topography (left) and force modulation image (right) of carbon black deposit in automobile tire rubber. 15μm scan.

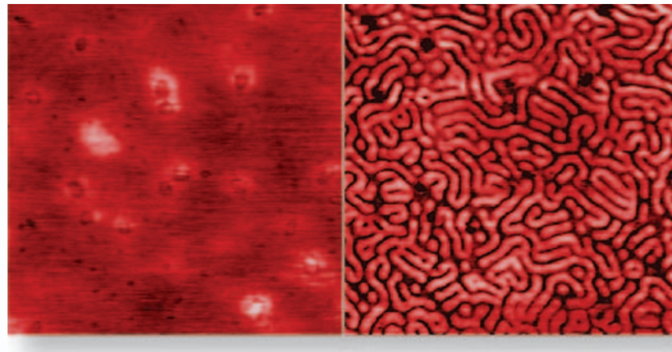

Figure 4. Contact mode topography (left) and force modulation image (right) of a two-phase block copolymer. The softer, more compliant component of the polymer maps in black. 900nm scans.

piezoelectric actuator to separately modulate the probe. This reduces or eliminates spurious excitation of scanner resonances. The actuator is usually driven at frequencies of 5 to 20 kilohertz for force modulation experiments.

The modulation signal is generated with a high-precision digital frequency synthesizer with advanced software functionality that allows the user to quickly select an optimum modulation amplitude and frequency. The cantilever oscillation amplitude is detected with high-speed circuitry providing a noise level of less than one Ångstrom over a bandwidth greater than one megahertz. The result is superior discrimination of sample stiffness and minimal susceptibility to topographic artifacts.

Examples

Veeco Instruments' force modulation imaging technique is particularly useful for detecting soft and stiff areas on substrates which exhibit overall uniform topography. For example, Figure 2 shows the force modulation image of a polished cross-section of an experimental carbon/epoxy composite of the type used in aeronautics, bicycle frames, and golf clubs. The material consists of 5μm-diameter graphite fibers (Apollo 45-850) embedded in a thermoplastically-toughened epoxy matrix. The Young's elastic modulus of the fibers is about 45 Msi compared to 0.5 Msi for the matrix. This difference in elasticity produces strong contrast in the force modulation image, whereas the topographical image (not shown) provides substantially less information. Figure 3 shows images of a carbon black deposit in a section of automobile tire rubber. The force modulation image (right) clearly differentiates the stiffer carbon black area in the center from the surrounding rubber.

Figure 4 shows the contact mode topography and force modulation images of a two-phase block copolymer. This set of images illustrates the resolving power of the force modulation imaging technique — while the topography image provides very little indication of the hetero-geneous nature of the sample, the force modulation image clearly maps the two phases of the material, resolving features as small as 10nm wide.

Summary

Force modulation imaging technique enables localized studies of relative difference in surface elasticity with nanometer-scale resolution.

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Note:
Veeco Instruments has been awarded a patent for the application of force modulation imaging.

References

1. Maivald P., Butt H. J., Gould S. A. C., Prater C. B., Drake B., Gurley J. A., Elings V. B., and Hansma P. K. 1991 Nanotechnology 2 103.

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