

FORCE MODULATION IMAGING APPLICATION NOTE

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Force Modulation Imaging is a scanning probe microscopy (SPM) technique that identifies and maps differences in surface stiffness or elasticity¹. It is one of several new techniques developed as extensions to the basic SPM topographical mapping capabilities. These techniques use a variety of surface properties to better differentiate among materials where topographical differences are small or unmeasurable.

Force Modulation Imaging can be used in a wide range of applications including identifying transitions between different components in composites, rubber and polymer blends, evaluating polymer homogeneity, imaging organic materials on hard substrates, detecting residual photoresist on integrated circuits, and identifying contaminants in a variety of materials.

The Technique

In standard contact mode SPM, the probe 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 force on the sample and, consequently, constant cantilever deflection.

With the force modulation technique, the probe or sample assembly is scanned with a small vertical (z) oscillation (modulation) which is significantly faster

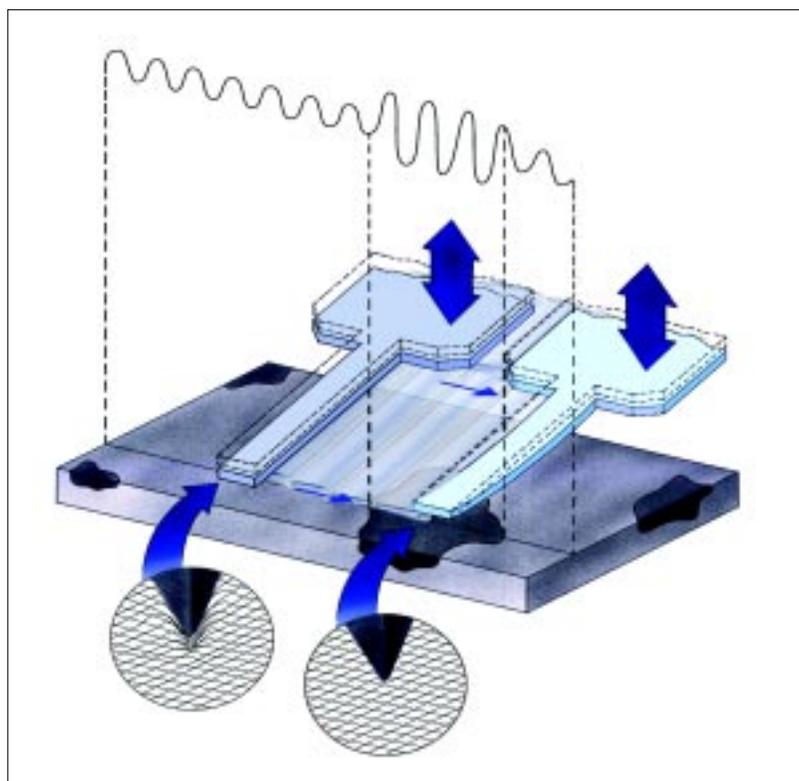


Figure 1: Diagram of Force Modulation principles showing probe scanning a low elastic modulus material from left to right. Signal amplitude increases when tip encounters higher elastic modulus (stiffer) sites (also see text).

than the scan rate (Figure 1). The force on the sample is modulated about the setpoint scanning force such that the average force on the sample is equivalent to that in simple contact mode.

When the probe is brought into contact with a sample, the surface resists the oscillation and the cantilever bends. Under the same applied force, a stiff area on the sample will deform less than

a soft area; i.e., stiffer surfaces cause greater resistance to the vertical oscillation and, consequently, greater bending of the cantilever. The variation in cantilever deflection amplitude 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).

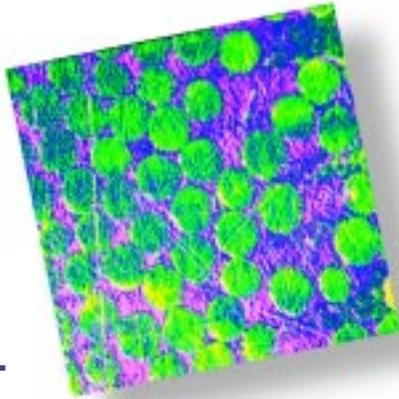


Figure 2:
Carbon fibers in
epoxy matrix.
Sample courtesy
Virginia Poly-
technic Institute.
40 μ m scan

Advanced Design

The Force Modulation technique was pioneered by Digital Instruments in 1989¹. Early designs added a modulation signal to the z section of the piezoelectric scanner to induce the vertical oscillation. While this technique has been somewhat successful and widely duplicated, it has some drawbacks. Adding the additional high-frequency piezoelectric modulation signal to the piezoelectric scanner can excite any of a large number of the scanner's mechanical resonances. This unwanted crosstalk can reduce the quality of topographic and force modulation images.

Digital Instruments has now implemented a second generation force modulation system that contains an additional piezoelectric actuator to separately modulate the tip position. This actuator can reduce or eliminate spurious excitation of scanner resonances. The actuator is generally driven at frequencies of approximately 5 to 20 kilohertz for force modulation experiments. Combined with Digital's advanced Interleaved Scanning, these new force modulation tip holders provide a wide range of new capabilities.

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 angstrom over a bandwidth greater than one megahertz. The result is a system with superior discrimination of sample stiffness and less susceptibility to modulation and topographic artifacts. Digital Instruments has been awarded a patent for the application of force modulation techniques to scanning probe microscopy.

Examples

Our force modulation 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 images 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 a 5 μ m-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 provides substantially less information.

Figure 3 shows images for 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.

The topographical image only hints at its presence.

Summary

Force Modulation Imaging has important applications where surface features must be differentiated, and in investigative studies of relative surface elasticity. As described, it has numerous uses in polymers, semiconductors, composite materials and other applications.

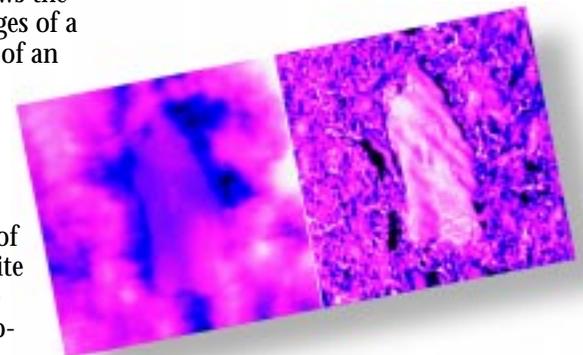


Figure 3:
Carbon black deposit in automobile
tire rubber. 15 μ m scan.

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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AN1-5/94