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Force Modulation Imaging

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Force modulation Imaging is a scanning probe microscopy (SPM) technique that identifies and maps differences in surface stiffness or elasticity[1]. 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.

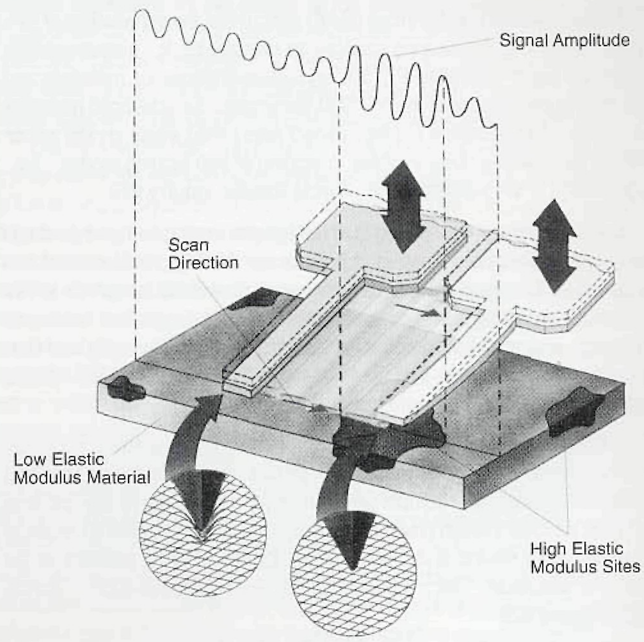
Force Modulation Imaging can be used in a wide range of applications including identifying transitions between different components in composites, rubber and polymer blends, evaluating polymer homogeneity, imaging organic materials on hard substrates, detecting residual photoresist on integrated circuits,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



Advanced Design

As a result of their pioneering efforts in 1989, Digital Instruments has been awarded a patent for the application of force modulation in scanning probe microscopy. 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 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.

A second generation force modulation system now 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.

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.

Examples

The 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 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 5 μm 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.

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. ■

1. Malvald P, Butt HJ, Gould SAC, Prater CB, Drake B, Gurley JA, Elings VB, and Hansma PK 1991 Nanotechnology 1 103.

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

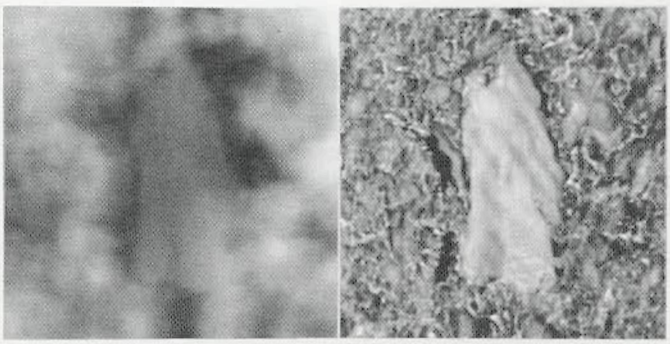

Figure 3: Carbon black deposit in automobile tire rubber. 15 µm scam

A Note on Molybdenum Apertures

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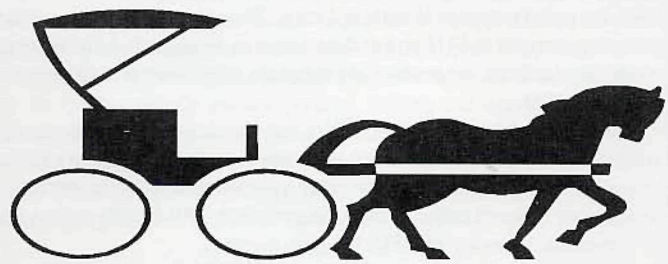
Unfortunately nature did not provide us with a perfect metal for fabricating electron optical structures. Even for the restricted field of apertures we must accept engineering compromises. Molybdenum is one of the good choices, which many of us use, but in a demountable system such as the microscope it must occasionally be cleaned. Unlike platinum, it cannot be cleaned by firing outside the vacuum in a reducing flame and one must be careful not to mistake it for platinum. It can be mechanically cleaned in air and washed with solvents and mild acids (check your hand book). Haine was perhaps the first to point out that it can be fired in a modest vacuum at 1400 deg C in a platinum or tungsten boat. This works quite well but eventually leads to trouble if frequently used. The problem arises from the fact that molybdenum has a very limited solubility for carbon and at saturation begins to form carbides which are insulators and are not reduced by firing in vacuum. To avoid this problem, first remove the bulk of carbonaceous film by mechanical cleaning. The small amount that is left will decompose during firing and the carbon which enters the aperture surface will be harmlessly dissolved in the metal. ■



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