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Advanced Nanoscale Characterization with diCaliber

Electric and Magnetic Force Microscopy

By: Thomas Müller, Ph.D, Applications Scientist
Veeco Instruments Inc.

INTRODUCTION

Atomic force microscopy (AFM) permits the characterization not only of nanoscale topography and mechanical properties, but also of electric and magnetic properties. AFM modes available for this purpose include electric force microscopy (EFM), magnetic force microscopy (MFM), and scanning electrostatic potential microscopy (SEPM). With its two integrated dual lock-in amplifiers and a powerful software package, the Caliber™ system offers all of these modes as standard features. By detecting magnetic and electric field gradients near the sample surface and measuring the local electrostatic potential, these AFM modes enable applications such as failure analysis of semiconductor devices and data storage media, detection of trapped charges, determination of contact potentials, mapping strength and direction of electric polarization, mapping magnetic domains, and detecting the presence of conductive components in composite samples.

Figure 1: The portable diCaliber mini-SPM

MAGNETIC FORCE MICROSCOPY

Magnetic Force Microscopy is used to characterize the magnetic field near the sample surface. MFM imaging makes use of several capabilities offered by the Caliber system.

First, the sensitive detection of varying magnetic fields is enabled by TappingMode phase imaging. When a magnetized probe is employed, the presence of a diverging magnetic field can cause a vertical force that varies during the (nearly vertical) oscillating motion of the cantilever, thus modifying the cantilever resonance properties and shifting the phase at constant drive frequency. Therefore, the phase image will contain a signature of the magnetic field. To prepare for phase imaging, lock-in parameters need to be adjusted. On Caliber this is accomplished in the TappingMode dialog, see also *Advanced Nanoscale Characterization with Caliber: Phase Imaging of Polymer Materials.*

Second, the sensitive detection of weak, long-range magnetic fields requires the elimination of short range "mechanical" sample interactions, which would otherwise dominate the phase signal. In some cases, magnetic forces can be detected simply by phase imaging with very gentle feedback conditions such that the probe "floats" above the surface. However, for samples with significant topography this method usually fails. To achieve the reliable decoupling of magnetic information from topography for any sample, Caliber offers the layered imaging mode. At each XY position, the acquisition of topography data is followed by lifting and/or lowering of the probe and data acquisition at additional Z-positions, leading to the generation of a "layered" 3D image file. Layered imaging can be used to acquire force volume data for mechanical characterization (see also Advanced Nanoscale Characterization with Caliber: Force Spectroscopy and Local Mechanical Properties) or for the generation of MFM or EFM images.

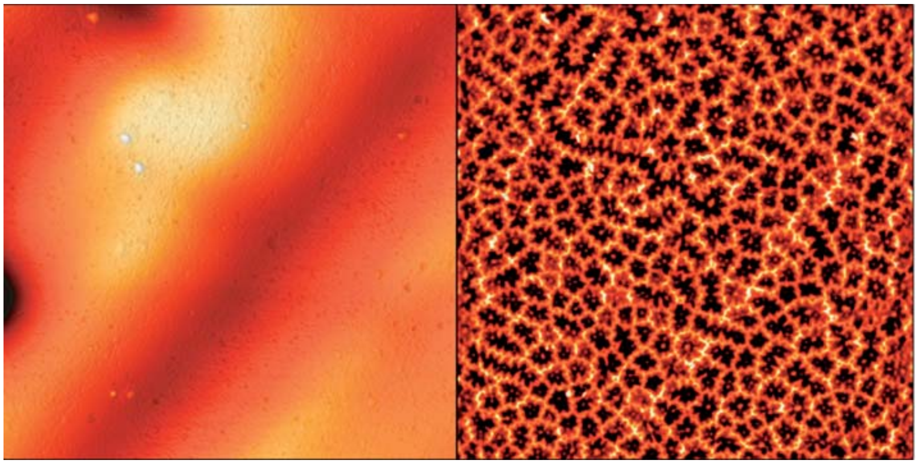


Figure 2: Topography (left) and MFM phase image (right) of a Ni-plated steel sample. The MFM data reveals the magnetic domain structure. Notice the successful decoupling of magnetic from topographic information. Image size 50µm.

Figure 2 shows topography and MFM data for a Ni-plated steel sample. The topography data channel reveals a rough surface with height variations exceeding 100nm in a 50µm image. None of the topography is reflected in the MFM phase data, which has an entirely different appearance. This successful isolation of magnetic information from topography was achieved using a probe lift height of 50nm for the acquisition of the MFM phase data. The zigzag lines dominating the MFM data reflect the magnetic domain structure present in this Ni-plated sample

ELECTRIC FORCE MICROSCOPY

Electric Force Microscopy is used to map out divergent electric fields. As in the case of MFM, the most sensitive detection is achieved by phase imaging, while the layered imaging mode on Caliber ensures the elimination of short-range "mechanical" interactions. In EFM, the presence of an electric field polarizes the conductive (usually metal-coated) probe, leading to a force on the probe. The nearly vertical motion of the probe during its oscillation cycle makes it sensitive to the vertical derivative of the vertical force component ($\partial F_Z/\partial z$).

Charges trapped on or beneath the sample surface often give rise to sufficiently divergent electric fields to be detected by EFM. In addition, EFM can be used to map the location of conductive sample parts. Using Caliber's built-in voltage supplies, a bias voltage can be applied between probe and sample, leading to charge accumulation on conductive sample parts, thereby enabling their detection by EFM. The presence of charges usually causes an attractive interaction with the (conductive) probe that decreases for increasing probe distance. The result is a downshift of resonance frequency or a downshift of phase at a constant drive frequency. Charged areas usually appear with dark (negative) EFM phase contrast relative to their surroundings.

The AFM images shown in Figure 3 were obtained on a thermoplastic vulcanizate containing rubber, polypropylene, and carbon black filler. The (regular) phase channel shown in the center panel provides information about mechanical sample heterogeneity that is not necessarily apparent from sample topography (left panel). Phase images of this thermoplastic vulcanizate sometimes exhibit a fine lamellar structure indicating surface regions enriched with polypropylene (see also *Advanced Nanoscale Characterization with Caliber: Phase Imaging of Polymer Materials).*

While the phase image in the center panel of Figure 3 was extracted from the layered imaging file at 0nm lift height (i.e., on the surface), the right panel shows phase data obtained at 50nm lift height. Note the completely different appearance of the two phase images, suggesting that different information is being provided. The data shown in Figure 3 was obtained at a sample bias of 5V. Removal of the sample bias has virtually no effect on the topography and regular phase data but causes the complete disappearance of the (EFM) phase contrast in the right panel, confirming its electrostatic origin.

The thermoplastic vulcanizate imaged in Figure 3 contains carbon black filler particles that are known to form conductive networks. Applying a bias voltage between probe and sample causes the charging of this conductive network. The numerous dark round features seen in the EFM image indicate the locations of carbon black filler particles near the sample surface. With the probe not actually penetrating below the sample surface, EFM provides a nondestructive technique for obtaining information about the density and aggregation state of a subsurface component

SCANNING ELECTROSTATIC POTENTIAL MICROSCOPY

Scanning Electrostatic Potential Microscopy is used to map the electrostatic potential on the sample surface. On diNanoScope systems, this capability is often referred to as Surface Potential or Kelvin Probe Microscopy. SEPM employs an AC voltage bias between probe and sample. Note that this differs from the EFM (see previous section), where electrostatic interactions lead to phase changes of a mechanically driven probe. The implementation of SEPM on Caliber differs from its NanoScope counterpart in that lift mode is not used. Instead, the AC bias application is performed at an off-resonant frequency, ω, concurrently with mechanical excitation and feedback operating at the resonance frequency, ω_0. Driving and detecting at two separate frequencies necessitates two separate lock-in amplifiers, which is a standard feature on the Caliber system.

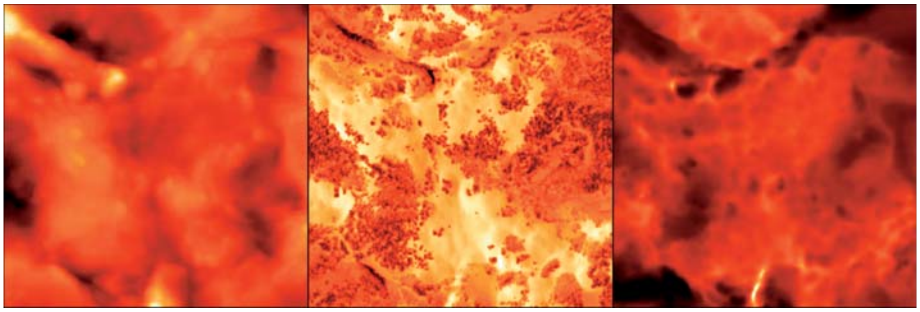
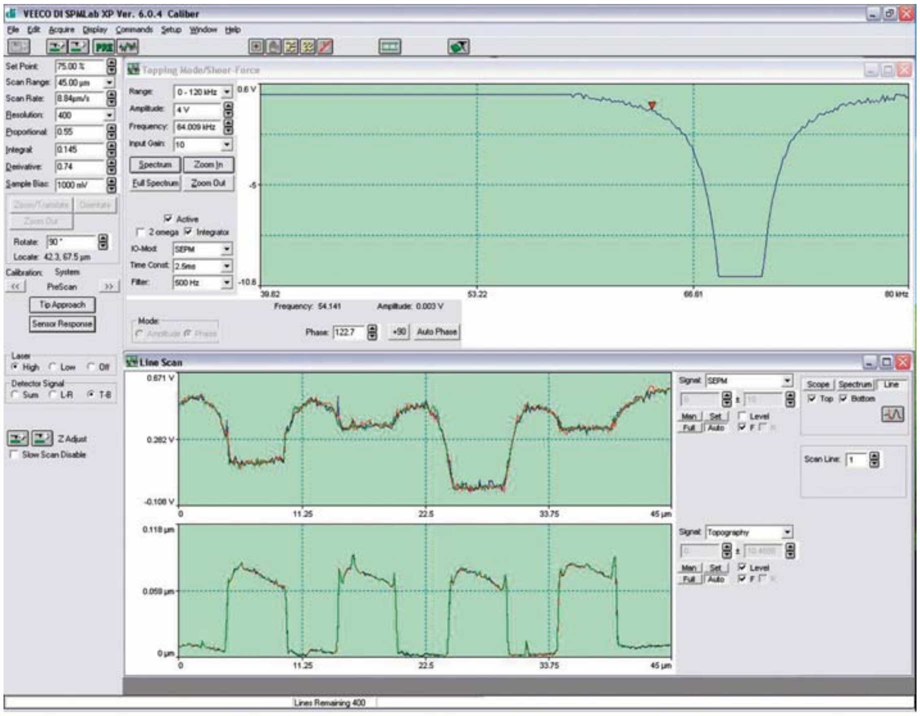


Figure 3: Topography (left), phase (center), and EFM phase (right) of a composite sample, containing rubber, polypropylene, and carbon black filler. The EFM phase image reveals the location of carbon black particles near the surface. Image size 4μm.



VEECO DI SPMLab XP Ver. 6.0.4 Caliber

Set Point: 75.00 %
Scan Range: 45.00 μm
Scan Rate: 8.84μm/s
Resolution: 400
Proportional: 0.55
Integral: 0.145
Derivative: 0.74
Sample Bias: 1000 mV
Rotate: 90 °
Locate: 42.3, 67.5 μm
Calibration: System

Tapping Mode/Shear-Force

Range: 0 - 120 kHz
Amplitude: 4 V
Frequency: 64.009 kHz
Input Gain: 10
☑ Active
☐ 2 omega ☑ Integrator
IO-Mod: SEPM
Time Const: 2.5ms
Filter: 500 Hz
Frequency: 54.141 Amplitude: 0.003 V
Phase: 122.7

Laser: ● High ○ Low ○ Off
Detector Signal: ○ Sum ○ L-R ● T-B
☐ Slow Scan Disable

Line Scan

Signal: SEPM
Signal: Topography
☑ Top ☑ Bottom
Scan Line: 1

Lines Remaining 400

Figure 4: Screenshot showing operation of SEPM mode. The tapping mode dialog shows the parameter setup for SEPM. The oscilloscope window shows topography and SEPM data. The probe is tracking across a serious of 4 conductors that can be seen to be at different potentials.

Two versions of SEPM are available with Caliber. The simpler open-loop SEPM only entails application of an AC bias to the probe and detection of probe oscillation amplitude at the bias frequency ω. Only when a DC potential offset is present, does the AC bias produce a driving force at frequency ω. Thus, the cantilever oscillation amplitude at ω can serve as a qualitative measure of surface potential variations. In the case of closed-loop SEPM, the cantilever oscillation at ω serves as an error signal for a feedback loop driving an additional DC bias. The DC bias therefore provides a quantitative measure of the electrostatic potential present in the region of space around the probe. When the voltages are applied to the sample, as is done on Caliber by default, the recorded SEPM data represents the sample potential variation with inverted sign.

Figure 4 shows a screenshot of the Caliber user-interface in SEPM mode. The SEPM lock-in and feedback parameters are adjusted in the TappingMode dialog shown in the top half of the screenshot.[1] The line scan window in the bottom half of the screen shows both, topography and SEPM data. Four equally tall features can be clearly seen in the topography channel, as the probe is accurately tracking across four metal conductors that are part of a test structure on a semiconducting substrate. While topographically similar, the four conductors are at different electrostatic potentials. With SEPM, the potential at the location of each conductor can be determined. In Figure 4, the SEPM measurement shows alternating electrostatic potentials in line with expectations. Thus, SEPM can aid in failure analysis of semiconductor devices by revealing the presence of short circuits or broken connections .

SUMMARY

With Magnetic Force Microscopy (MFM), Electric Force Microscopy (EFM), and Scanning Electrostatic Potential Microscopy (SEPM) Caliber offers nondestructive AFM techniques for detecting divergent magnetic and electric fields and variations in electrostatic potential on insulating, conducting, and semiconducting materials. As with other AFM modes, the characterization can be conveniently performed at ambient conditions. Applications range from failure analysis of semiconductor devices to quantifying electrical and magnetic properties of materials in research.

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[1] To gain access to these parameters, add “SEPM” to “HardwareOptions” in the [Acquisition] section of the topospm.ini file and add the line “IOModinstalled=IOMod0, IOMod1” to the [Flipper] section in the stages.ini file

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Printed in U.S.A. AN102, Rev. A0 3/07