The World Leader In Scanning Probe Microscopy

Phase Imaging: *Beyond Topography*

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Phase Imaging is a powerful extension of TappingMode™ Atomic Force Microscopy (AFM) that provides nanometer-scale information about surface structure often not revealed by other SPM techniques. By mapping the phase of the cantilever oscillation during the TappingMode scan, phase imaging goes beyond simple topographical mapping to detect variations in composition, adhesion, friction, viscoelasticity, and perhaps other properties. Applications include identification of contaminants, mapping of different components in composite materials, and differentiating regions of high and low surface adhesion or hardness. In many cases phase imaging complements lateral force microscopy (LFM) and force modulation techniques, often providing additional information more rapidly and with higher resolution. Phase imaging is as fast and easy to use as TappingMode AFM — with all its benefits for imaging soft, adhesive, easily damaged or loosely bound samples — and is readily implemented on any MultiMode™ or Dimension™ Series SPM with NanoScope® III controller equipped with an Extender™ Electronics Module.

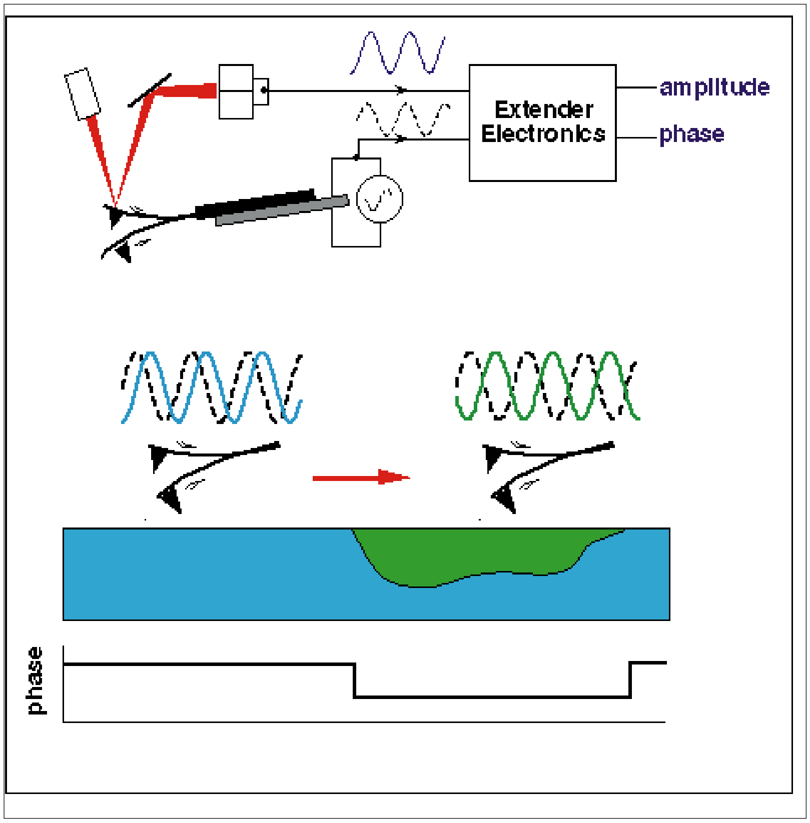

Figure 1: Phase imaging uses the Extender Electronics Module to measure the phase lag of the cantilever oscillation (solid wave) relative to the piezo drive (dashed wave). The amplitude signal is used simultaneously by the NanoScope III controller for TappingMode feedback. Spatial variations in sample properties cause shifts in the cantilever phase (bottom) which are mapped to produce the phase images shown here.

How It Works

In TappingMode AFM, the cantilever is excited into resonance oscillation with a piezoelectric driver. The oscillation amplitude is used as a feedback signal to measure topographic variations of the sample (see the TappingMode application note available from Digital Instruments). In phase imaging, the phase lag of the cantilever oscillation, relative to the signal sent to the cantilever's piezo driver, is simultaneously moni-

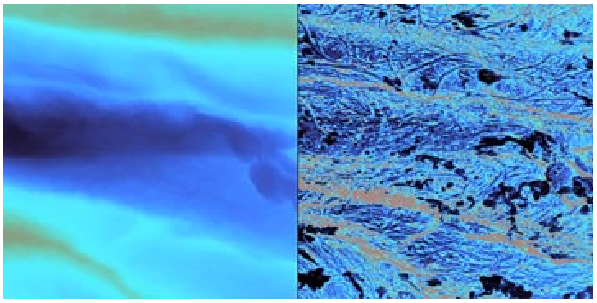

Figure 2: Phase (right) and TappingMode (left) images of wood pulp fiber. The phase image highlights cellulose microfibrils. In addition, a lignin component appears as light areas in the phase image, but is not apparent in the topography image. 3µm scan by Don Chernoff, Advanced Surface Microscopy; sample provided by Dr. Damaris E. Doro Pereira, Aracruz Celulose.

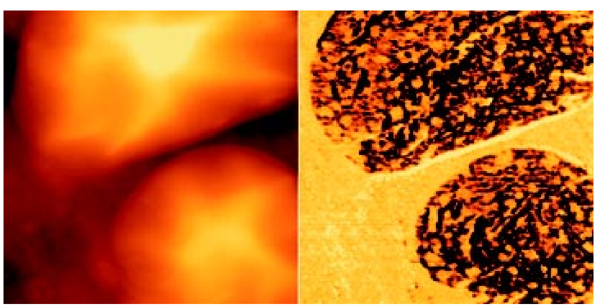

Figure 3: TappingMode (left) and phase (right) images of a composite polymer embedded in a uniform matrix. The high resolution of the phase contrast image highlights the two-component structure of the composite regions. Sample courtesy of Raj Michael.

tored by the Extender Electronics Module and recorded by the NanoScope III SPM controller (see our Extender Electronics application note). The phase lag is very sensitive to variations in material properties such as adhesion and viscoelasticity.

Once the SPM is engaged in TappingMode, phase imaging is enabled simply by displaying a second image and selecting the Phase data type in the NanoScope software. Both the TappingMode topography and phase images are viewed side-by-side in real time. The resolution of phase imaging is comparable to the full resolution of TappingMode AFM. Phase imaging can also act as a real-time contrast enhancement technique. Because phase imaging highlights edges and is not affected by large-scale height differences, it provides for clearer observation of fine features, such as grain edges, which can be obscured by rough topography.

Examples

Phase imaging is useful for differentiating between component phases of composite materials. The bulk of the wood pulp shown in Figure 2 consists of cellulose microfibrils that are highlighted by phase imaging. In addition, a lignin component — not apparent in the topography — appears as areas of light contrast atop the cellulose component. Similarly, the two-phase structure of the polymer blend in Figure 3 is shown clearly by the high resolution of phase imaging, even as TappingMode tracks relatively large (~1µm) topographical variations.

Phase imaging can also identify surface contaminants and evaluate processing steps. The integrated circuit bond pad in Figure 4 is covered with remnant spots of polyimide that an etching process failed to remove. Note that the polyimide is barely visible in the topographic image, but produces very large phase shifts (~120 deg). In Figure 5, the phase image of a human hair shows endocuticle debris (dark region) left behind by the erosion of the overlying cuticle in the lower left. Such information may help compare the effectiveness of various cosmetic treatments. Figure 6 shows topography and phase images of a SiO_2 crucible in which boules of crystalline Silicon were grown at 1450˚C. The imaged area corresponds to a region of characteristic Si contamination, visible as brown rings to the naked eye, which prevents re-use of these crucibles. Although no phase contrast was anticipated in this case, the phase image shows a complex multi-component structure which may serve as a signature of the contamination and give clues as to the nature of the contamination process.

A combination of these applications is shown by the images of a composite magnetic recording head in Figure 7. These heads typically have two magnetic pole pieces separated by a narrow diamagnetic gap. The central phase shows a section of a pole piece and the gap, with the gap appearing as a light band across the top. This distinction is not apparent in the topography. Note also the vertical string of contaminants in the first two images, shown in close-up by the right phase image. The phase contrast of the contaminants matches that of the gap material, suggesting that this material comprises the contaminants. For comparison, contact LFM produced no useful contrast, and the contaminants were swept away during imaging.

What information does phase imaging give about a sample? The MoO_3 crystallites in Figure 8 produce large contrast in the phase image relative to the MoS_2 background. The LFM image of the same area shows that the crystallites have relatively strong surface friction and adhesion,[1] suggesting that phase imaging is sensitive to these properties. Also, the advantages of TappingMode for imaging delicate samples allow phase imaging to be done in the many cases where the lateral forces of LFM can cause sample damage and produce poor resolution.

Differences in surface modulus and viscoelasticity also appear to produce phase contrast. For example, force modulation indicates that the polymer composites in Figure 3 have greater elasticity than the matrix in which they are embedded.

Other tip-sample interactions may also affect the phase signal. Although there is currently no simple correlation between phase contrast and a single material property, the examples shown here demonstrate that phase imaging gives valuable information for a wide range of applications, in some cases giving contrast where none was anticipated from the material properties.

Summary

Phase imaging is a powerful tool for mapping variations in sample properties at very high resolution. It can be turned on while using TappingMode AFM with no cost in speed or resolution, and all NanoScope users are encouraged to add it to their SPM repertoire. Phase imaging can complement force modulation and LFM methods, often with superior image detail, and can in some cases provide information not revealed by these or other SPM techniques. The rapidly growing list of phase imaging applications includes characterization of composite materials, mapping of surface friction and adhesion, and identification of surface contamination. Phase imaging promises to play an important role in the ongoing study of material properties at the nanometer scale.

[1] Y. Kim and C. M. Lieber, *Machining oxide thin films with an atomic force microscope: pattern and object formation on the nanometer scale,* Science 257 (1992) p.375.

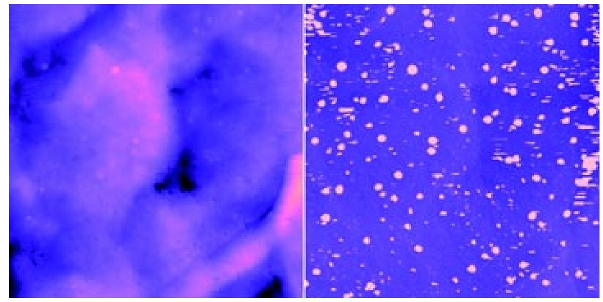

Figure 4: Bond pad on an integrated circuit imaged by TappingMode (left) and phase (right). Areas of the pad contaminated with polyimide produce light contrast with phase shifts of over 120 deg. 1.5µm scan.

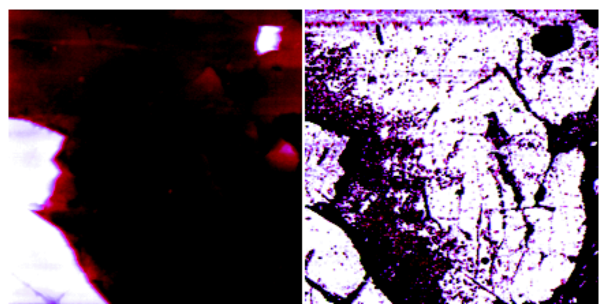

Figure 5: TappingMode (left) and phase (right) images of human hair cuticles. The phase image shows remnants of endocuticle (dark region) left behind as the upper cuticle (lower left) flaked away. Sample courtesy of JoAnne Crudele, Helene Curtis. 10µm scan.

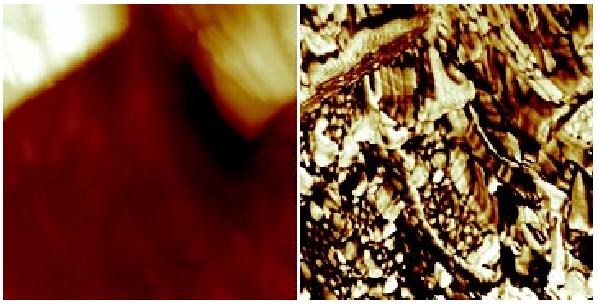

Figure 6: 2.5µm images of a quartz crucible used to grow crystalline silicon at high temperature. The multi-component structure in the phase image (right) is likely caused by Si contamination of the crucible that prevents its re-use. Sample provided by Dr. Robert Fisher, University of Washington.

Figure 7: Composite magnetic recording head; 3µm TappingMode and phase images (left and center), and 1 µm phase close-up of contaminants (right). Phase imaging distinguishes the magnetic pole piece (dark) from the gap; the latter appears as a white band across the top of the central phase image. A vertical string of contaminants shows light phase contrast matching that of the gap, suggesting the gap material as the origin of the contamination. Images courtesy Don Chernoff, Advanced Surface Microscopy.

Figure 8: Phase (left) and lateral force (right) images of MoO_3 crystallites on a MoS_2 substrate; 6µm scans. The crystallites show light contrast in the phase image, and dark contrast, corresponding to high friction, in the LFM image. Phase gives superior feature detail, and also shows dark regions on the background likely caused by contamination or adsorbed water. Sample provided by Dr. C.M. Lieber, Harvard University.

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