TappingMode™ Imaging
Applications and Technology

The World Leader In Scanning Probe Microscopy

By: C.B.Prater, P. G. Maivald, K. J. Kjoller, M.G. Heaton

Figure 1: TappingMode image of purified collagen monomer and oligomer molecules without telopeptides. 2µm scan courtesy Advanced Surface Microscopy, Indianapolis, IN.

Introduction

TappingMode imaging is a key advance in atomic force microscopy (AFM) of soft, adhesive or fragile samples. This patented technique allows high resolution topographic imaging of sample surfaces that are easily damaged, loosely held to their substrate, or otherwise difficult to image by other AFM techniques. Specifically, TappingMode overcomes problems associated with friction, adhesion, electrostatic forces, and other difficulties that can plague conventional AFM scanning methods. The technique has proven extremely successful for high resolution imaging of a wide variety of samples including:

- silicon wafer surfaces
- thin films
- metals and insulators
- photoresist
- polymers
- biological samples

and numerous others (e.g., Figure 1). TappingMode makes imaging these surfaces routine in ambient air or fluids and represents a significant advance in AFM technology.

Two conventional scanning modes — contact mode and non-contact mode — have been used for some time with varying success for a range of materials. Each has limitations which are discussed below and contrasted with TappingMode scanning.

Conventional Methods

In conventional contact mode AFM (Figure 2), the probe tip is simply dragged across the surface and the resulting image is a topographical map of the surface of the sample. While this technique has been very successful for many samples, it has some serious drawbacks. The dragging motion of the probe tip, combined with adhesive forces between the tip and the surface, can cause substantial damage to both sample and probe and create artifacts in image data.

Under ambient air conditions, most surfaces are covered by a layer of adsorbed gases (condensed water vapor and other contaminants) which is typically several nanometers thick. When the scanning tip touches this layer, capillary action causes a meniscus to form and surface tension pulls the cantilever down into the layer (Figure 3). Trapped

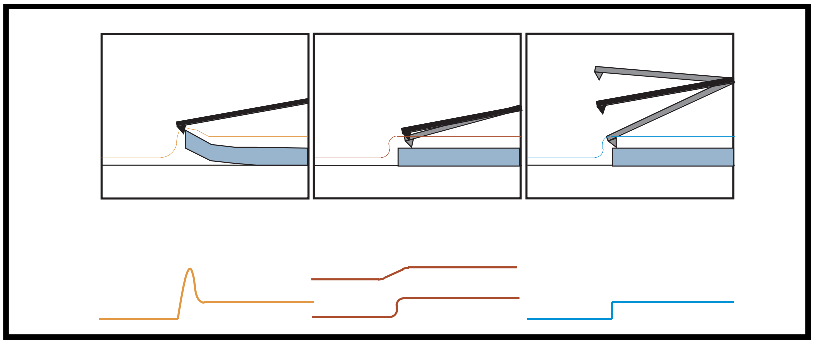


Figure 2: Comparison of contact mode, non-contact mode and TappingMode scanning techniques. Contact mode imaging (left) is heavily influenced by frictional and adhesive forces which can damage samples and distort image data. Non-contact imaging (center) generally provides low resolution and can also be hampered by the contaminant layer which can interfere with oscillation. TappingMode imaging (right) eliminates frictional forces by intermittently contacting the surface and oscillating with sufficient amplitude to prevent the tip from being trapped by adhesive meniscus forces from the contaminant layer. The graphs under the images represent likely image data resulting from the three techniques.

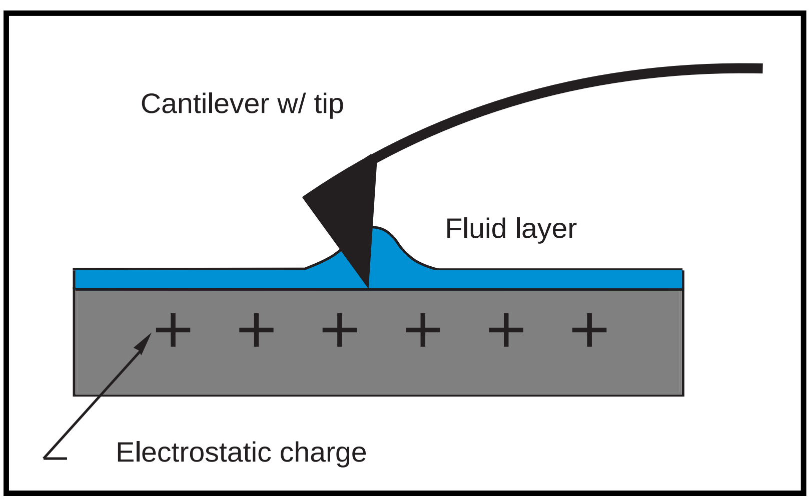


Figure 3: In contact AFM, electrostatic and/or surface tension forces from the adsorbed gas layer pull the scanning tip toward the surface.

electrostatic charge on the tip and sample can contribute additional adhesive forces. These downward forces increase the overall force on the sample and, when combined with lateral shear forces caused by the scanning motion, can distort measurement data and cause severe damage to the sample, including movement or tearing of surface features.

Some researchers have overcome the problems associated with the adhesive forces by operating AFMs with the sample immersed in fluid. When scanning in fluids, the overall forces in contact mode are lower than in ambient air because the fluid layer/meniscus is not present and electrostatic forces can be dissipated or screened. However, because hydrated samples are often substantially softer than dried samples, tracking forces can still cause reduced image quality and sample damage due to deformation and/or movement of the sample by the scanning probe. In addition, many samples, such as semiconductor wafers, can not practically be immersed in fluid.

An attempt to avoid this problem is the non-contact mode in which the probe is held a small distance above the sample (Figure 2). Attractive Van der Waals forces acting between the tip and the sample are detected, and topographic images are constructed by scanning the tip above the surface. Unfortunately, the attractive Van der Waals forces from the sample are substantially weaker than the forces used by contact mode — so weak in fact that the tip must be given a small oscillation so that AC detection methods can be used to detect the small forces between tip and sample. The attractive forces also extend only a small distance from the surface, where the adsorbed gas layer may occupy a large fraction of their useful range.

Hence, even when the sample-tip separation is successfully maintained, non-contact mode provides substantially lower resolution than either contact or TappingMode. In practice, the probe is frequently drawn to the sample surface by the adsorbed gases' surface tension, resulting in unusable data and sample damage similar to that caused by the contact technique. In addition, the non-contact mode is generally impractical for routine scanning in fluids because the Van der Waals forces are now even smaller, a substantial limitation for biological samples in particular.

TappingMode Imaging in Air

TappingMode imaging overcomes the limitations of the conventional scanning modes by alternately placing the tip in contact with the surface to provide high resolution and then lifting the tip off the surface to avoid dragging the tip across the surface. TappingMode imaging is implemented in ambient air by oscillating the cantilever assembly at or near the cantilever's resonant frequency using a piezoelectric crystal. The piezo motion causes the cantilever to oscillate with a high amplitude (the "free air" amplitude, typically greater than 20nm) when the tip is not in contact with the surface. The oscillating tip is then moved toward the surface until it begins to lightly touch, or "tap" the surface. During scanning, the vertically oscillating tip alternately contacts the surface and lifts off, generally at a frequency of 50,000 to 500,000 cycles per second. As the oscillating cantilever begins to intermittently contact the surface, the cantilever oscillation is necessarily reduced (Figure 4) due to energy loss caused by the tip contacting the surface. The reduction in oscillation amplitude is used to identify and measure surface features.

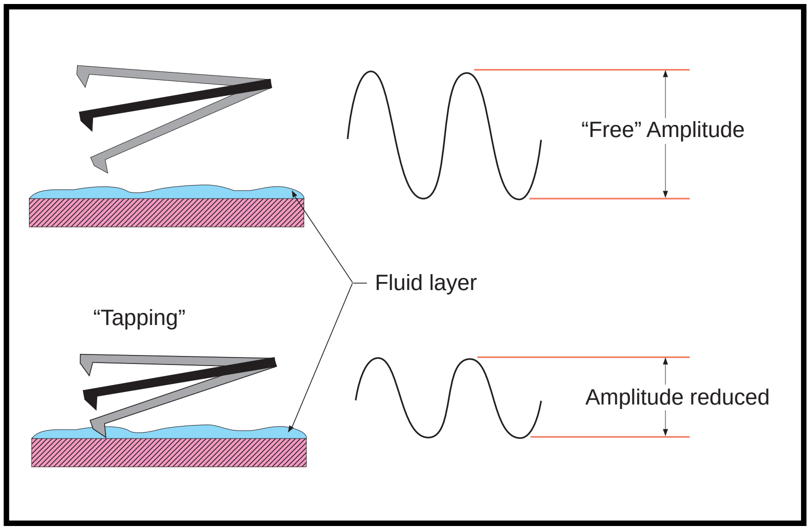
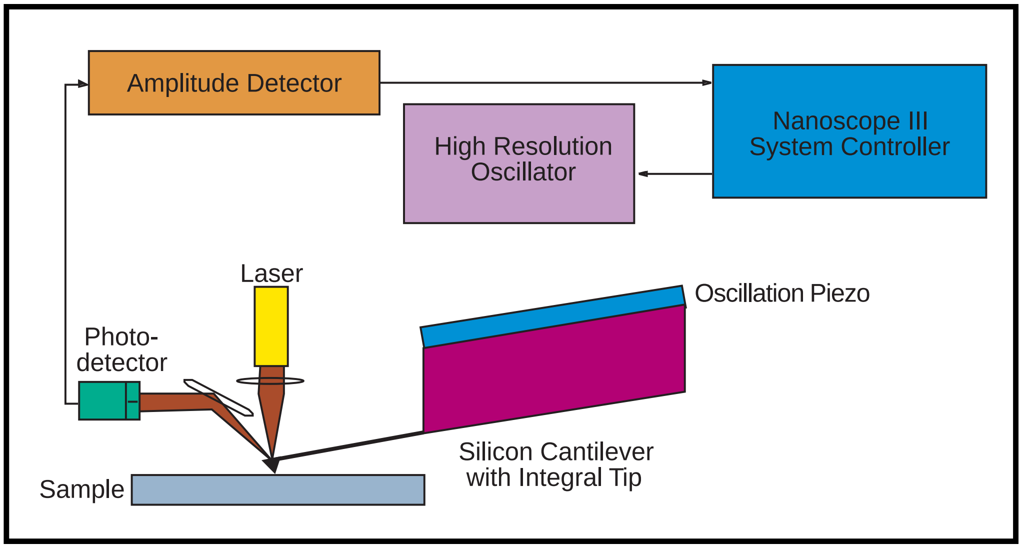


Figure 4: TappingMode cantilever oscillation amplitude in free air and during scanning.

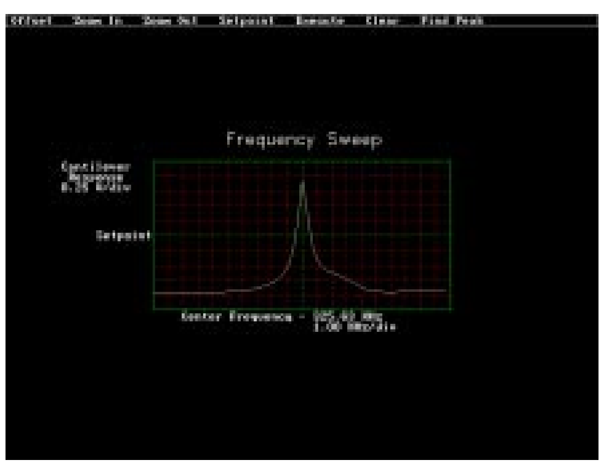
Figure 5: Block diagram for TappingMode operation.



During TappingMode operation, the cantilever oscillation amplitude is maintained constant by a feedback loop (Figure 5). Selection of the optimal oscillation frequency is software-assisted and the force on the sample is automatically set and maintained at the lowest possible level (Table 1 and Figure 6). When the tip passes over a bump in the surface, the cantilever has less room to oscillate and the amplitude of oscillation decreases. Conversely, when the tip passes over a depression, the cantilever has more room to oscillate and the amplitude increases (approaching the maximum free air amplitude). The oscillation amplitude of the tip is measured by the detector and input to the NanoScope III controller electronics. The digital feedback loop then adjusts the tip-sample separation to maintain a constant amplitude and force on the sample.

TappingMode inherently prevents the tip from sticking to the surface and causing damage during scanning. Unlike contact and non-contact modes, when the tip contacts the surface, it has sufficient oscillation amplitude to overcome the tip-sample adhesion forces. Also, the surface material is not pulled sideways by shear forces since the applied force is always vertical (see sidebar on inside back cover for additional discussion of tip-sample forces).

Figure 6: The cantilever tune screen assists the operator in selecting the optimum TappingMode oscillation frequency.



Another advantage of the TappingMode technique is its large, linear operating range (Figure 7). This makes the vertical feedback system highly stable, allowing routine reproducible sample measurements. Several references which discuss TappingMode imaging are listed at the end of this application note.

TappingMode Imaging in Fluids

Similar advantages are realized with TappingMode operation in fluids. In this case, however, the fluid medium tends to damp the cantilever's normal resonant frequency. Instead, the entire fluid cell can be oscillated to drive the cantilever into oscillation. When an appropriate frequency is selected (usually in the range of 5,000 to 40,000 cycles per second), the amplitude of the cantilever will decrease when the tip begins to tap the sample, similar to TappingMode operation in air.

Once the cantilever is set into oscillation, the NanoScope III digital feedback system adjusts the position of the tip to maintain a constant oscillation amplitude. Again as in air, the oscillating cantilever eliminates frictional and shear forces on the sample. In addition, the process of repetitively contacting the surface and pulling the tip off at a high rate allows the tracking force to be maintained constant at a minimum value.

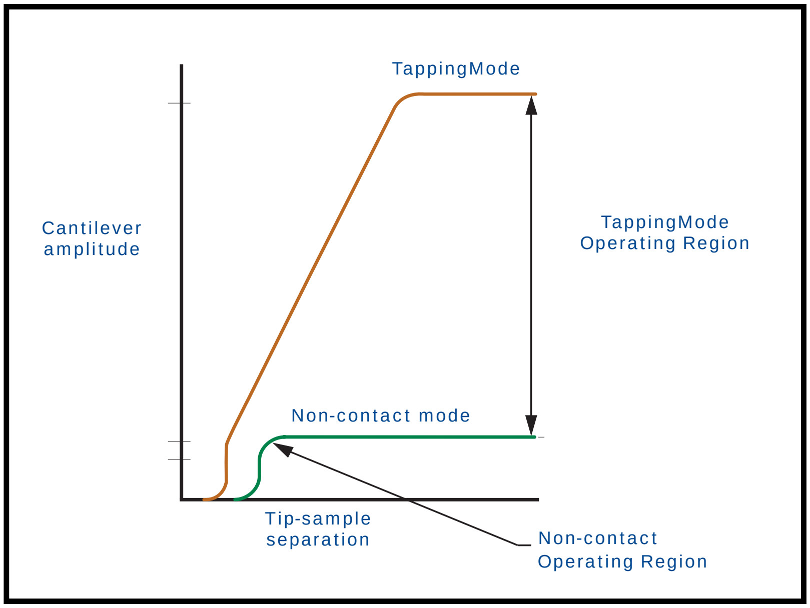


Figure 7: Comparison of large linear operating range for TappingMode vs. small operating range for non-contact mode.

Drive Frequency Range	10KHz to 1MHz
Drive Voltage Range	0-20V_{pp} with 1mV RMS noise level
Drive Amplitude and Frequency Adjustment	Digitally selected. Software control and display of TappingMode parameters allows fast, semi-automated on-screen optimization
Detector	RMS-to-DC amplitude detector provides phase-independent amplitude signal; Noise level > 0.5Å RMS
Cantilevers	Etched silicon cantilevers; 60-400KHz resonant frequencies
Tip-Sample Approach	Motorized sample approach automatically brings cantilever into TappingMode operation at lowest possible tracking force

Table 1: TappingMode Specifications.

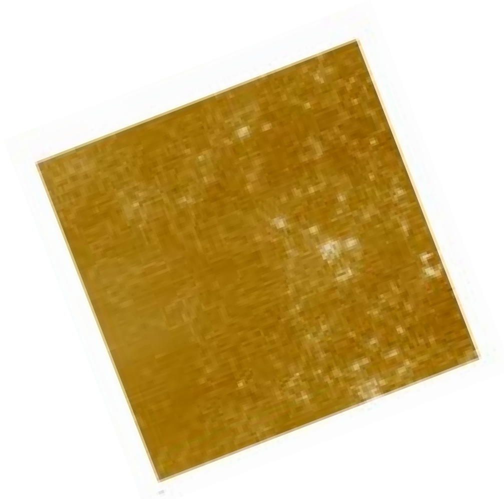


Figure 8: TappingMode image scanned in air of kinetoplast DNA from the trypanozome of a malarial parasite. 2µm scan courtesy Oak Ridge National Labs, Oak Ridge, Tennessee.

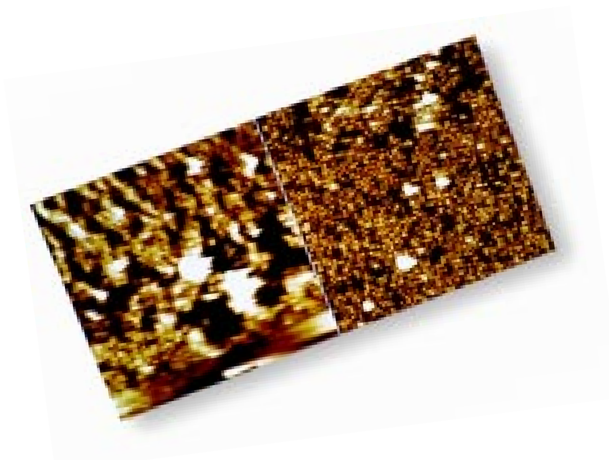


Figure 9: Comparison of contact mode (left) and TappingMode (right) images of RNA polymerase scanned in fluid (buffer). Note that the streaks and haziness common to even low force liquid contact mode are not present in the fluid TappingMode image. 1µm scan; sample courtesy Helen Hansma, University of California, Santa Barbara.

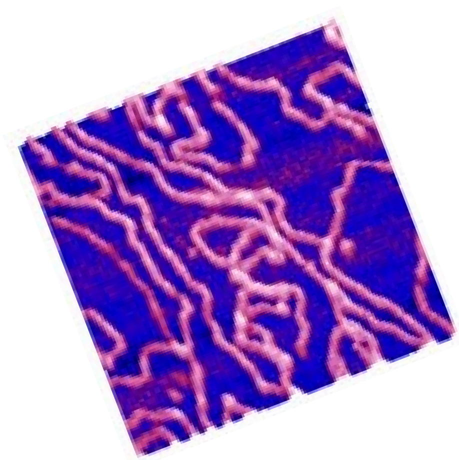


Figure 10: Lambda Hind III DNA imaged on mica with TappingMode in water. The sample was scanned continuously for over one hour without damage. Contact mode scanning of the same material caused damage in less than one minute — before the scan could be completed. 500nm scan courtesy M. Bezanilla, University of California, Santa Barbara.

TappingMode avoids the force instabilities caused by thermal drift in contact mode, resulting in time savings and improved image and measurement quality. Stable imaging forces of less than 200pN have been measured during TappingMode operation. Refer to the sidebar (right) for additional discussion of tip-sample forces.

Examples

Figures 8 through 14 illustrate the capabilities of TappingMode for imaging a variety of soft surfaces. Figures 8 through 10 show biological samples imaged in both fluid and air, illustrating the dramatic improvement in image quality for TappingMode relative to conventional contact mode in both environments.

Figure 11 illustrates the capabilities of TappingMode relative to contact mode for semiconductor materials using side-by-side comparisons. Figures 12 through 14 are TappingMode images for a polymer and two thin films.

Summary

To obtain quality images, it is critical that the microscope tip not damage the surface being scanned but that it contact the surface to obtain high resolution measurements. This is where TappingMode imaging excels. For many materials, this technique provides the highest resolution possible without sample damage. TappingMode imaging has opened a wide variety of applications and continues to expand the applicability of SPM to new materials and surfaces.

More on Tip-Sample Forces in TappingMode

One of the key advantages of TappingMode imaging over conventional contact AFM is the low forces generated during scanning. Because the tip only contacts the surface briefly during each oscillation, there are no lateral frictional forces applied to the sample by the tip that can tear the sample, distort data or dull the tip.

The brief contact force is less than one might expect. In TappingMode the cantilever is oscillated at or near its resonant frequency. Once the cantilever amplitude is stabilized at the desired setpoint, the sample must absorb only the small force due to the increased amplitude during a single oscillation cycle; i.e., the time between two consecutive “taps.” Because the cantilevers used in TappingMode have a high quality factor (“Q”), the amplitude gained in one cycle is only about 0.01nm under typical imaging conditions. The force due to this small amplitude increase can be absorbed by the vast majority of samples with no damage to tip or sample. Because of these gentle scanning forces, TappingMode has been used successfully to reproducibly image such samples as polymers, unbaked photoresist and DNA, as well as numerous other fragile samples. Also, we have repetitively imaged the angstrom-level microroughness of the same 1µm region of a silicon wafer continuously over a 24-hour period without degradation of the image or damage to the sample.

Finally, the cantilever is oscillated at frequencies from 50KHz to 500KHz. At these frequencies, many surfaces become stiff (viscoelastic) and can more easily resist forces from the probe tip. This property further reduces the possibility of sample damage for extremely soft samples such as polymers, biological specimens, and others and causes less distortion of the sample due to tip forces.

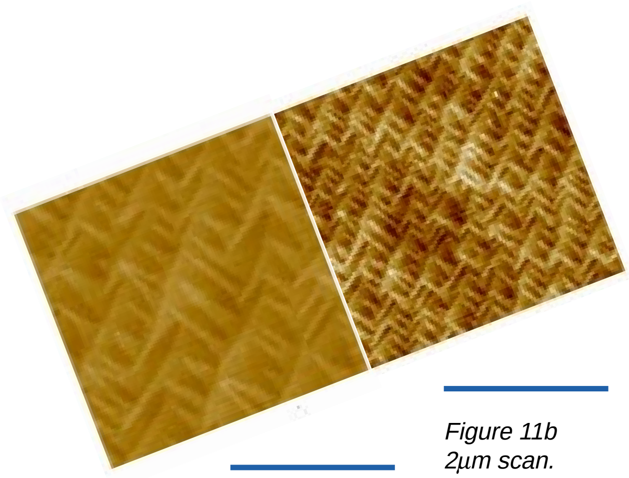


Figure 11a 1µm scan. TappingMode.

Figure 11b 2µm scan. TappingMode.

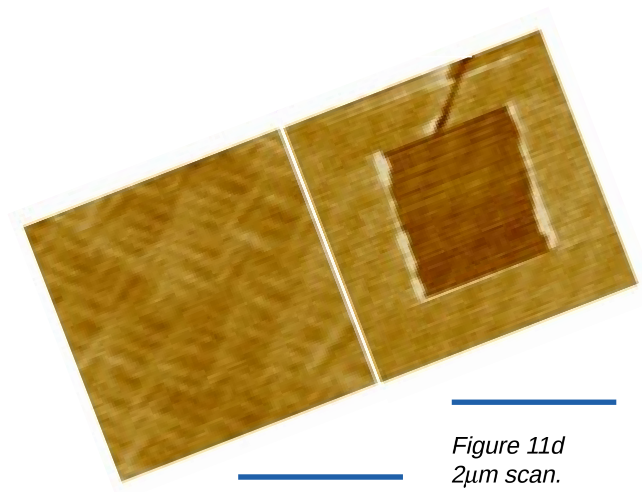


Figure 11c 1µm scan. Contact mode.

Figure 11d 2µm scan. Contact mode.

Figure 11: Contact and TappingMode images for the same (100) epitaxial wafer. In both cases, the left image was taken first and the scan size was immediately doubled and re-scanned to include the area imaged in the first scan. The TappingMode images show no surface alteration and better resolution. Conversely, the damaged area of the first scan can be easily seen in Figure 11d. Contact mode imaging is extremely inconsistent for silicon surfaces; in this case material has been removed by the scanning tip, while in other cases, additional oxide growth or more subtle changes may occur. This type of surface alteration often goes undetected since most researchers do not check for damage by rescanning the affected area at lower magnification.

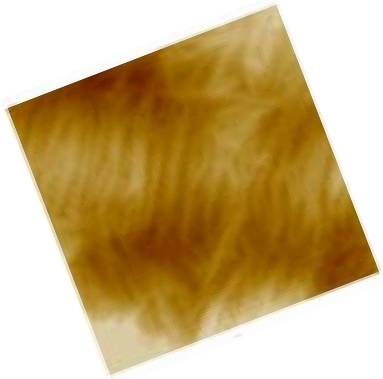


Figure 12: TappingMode image of high density polyethylene from a shopping bag. The structures in the image are the polymer lamellae which are approximately 30nm thick and all oriented in the same direction to increase the tensile strength. This structure could not be seen with contact mode since the features were altered by the tip dragging across the surface. 675nm scan.

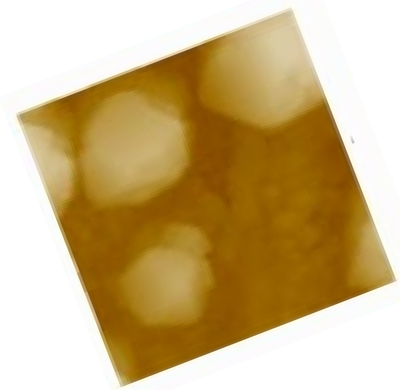


Figure 13: Chemical vapor deposited (CVD) diamond film. During film formation, seed crystals of diamond are placed on a silicon wafer which is then placed in the CVD deposition chamber in which growth is initiated to produce the thin film. This image shows the film at early initiation of growth. The TappingMode technique was used to more accurately profile the crystals and to avoid moving the seed crystals on the surface. 1µm scan. Sample courtesy of Stanford University.

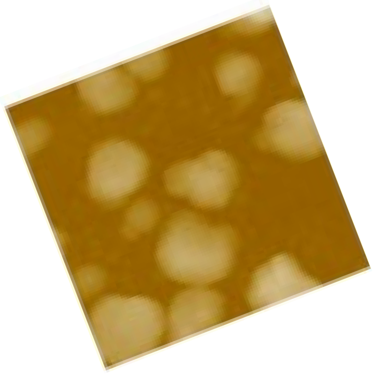


Figure 14: Thermally evaporated gold film, 60Å thick, deposited onto an oxidized silicon wafer. The films were used to build strain sensors with higher strain sensitivity than continuous films. 500nm scan courtesy L. ChunShien, P. Hesketh, and G. Maclay, University of Illinois at Chicago.

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A current bibliography of TappingMode AFM references is available on request from Digital Instruments.

Digital Instruments

520 East Montecito Street
Santa Barbara, California 93103
T: (800) 873-9750
T: (805) 899-3380
F: (805) 899-3392
Email: info@di.com

Distributors W orld W ide