Electrical testing of soft delicate samples using Torsional Resonance Mode and TUNA®

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Torsional Resonance and TR-TUNA are covered by the following patents:
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INTRODUCTION

Scanning tunneling microscopy (STM) employs a biased sharp metal probe in close proximity to a surface. When the tip-sample separation is small, there is a finite probability that electrons will tunnel across this gap. As the tip is scanned across the sample the tunneling current is utilized as a feedback signal in order to maintain the tip-sample separation. STM can be used to measure properties of metals, semiconductors and other materials having medium to high conductivities.

Since STM utilizes the tunneling current as the feedback signal the technique suffers from one significant drawback. The sample being scanned must have some electrical conductivity to allow the feedback loop to operate throughout the scan. In general, STM cannot be used to scan an electrically conductive region within an insulating matrix. In order to overcome this shortcoming the tunneling atomic force microscope (TUNA®) has been developed.

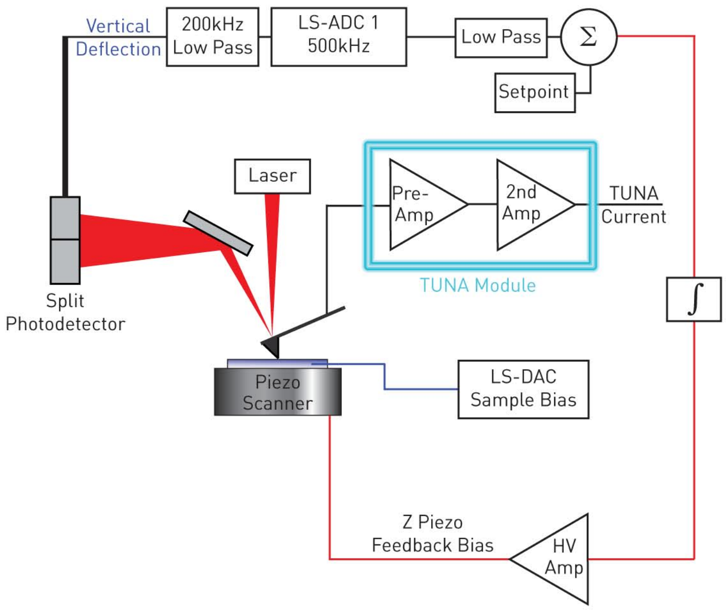

Figure 1. The configuration of the AFM when used for collecting conventional TUNA data. The system uses a low-passed vertical deflection for feedback.

TUNNELING ATOMIC FORCE MICROSCOPY

The TUNA® sensor from Veeco Instruments, shown schematically in figure 1, is designed to measure ultra-low currents on low-conductivity samples. A low voltage DC bias is applied between the sample and a conductive tip as the tip is scanned across the sample in Contact Mode.

As with regular Contact Mode imaging, the cantilever deflection is used as a feedback source in order to maintain a constant tip-sample force. During scanning, any tip-sample current present is passively measured using a linear current amplifier with a current range of 60fA to 1mA. Thus, the TUNA® technique is immune to conductivity variations that would quickly destroy an STM tip. In this way, the sample's topography and the tip-sample current are measured simultaneously, enabling a direct correlation of sample location with its measured electrical properties.

TUNA® is especially useful for the evaluation of thin dielectric films where the current tunneling between the tip and the dielectric film strongly depends on film thickness, leakage paths (possibly caused by defects) and charge traps. All of these may significantly affect the properties and the integrity of the whole film.

TUNA® can be applied to many research or manufacturing areas on a wide range of materials. However, the use of Contact Mode for topographic feedback is a limiting factor. For samples that require low imaging forces, in either or both the vertical or lateral directions, Contact Mode may not be applicable. It is therefore difficult to extend TUNA® imaging to the study conductive polymers, organics or other soft conducting materials or loosely bound samples such as nanowires.

TORSIONAL RESONANCE MODE

Atomic force microscopy has achieved tremendous benefits from a variety of oscillating tip modes of operation, most notably Tapping Mode®. During Tapping Mode® the AFM cantilever is oscillated at its fundamental flexural resonance. This has the advantage of largely eliminating lateral forces that tend to damage the tip and/or sample when imaging in contact mode. The vertical interaction force is also substantially reduced due to high mechanical Q of the cantilever, permitting imaging of delicate soft samples.

When imaging in Tapping Mode®, the AFM tip position varies relative to the sample by tens of nanometers, and may spend up to 99% of its oscillation cycle in a regime having no near-field interaction with the surface, figure 2. This is advantageous for eliminating tip wear and sample damage, but is problematic for the measurement of properties that demand near-field interactions – such as TUNA®.

It is known however, that AFM cantilevers can oscillate at many different modes[1–10], including higher order flexural and torsional or twisting modes. These extended modes enable the study of widely differing tip–surface interactions. Consider the case of imaging with the cantilever oscillating in the first torsional resonance mode.

In this mode, lateral forces that act on the tip cause a change in the torsional resonant frequency, amplitude, and/or phase of the cantilever[11]. AFM measurements at torsional resonances have many advantages but the key advantage for TUNA® is the ability to achieve low-force scanning whilst maintaining the tip in the near-field.

With the ability to maintain the probe tip in the near-field (at the boundary between direct contact and the long-range force regime), we are once again able to measure tunneling currents between the tip and sample.

The implementation of a torsional drive system for use with AFMs from Veeco Instruments is a simple modification to the cantilever holder found on all Tapping Mode® enabled AFMs. The holder employs two parallel actuators, marked as piezos 1 and 2 in figure 3.

To excite the torsional resonance, drive signals of opposite phase are applied to these two piezos. Due to the asymmetry of the tip mass about the rotation axis this actuation will create a rotation of the cantilever substrate about its center point. Since the tip is far away from the center, the trajectory of the tip is almost horizontal. As with conventional Tapping Mode® the torsional cantilever deflection is detected using a four-segment photodetector.

AFM uses the angular displacement of the cantilever and the resulting change in the reflected laser beam angle at the photo-detector. For flexural bending of a cantilever, the bend angle θ_f is given approximately by the relationship:

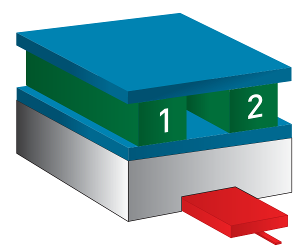

Figure 3. The TR probe holder employs two parallel actuators using drive signals of opposite phase.

The cantilever length serves as the lever arm that converts vertical

$$\theta_f = \frac{3}{2}\frac{z}{L}$$

where:
z is the vertical tip deflection
L is the length of the cantilever

deflection to an angular change. Typical values of cantilever lengths are on the order of 100µm. In this case the tip needs to move at least a few nanometers to generate an angular deflection signal large enough to be detected.

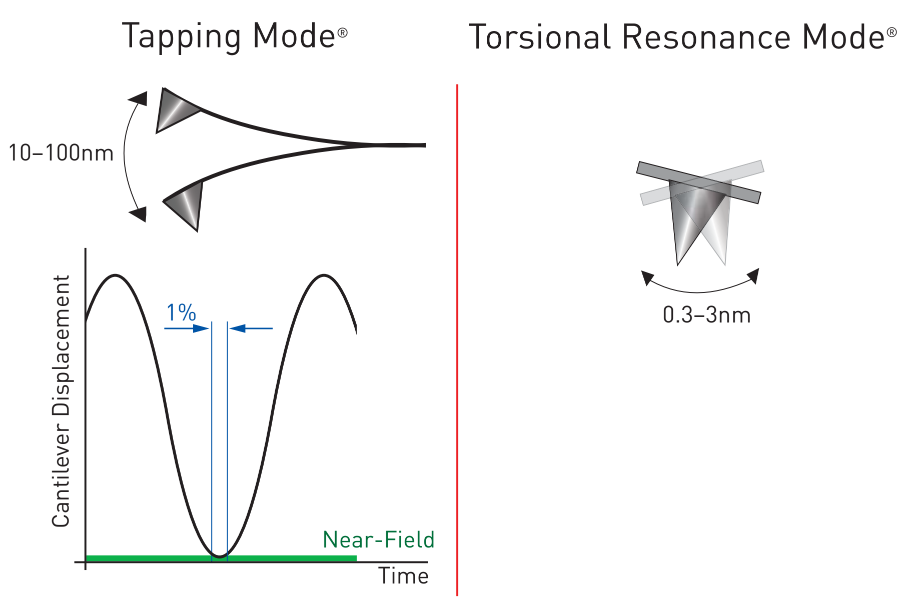

Figure 2. The tip is in close proximity to the sample for approximately 1% of the oscillation cycle when using standard Tapping Mode. In comparison the tip can be in close proximity to the surface all of the time when using Torsional Resonance Mode with small oscillation amplitudes

The lever arm for torsional motion is much shorter as it corresponds to the tip height plus half the cantilever thickness (this is the distance from the tip apex to the center of torsional rotation). AFM tip heights are typically 10 to 20 times shorter than the cantilever length L. So torsional detection is 10 to 20 times more sensitive to tip motion than flexural detection. As a result it is much easier to detect sub-nanometer tip motion laterally with the same detection electronics and optics.

The increased torsional detection sensitivity translates into typical tip oscillation amplitudes of the order of a couple of nanometers or less. Compare this to the 30-100nm oscillation amplitudes typically used for Tapping Mode® and it is easy to see why the cantilever tip remains in the near-field during the entire imaging process. The remainder of the feedback system used for TR-Mode® imaging is identical to that used with Tapping Mode®.

TR TUNA®

Two samples that are difficult to image in contact mode are presented here using TR-Mode® for topographical feedback.

The first sample consists of a loosely distributed network of carbon nanotubes that are weakly-bound to a gold on silicon substrate. The substrate was in the form of a silicon wafer with a non-conducting native oxide over it's surface. Gold was patterned to generate a conducting grid on to which was placed the network of conducting carbon nanotubes from suspension.

Due to the loosely-bound nature of the nanotubes to the gold/silicon oxide surface this sample is not able to be imaged using standard contact mode based TUNA® techniques (where high lateral forces simply push the nanotubes around the surface). With either Tapping Mode® or TR-Mode® it is possible to obtain stable images of the nanowires.

Topography from this sample is presented as figure 4 (left). Part of the gold grid can be seen in the upper right-hand quadrant of the image.

The current image, figure 4 (right), shows the tunneling current as measured by the TUNA® sensor. It can be seen that only those nanotubes that have an electrical connection to the gold grid exhibit contrast. Only those nanowires directly connected to the gold grid, not simply overlapping each other, form a conductive path between the imaging probe and the counter-electrode.

The second sample imaged with a 10μm scan was a carbon-black filled polymer that is also difficult to image in contact mode. When imaged in Tapping Mode® the topography is recovered but an applied tip-sample bias of up to 10 Volts yielded no measurable current data.

When scanning in TR-Mode®, not only is the topography, shown as figure 5, measured correctly, but in addition significant contrast is observed in the TUNA® current. This TUNA® current is detected with a tip-sample bias as low as 0.5 Volts. Variations in the measured TUNA® current for biases of –0.5, –1.0 and –2.0 Volts are given as figure 5.

There are only a few bright spots in figure 5 (b), which indicates the conductive areas at this sample bias voltage. With increasing sample bias voltage, the effective barrier for tunneling is reduced. Therefore, the total area of the sample exhibiting a measurable TUNA® currents is seen to increase.

With two simple samples the unique strength of applying TR-Mode® to TUNA® current measurements on soft delicate samples is clearly demonstrated. It can be seen that the use of TR-Mode® extends the sample space available to TUNA® to include those samples traditionally believed

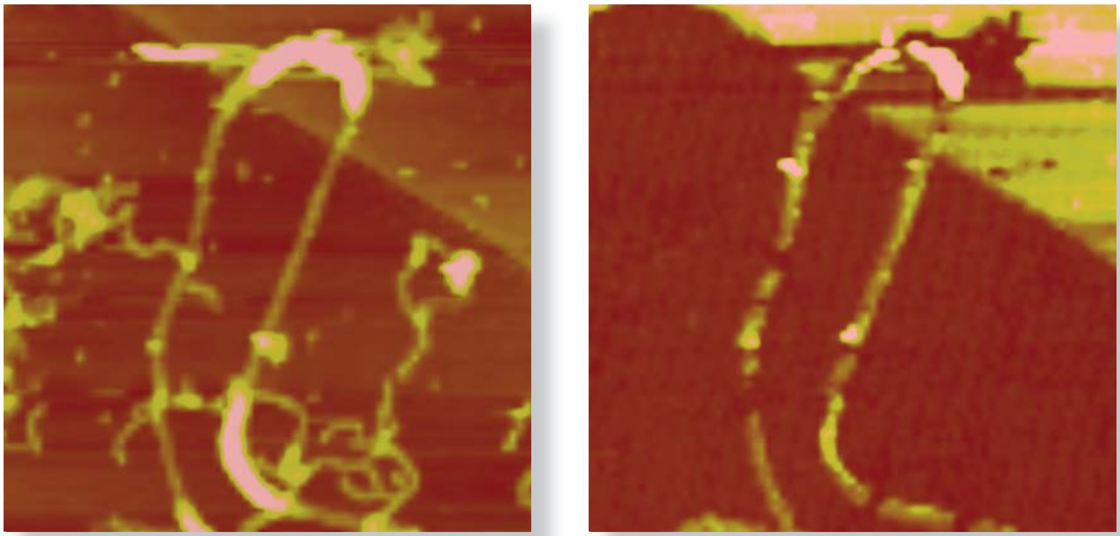

Figure 4 Carbon nanotubes deposited from solution on to a gold grid are shown as topography (left) and current (right). The TUNA® data shows only those nanotubes that are electrically connected directly to the gold grid and not simply connected to each other.

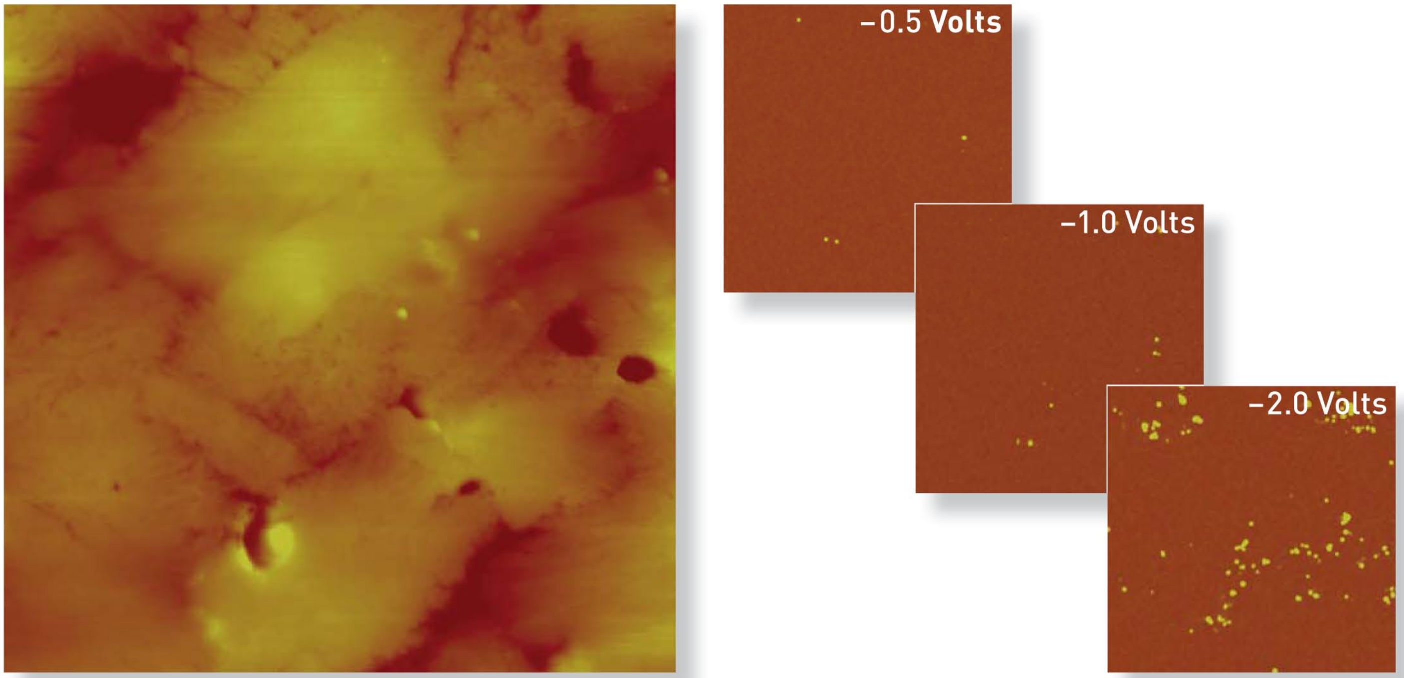

Figure 5 Carbon black topography (left) shows the ability of TR-Mode® to image soft samples excluded by contact mode. No measurable TUNA current was detected with tip-sample biases of up to ±10 Volts with Tapping Mode® but significant current was measured for bias between 0 and –2 Volts (right) when imaged using TR-Mode®.

too soft or delicate for Contact Mode imaging and hence TUNA®. This now includes conducting polymers, loosely bound nano-materials and organic thin-films as well as more traditional thin-film samples such as low-k dielectrics.

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