RHK Technology Brief

Application ✲ Tutorial ✲ Technology

Intermittent-Contact AFM Imaging with RHK Technology System - *SPM 100 Versatility Shines in Data Flexibility*

Kapila Wadu-Mesthrige, Jayne Garno and Gang-Yu Liu

Department of Chemistry, Wayne State University Detroit, MI 48202

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Principle

In conventional contact mode imaging, the AFM tip is always in contact with the surface. When imaging in air, the total force, or load, includes both the deformation force due to the cantilever bending and the adhesive force or capillary interaction. Such force can be quantitatively determined from the approach and retreat curve, also known as the force curve. Typically, images are acquired in the repulsive interaction region, where the cantilever bends upwards. During the scan, the tip deformation is maintained constant via regulating the height of the sample. The output of the z-piezo voltage of the sample manifests itself in the topographic image, and the imaging mode under this configuration is defined as "constant force mode." For high-resolution imaging and low deformation of the surface, one should reduce the total as well as the deformation force. Thus, the highest resolution AFM images, e.g. images with true molecular resolution, were all acquired in liquid media, where the adhesive force is reduced dramatically.

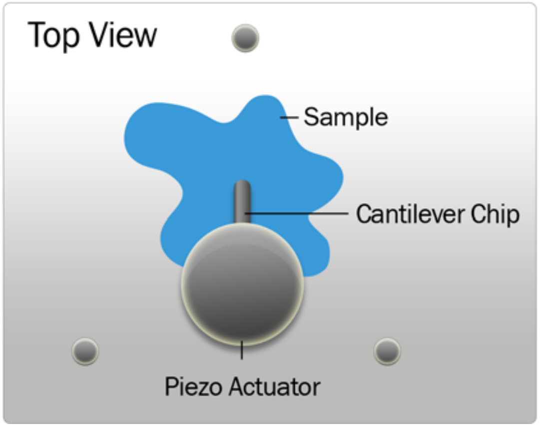

Figure 1. *Details of AFM cantilever holder*

For soft materials, especially biological samples, the tip-surface adhesive interaction is strong, causing (i) large deformation of the surface at contact, and (ii) tip-surface sticking. As a result, stick-and-slip motion of the tip occurs during the scan, which produces spikes in the AFM images. Thus, the resolution for these soft materials is generally poorer than inorganic surfaces, or thin organic films.

A few techniques have been developed to improve the resolution of soft materials such as cryogenic AFM, non-contact, or partial contact mode imaging. The most commonly used non-contact or partial contact technique is known as "intermittent contact" (IC) imaging. In IC mode, the cantilever is modulated on or near its natural resonance frequency (f0) and at a desired amplitude (ΔZ). When the tip is far above the surface ($>>\Delta Z$), the modulation amplitude and phase remain constant. When the tip is brought into the attractive regime or within the magnitude of ΔZ, the modulation amplitude decays sharply due to the shift in its natural resonance. Therefore, high sensitivity to surface features arises from the ability to detect this shift very precisely. The magnitude of the decay depends on the tip-surface interaction, which in turn, is determined by the modulation amplitude (ΔZ), tip-surface separation, and functionalities of both the tip and surface. The feedback circuit keeps the modulation amplitude constant (such as 50% of the original ΔZ) during image acquisition. In IC mode, the average distance between the tip and the surface is an amount corresponding to no contact; however, the tip may touch the surface periodically during modulation.

The advantages of IC mode are (1) the brief contact time between the tip and the surface, which effectively removes the stick-and-slip behavior; and (2) the average tip-surface distance remains in the non-contact regime, which reduces the surface deformation when there is brief contact. The disadvantage of this mode is that the images are acquired in the attractive regime, which is not as sensitive as the repulsive region used during contact mode imaging. In other words, the intrinsic resolution of IC mode is not as high as the contact mode for hard and nonadhesive surfaces. Therefore, we recommend that this mode be used only for "soft and sticky" samples.

Procedure with SPM 100

Many atomic force microscope systems can perform this type of IC imaging, but the SPM 100 from RHK Technology was built with maximum flexibility in mind. This design philosophy excels in these experiments which have unique constraints. Here we provide details where a home-built AFM scan head and RHK electronics (SPM 1000 and AFM 100) are used. Similar configurations can be easily adapted for various commercial scan heads.

1. Cantilever mounting_

The cantilever should be mounted onto a piezo actuator using either a mechanical mounting or a suitable glue such as Duco Cement (Devcon® |Duco Cement, Devcon Corp., Wood Dale, IL 60191). Depending upon the actual surface and imaging media, one may choose cantilevers with a desired force constant and geometry. To image proteins under ambient conditions, we choose a Si lever (e.g. Park Scientific, Ultralever) with a force constant of 2.2 N/m. In the home-built AFM used in the study outlined here, a disk shaped piezo actuator is used as shown in Figure 1. Since the cantilever is modulated by applying a sinusoidal voltage to the piezo actuator as illustrated in Figure 2, one needs to isolate the piezo disk and the cantilever holder. An insulating cantilever holder may be used, or a nonconductive coating must be added if a metal cantilever holder is used.

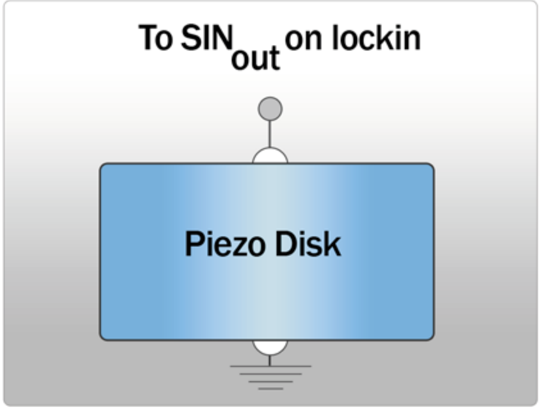

Figure 2. *Piezo Actuator*

2. Modulation Frequency_

The most straightforward method to determine the natural resonance frequency of a cantilever is to use the built-in FFT routine in the SPM 32 (or XPMPro) program. This is accessed through the Acquire/Spectroscopy/Noise Power menu item. The FFT of the input signal will be computed, and a graph similar to Figure 3 will be produced. Note this method can only be used if the resonance of the cantilever is below 74 kHz (125 kHz if the DT2821 250 kHz upgrade option is installed or XPMPro is used). Another method is to monitor the amplitude of the cantilever vibration with one of the Aux channels while scanning the driving frequency. When the cantilever is excited with a frequency near its resonance, the amplitude will start to increase and this will appear as an increased input signal on the Aux 1 channel. A typical free cantilever (Park sharpened, k = 0.1 N/m) noise spectrum is shown in Figure 3. A “stiff” Park ultralever with k = 2.2 N/m typically exhibits a resonance at 82 kHz.

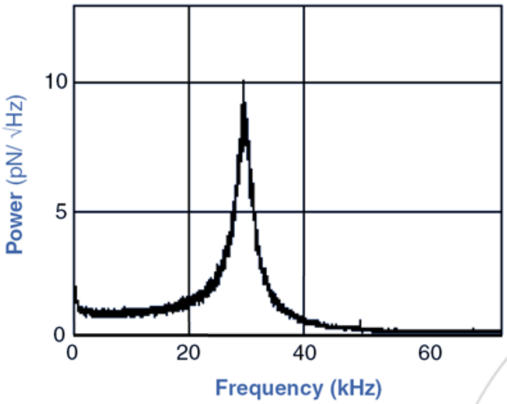

Figure 3. *FFT of cantilever oscillation*

The frequency spectra provide a good guide for intermittent contact conditions. The modulation frequency (f) should be on or very near the peak of the spectrum which is the natural frequency of the cantilever. The amplitude should be sufficiently high (especially under ambient conditions) in order to overcome the capillary forces and pull the cantilever off the surface after brief contact (~1nm). The variation of the waveform shown in the oscilloscope during approach can also be used to optimize ΔZ.

3. Imaging Conditions_

It is a simple transition from contact to IC mode imaging. In addition to the SPM 100 and AFM 100 electronics, a lock-in amplifier is required and its signal may have to be attenuated before inputting into the feedback electronics. The connection is illustrated in Figure 4. This lock-in amplifier provides complete control of the modulation signal and also superior performance can be achieved compared to other setups which have the lock-in amplifier integrated into the SPM control electronics. With the freedom to choose any external lock-in amplifier, a high quality unit can be used which provides both a greater bandwidth of exciting frequencies and a more sensitive response compared to an integrated unit whose specifications may not even be fully published.

To understand the connection, first follow the signal for feedback: SIN_{Out} of Lock In Amplifier ⟶ piezo disk (cantilever modulation) ⟶ preAmp ⟶ Input of AFM 100 ⟶ Normal Force Output of AFM 100 ⟶ Input of Lock In ⟶ (amplified) ⟶ CH1_{Out} (R) of Lock In ⟶ Attenuation Box (Gain = – 0.1 to –10) ⟶ External Input of AFM 100 (set for external/lock-in instead of error) ⟶ Feedback input of SPM 100. The wiring allows the amplitude of cantilever modulation to be the signal used for feedback. Second, the three imaging signal inputs should be topography, Aux1 (R from CH1 of Lock-in) and Aux 2 (Θ from CH2 of Lock-in). Third, an oscilloscope is used to monitor signals from Sin_{Out} of Lock-in, and Normal force from AFM 100.

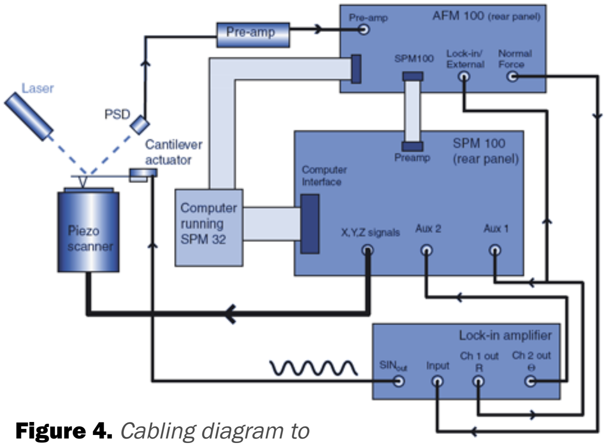

Figure 4. *Cabling diagram to perform intermittent contact measurements with RHK equipment.*

4. Force curve acquisition_

By using the SPM 100 coupled with an AFM 100 and an external lock-in amplifier as described above, it is trivial to acquire force-distance curves while oscillating the cantilever. The vast amount of flexibility offered by the RHK instrument permits an ease in acquiring this type of data that is unmatched by any competitors. Simply setup a spectroscopy routine to ramp the distance while recording the Preamp input which, in this case, is the signal from the photodiode. This would be what the SPM 100 considers the tunneling current signal so it is like setting up the unit to perform I-Z spectroscopy if operating an STM. We recommend the acquisition of this type of force curve to guide the determination of the setpoint as well as optimizing modulation conditions. A typical force curve acquired under these conditions is shown in Figure 5.

Each force curve contains three regions, (1) tip is far away from the surface where the cantilever modulation amplitude is constant; (2) “contact” region, where the modulation amplitude decays with the decreasing tip-surface distance; and (3) repulsive region, where the average tip-surface distance is in contact and the tip modulation is dramatically attenuated but not entirely eliminated. The working setpoint should be selected from somewhere within region 2. Because of the flexibility of the RHK system, any point along the curve can be selected as the setpoint simply by changing the feedback "current" setting. This allows the individual user to completely determine how hard to tap the surface. Since any point along the Y axis can be selected, the amplitude of the cantilever oscillation is known at that height above the surface. For instance, if the feedback setpoint is selected so the constant amplitude is towards the left side of region 2, the tip will just lightly touch the surface when it is at the far end of its amplitude swing. However, a different condition can be selected where the tip contacts the surface very early in its motion, thereby striking the sample quite vigorously; and the imaging mode approaches conventional contact mode. RHK offers the freedom of choosing the amount of contact whereas, other systems automatically select a setpoint. This freedom can prove very useful in utilizing different tapping conditions for different materials. There is a tradeoff between increasing the resolution while minimizing the sample damage which can be optimized with this setup. An analogy can be drawn of tapping egg shells with a hammer compared to tapping steel ball bearings: a slightly different "touch" is required in the two cases and only RHK can provide the ability to adjust this "touch".

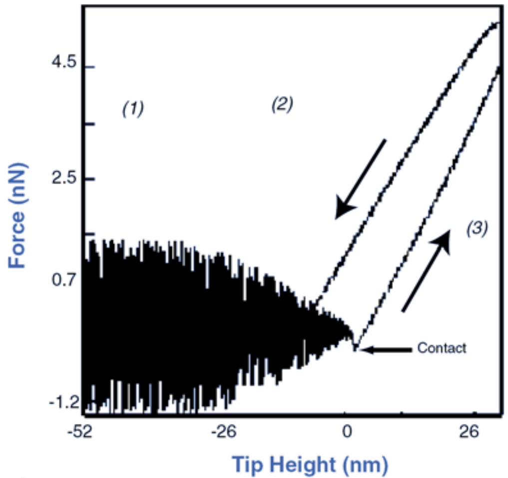

Figure 5.
Force-Distance curve taken with oscillating tip.

5. Imaging_ First, make sure the imaging input channels are Topography, Aux. 1 and Aux. 2. Second, reset the polarity for tapping mode imaging. Change the Z position polarity switch on the back panel of STM 100 to NEGATIVE, switch the polarity on SPM 100 front panel from positive to NEGATIVE position, change the FEEDBACK switch on the front panel of the AFM 100 from ERROR position to LOCKIN/EXTERNAL position.

Note that the set point value used for contact mode (AFM 100) is disabled during IC mode imaging. The set point is determined using the Current Set on the front panel of SPM 100. The initial current set point is typically 4.0 to 6.0 nA while the bias voltage is set to zero. A fast way to choose a good feedback setting is to approach the surface towards the cantilever while monitoring the cantilever modulation by displaying the output of the lock-in amplifier on an oscilloscope. As the tip and surface distance becomes sufficiently close, the modulation amplitude begins to decay. Stop approaching at the desired amplitude e.g. 50% of the original value. Adjust the Current Set until the feedback is in range. We recommend the z-voltage to be 50 V for sufficient imaging dynamic range.

An additional feature of the RHK electronics is the flexibility offered by using a separate lock-in amplifier and the SPM 100. With the ability of the SPM 100 to monitor 5 different channels during a scan, any of these signals from the lock-in can be used as the feedback signal. In theory, images can be obtained in constant phase, constant amplitude, or constant height mode. Images acquired in one of these other modes may unravel some mysteries concerning the nature of the surface provided that the data is interpreted properly as the interpretation of features in an image when taken in constant phase mode is different than interpreting features seen in constant amplitude mode. A question that must be addressed if using this constant phase mode is whether the contrast in this type of image would be interpreted the same as contrast in a phase image when acquired using conventional constant amplitude imaging. Two examples of intermittent-contact mode images taken in air are shown in Figure 6.

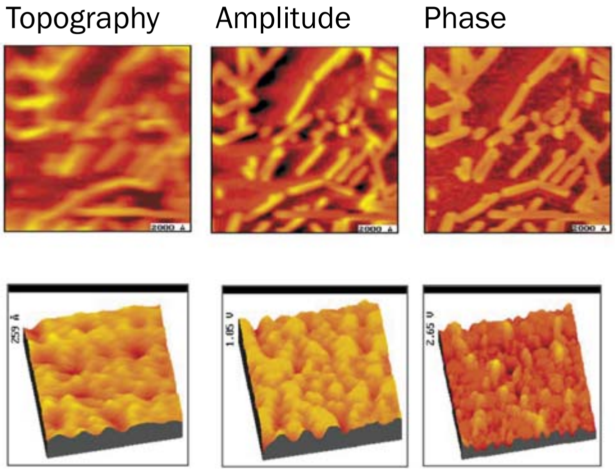

Figure 6.
Top, Tobacco Mosaic Virus on mica. Bottom, Ferritin on mica. Imaged in air using a cantilever with k=2.2 N/m

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RHK Technology Inc.
1050 East Maple Road
Troy, MI 48083
tel 248 577 5426
fax 248 577 5433

www.rhk-tech.com
info@rhktech.com