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Displacement Measurement of Thin Ferroelectric Films using an Atomic Force Microscope with an RT6000S

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We thankfully acknowledge Kevin Kjoller of Digital Instruments for his assistance in reviewing this article for correctness with respect to atomic force microscopes.

Summary:

Michelle Bell and I (Joe Evans) visited Digital Instruments on September 21 and 22, 1998. We met Kevin Kjoller who is the Manager of the Applications Lab.

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Kevin helped us hook an RT6000S to their Dimension 3100 AFM and take measurements of a 1µ 4/20/80 PNZT film we made in Radiant's clean room. The measurements were taken with PIEZO on an RT6000S using the AFM as the sensor. Our 1µ film showed ~35Å of displacement peak to peak during the tests. This is a strain of $3.5x10^{-3}$, a very large value!

The text below will describe how the system is hooked up, how to make a measurement, what not to do, and the sources of measurement error in the system.

AFM Operation:

The Atomic Force Microscope uses a small cantilever with an approximately 20 nm diameter tip as its sensor probe. The usual operation of the AFM is to measure surface topology with sub-angstrom level resolution by placing the tip close to or in contact with the sample surface and monitoring the displacement of the cantilever caused by the surface as the tip is scanned across it. The control system actuates a bulk piezoelectric actuator attached to the cantilever in order to hold the cantilever at the same height above, or with the same contact force against, the sample surface. See Figure 1 below:

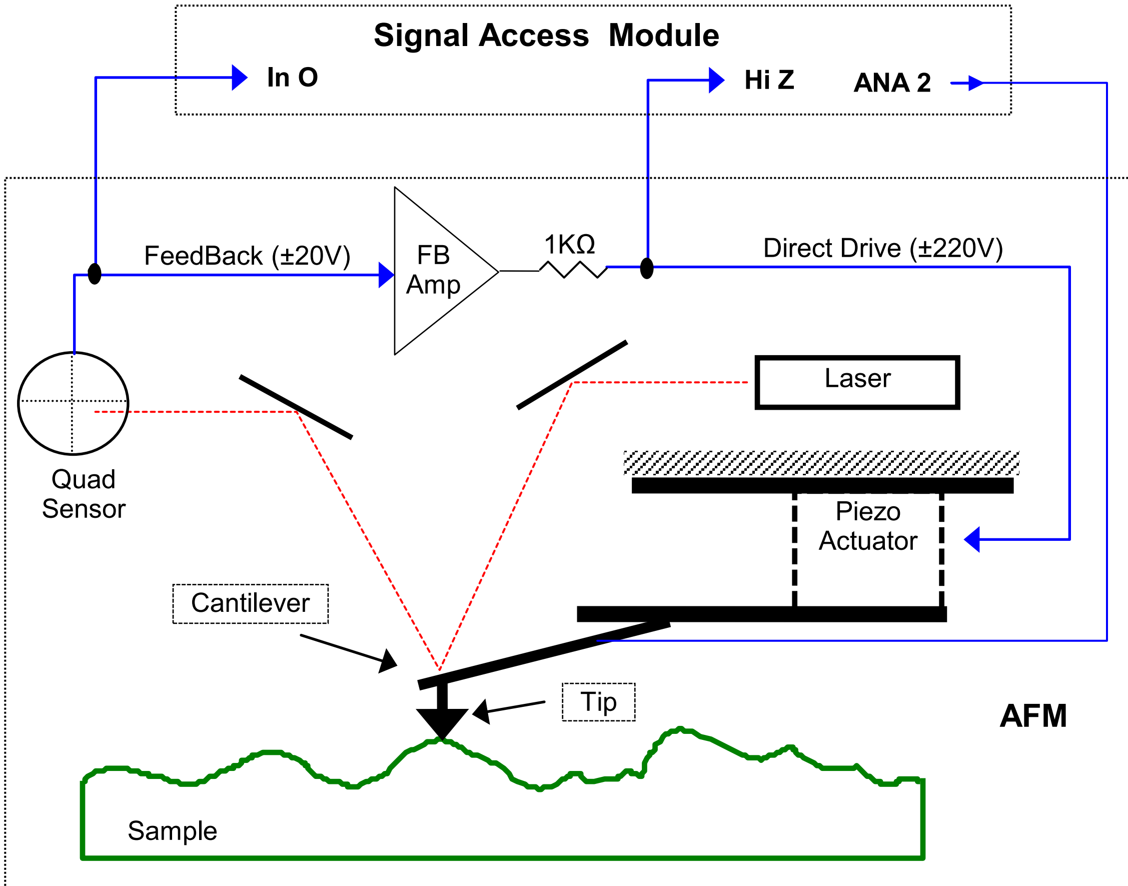

Figure 1
Digital Instruments Dimension 3000 AFM System Configuration

The **Signal Access Module** is an optional attachment to the AFM that allows the user to gain access to internal AFM signals.

If a metallized tip and conducting cantilever are used in the AFM, the RT6000 can make contact to the top electrode of a ferroelectric capacitor with the cantilever tip and electrically actuate the capacitor while monitoring the cantilever displacement. Since the AFM is sensitive to subangstrom vertical displacements, the thin film piezoelectric response for micron thick films is viewable. There are other possible measurement configurations as well:

1. Make contact to the capacitor top electrode with other connections and use a non-conducting AFM tip to measure displacement.

2. Use the AFM cantilever and tip to apply a calibrated force to the top electrode and measure the polarization generated by the capacitor. This is the classic piezoelectric measurement!

AFM Configuration

Both a Dimension 3100 and a Dimension 3000 were used for the testing. To the RT6000, either system is identical. A **Signal Access Module** is required on the AFM to do the PIEZO measurements. Additionally, unless the sample is in a total isolation environment, ambient sonic energy in the test area will oscillate the sensor tip with higher amplitudes than the film will generate during actuation. This includes speech! A Digital Instruments VT103 isolation chamber, which is an electrically and sonic isolated chamber sitting on a vibration isolation table, had to be used to eliminate sonic noise sources. To measure thin ferroelectric films, this isolation chamber is absolutely necessary.

There is one set of jumpers on the circuit board inside the rear cover of the AFM. These jumpers must be set a certain way in order to direct the **ANA2** input BNC of the **Signal Access Module** to the AFM cantilever. The chuck should be grounded to prevent it from injecting noise into the measurement. Figure 2 below shows the jumper settings used. Please refer to the NanoScope system manual when changing the jumpers.

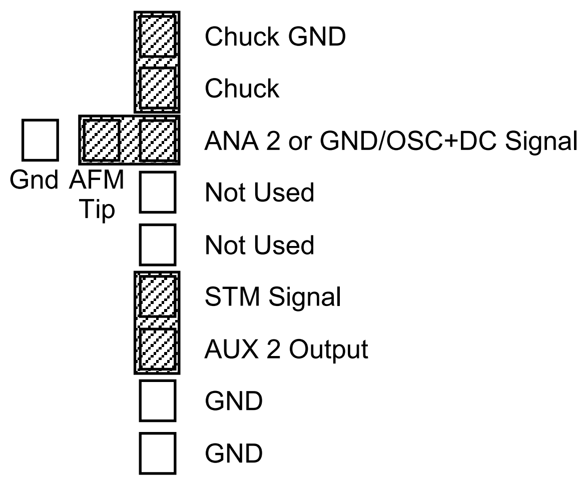

Figure 2
AFM Jumper Settings for the Piezo Measurement with the RT6000
(These are the only jumpers on the AFM circuit board.)

The AFM operator must do a noise test of the system prior to making piezoelectric measurements. This is a standardized test the system runs with a calibrated sample to look for any unacceptable noise sources. In our case, there was a strong 12Hz oscillation in the system that came from a vacuum leak in the sample chuck. The chuck had not been installed correctly. Kevin re-installed the chuck and the noise dropped to the certified limit. That limit is a rms value of 0.5Å, acceptable for thin film measurements.

The cantilever used was a standard 225µ length with a metallized tip, model MESP. A two times increase in the system sensitivity can be achieved by using a short 125µ cantilever with metallized tip. In the best configuration, the cantilever tip must have a metal **Sphere** attached to it to minimize surface noise from the system drift. (See below)

Sample Description:
The sample used in the experiment was a 4/20/80 PNbZT film fabricated at Radiant. The film was 1 micron thick deposited as 12 layers of 800Å each. The bottom electrode was global high temperature platinum 1500Å thick. The PNZT remained unetched except for a large spot near the flat intended for the bottom electrode electrical connection. The top electrode was patterned with the TE mask layer in 1000Å of platinum. A rough estimate of the expected displacement from the sample was 10Å.

A small, metal cat whisker was attached to the bottom electrode through the hole etched in the PNZT. The attachment was made with conductive silver epoxy. After the glue was cured, tape was placed over the whisker to give it mechanical strength. A 2 inch length of the whisker extended from the tape for connection to the RT6000 with micrograbber clips and coax cable.

RT6000 Configuration
The normal connections to the RT6000 for PIEZO measurements are the DRIVE and RETURN to the sample, the MCh1 to the sensor, and the ground plug to the test stand. For the AFM, the DRIVE was connected to the AFM tip through **ANA2** of the **Signal Access Module** (See **AFM Configuration**). The RETURN was connected to the sample cat whisker using a coax cable to a wire with a micro-grabber on one end. Tape was used to hold the wire to the VT103 frame to reduce its physical vibration of the microscope.

The MCh1 was initially connected to the **IN O** output of the AFM **Signal Access Module**. The **IN O** signal is taken from the **Feedback** signal shown in Figure 1. The output of the **IN O** signal from the AFM only generated 130mV for the displacement of the 1µ thick film. This resulted in too small of a Signal to Noise ratio to give good measurements (see below). The **Direct Drive** signal (labeled **Hi Z** on the **Signal Access Module**) was then used. It generated 10 times the output voltage of **IN O** and produced good results.

When sensing the **Hi Z**, the AFM operator must set the tip deflection so that the **Z-Center** value noted in software is at 0V. (**Hi Z** and **Z-Center** are essentially the same parameter.) Fluctuations caused by a 1µ film will be ±1V from zero. A 4µ film should generate a ±4V signal. If the **Hi Z** signal is allowed to exceed ±20V (as can happen when the tip is raised to move it to another location), the high voltage may blow the input stage of the MCh input amplifier of the RT6000. <u>A protection circuit must be used between the **Hi Z** output and the MCh1 input (see below). This protection circuit can be acquired from Radiant.</u>

A final connection that was most important was to connect the RT6000 earth ground (the green banana plug) to the AFM test equipment and test stand. Great care was required here to limit the electrical noise in the polarization hysteresis curve.

Measurements
We first tried the measurements on a Dimension 3100 situated on an air table without any isolation from the ambient sound environment. We found that the ambient noise level far exceeded the piezoelectric displacement of the film. Our voices could be seen in the AFM signal! We moved to a Dimension 3000 system with a VT103 isolation chamber. We did achieve a butterfly loop from the film on this system using the **In O** output but the Signal to Noise (S:N) was only about 5:1. See Figure 3 below.

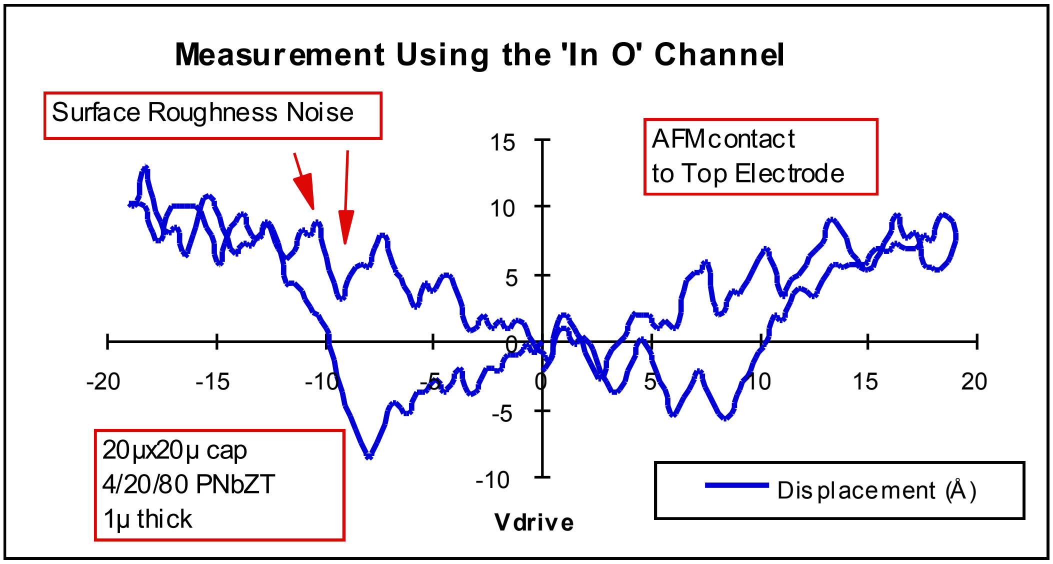

Figure 3
Piezo Butterfly Loop of 1µ PNZT Film Measured using In O Channel

Figure 3 shows that we could measure the displacement but we had more work to do to identify and eliminate noise.

During the second day, we experimented or corrected the following things and achieved a significantly improved signal:

1. Executed a standard noise test on the AFM, identified the improperly installed chuck, and corrected the problem so that the certified noise limit of 0.5Å rms was achieved.

2. Switched the MCh to the **HI Z** channel of the **Signal Access Module** and achieved a 10 times increase in signal with a 10 times reduction of S:N.

3. Connected both the AFM microscope in the VT103 as well as the outside frame of the VT103 to the RT6000 earth ground to minimize electrical noise.

4. Tried various cantilever configurations including a short cantilever and a stainless steel cantilever. The standard metallized 225µ cantilever worked best. We did not have available a tip with a large sphere installed on it.

5. Tried a cantilever with a pigtail electrical connection connected directly to the RT6000 instead of going through **ANA 2** of the **Signal Access Module**. There was no difference in electrical noise but the wire attachment to the pigtail induced vibration in the cantilever needle. Do not use the pigtail cantilever module.

With the final configuration:

1. DRIVE = **ANA2**
2. RETURN = Cat Whisker
3. MCh1 = **Hi Z (Direct Drive)**
4. 225µ metallized cantilever

we measured a 20µx20µ capacitor on the sample with PIEZO and achieved the following results:

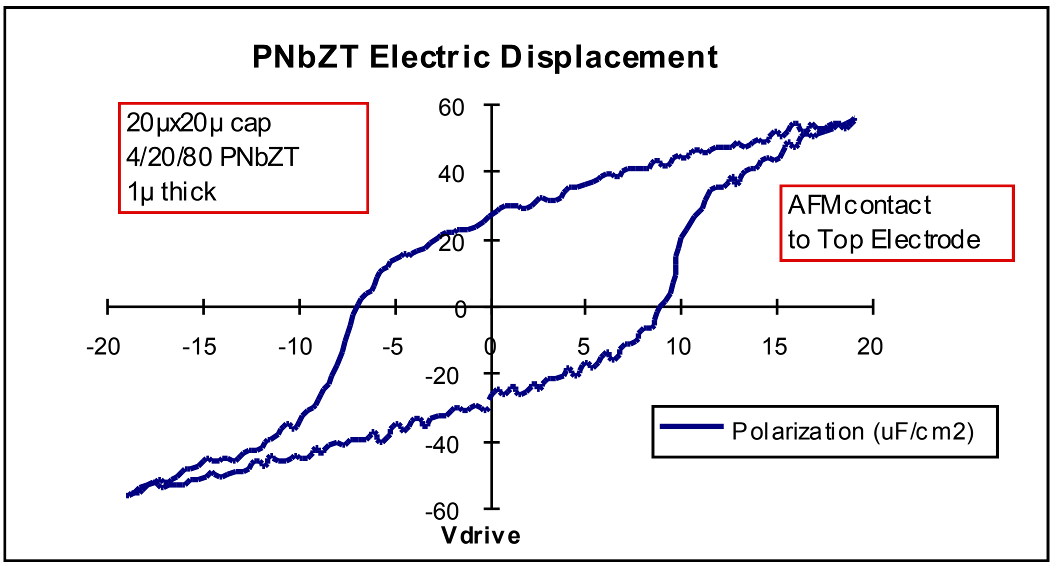

Figure 4
Polarization Hysteresis for 1µ 4/20/80 PNZT Film

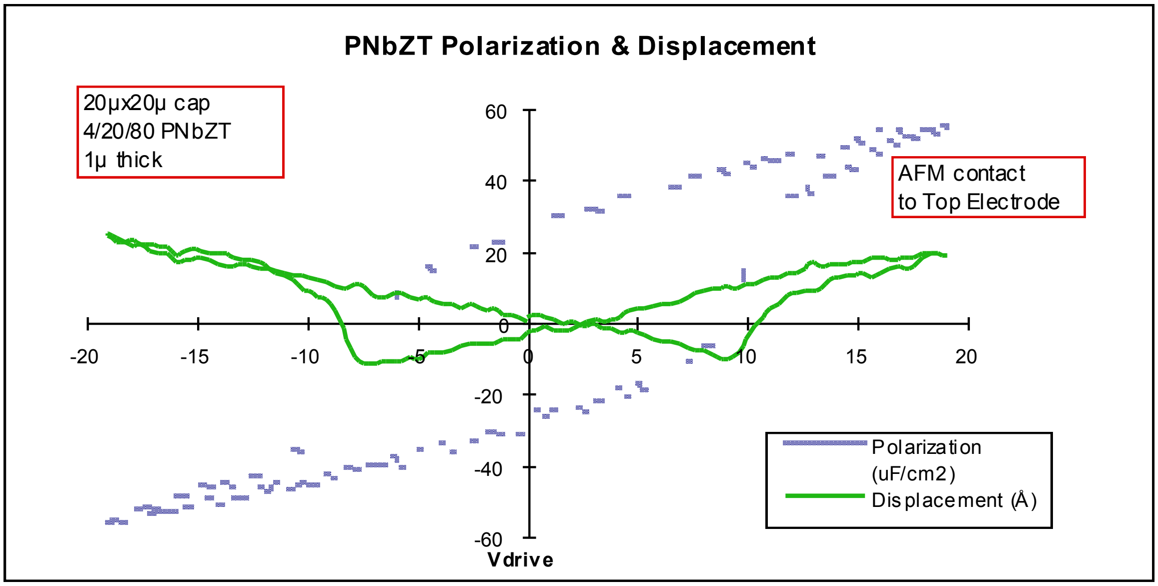

Figure 5
Displacement Hysteresis for 1µ 4/20/80 PNZT Film
(Shown with the polarization hysteresis.)

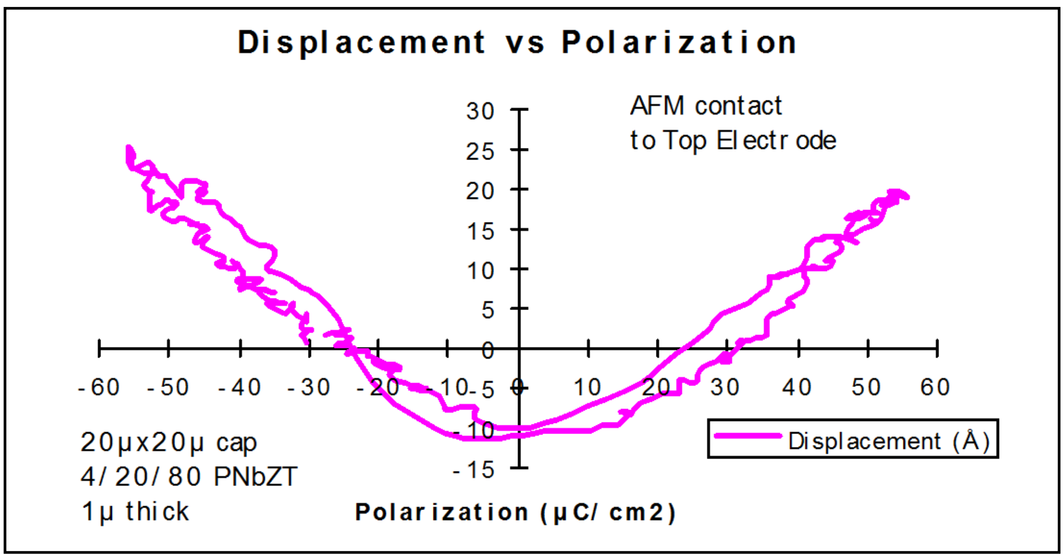

Figure 6
Displacement vs. Polarization for 1µ 4/20/80 PNZT Film

These are good and useful results!

Sources of Measurement Error
We identified several sources of errors that can be induced in the measurement of the piezoelectric displacement of the samples.

Feedback Amplification
The **Feedback** or **In0** signal in Figure 1 is actually the error signal to the AFM that the cantilever is out of position. The feedback control system then applies the appropriate voltage to the piezoelectric actuator shown in Figure 1 to move the cantilever back at its original position relative the sample surface. So, both the **Feedback** signal and the **Hi Z** signal are related in amplitude to how far the sample ferroelectric capacitor pushes the tip away from its rest position. The **Integral and Proportional Gain** values set in the AFM software control window by the AFM operator determines the speed with which the feedback control system of the AFM corrects the cantilever displacement with the piezoelectric actuator. If these gain values are reduced to zero, then no change to **Hi Z** occurs for any change in the **Feedback** signal. The system does not attempt to return the cantilever to its rest position. If, however, the **Integral and Proportional Gain** values are set high (≥2), then the **Hi Z** signal will move the cantilever up or down very quickly to take out the vertical deflection of the needle by the sample. This is important to understand when selecting either **In O** or **Hi Z** for capturing the AFM output. If the AFM response is fast relative to the RT6000 hysteresis speed, then it will correct the cantilever position faster than the hysteresis is executed and no error signal will show up at **In O**! So, when using **In O** for the MCh input, the gain values must be turned down to 0.001 or less by the AFM operator. On the other hand, with low gain values, no change will occur in the **Hi Z** signal. If **Hi Z** is used for the MCh acquisition, then the gain values must be set to 2 or greater.

There is another consideration. The **In 0** signal has a small input range and so with a small change in the height of the tip or sample this signal can drift out of range. In normal operation, any change in the **In 0** signal will be compensated for by changing the **Hi Z** signal to keep the value at zero. If the **Integral and Proportional gain** values are set low to use the **In 0** signal for the MCh input, than the **In 0** signal can quickly drift out of range. With the gains turned up high, the **Hi Z** signal will need to be used for the hysteresis measurement. Thus, whenever possible, the **Hi Z** channel should be used for the MCh connection instead of **In 0**.

Surface Roughness
The surface roughness of the top electrode on the ferroelectric sample averaged about 150Å. Since the piezoelectric displacement was on the order of 30Å, the surface roughness was 5 times higher than our signal to be measured. This is a problem because the AFM cannot hold the sample chuck perfectly still in a horizontal direction. Vibration noise will cause a constant, periodic motion of the tip horizontally across the sample. If the cantilever tip is resting on the sidewall of a grain, then the vertical slope of the sidewall will cause periodic vertical fluctuations of the cantilever tip and show up in the measurements. The rougher the sample surface, the greater the amplitude of this periodic noise to the point that it is greater than the electrically activated displacement to be measured. See Figure 7 below for a visual description of this effect. Also due primarily to thermal effects the tip of the cantilever will translate slowly across the surface of the sample. This is caused by uneven heating of the components holding the tip relative to the sample, with large changes in temperature (more than a couple degrees) the tip can translate several nanometers in the time it takes to make a hysteresis measurements. If the tip is positioned on a part of the sample that has a high slope such as a sidewall of one of the grains in the film, this drift will cause a change in the measured displacement of the sample.

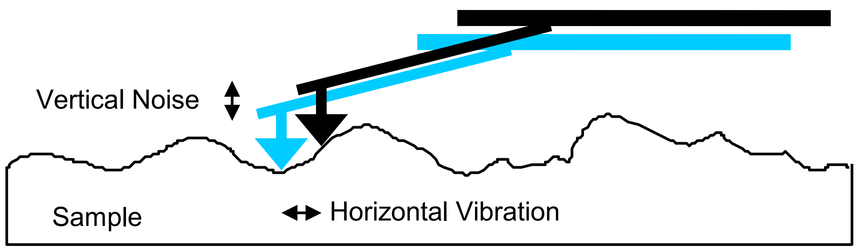

Figure 7
Surface Roughness Measurement Noise Source due to Vibration

There are six actions that the researcher can take to mitigate this effect though it cannot be eliminated totally.

1. Make the sample surface as smooth as possible.

2. Reduce the measurement system vibration as much as possible.

3. Keep the **Feedback Amplification** as high as possible.

4. Use the **Standard Laser Diode** supplied with the AFM. Some optional laser diodes available from Digital Instruments for specialized applications can have more low frequency noise which makes this measurement more difficult.

5. Use a cantilever with a very large tip on it. Normally, AFM tips are made with diameters smaller than 20nm. Digital Instruments can provide information on how to glue a large diameter metal sphere onto the tip or provide you with information on companies that offer this service. This special cantilever would further reduce surface roughness noise by having a large contact area to average the surface roughness.

6. Place the tip on the flattest portion of the sample. If no flat portion is available, a trough is better than a hilltop.

Figure 8 shows a measurement made with an optional **High Speed Laser Diode** and the **Standard Laser Diode** of a 100µx100µ capacitor. The reduction of the surface roughness noise level is apparent.

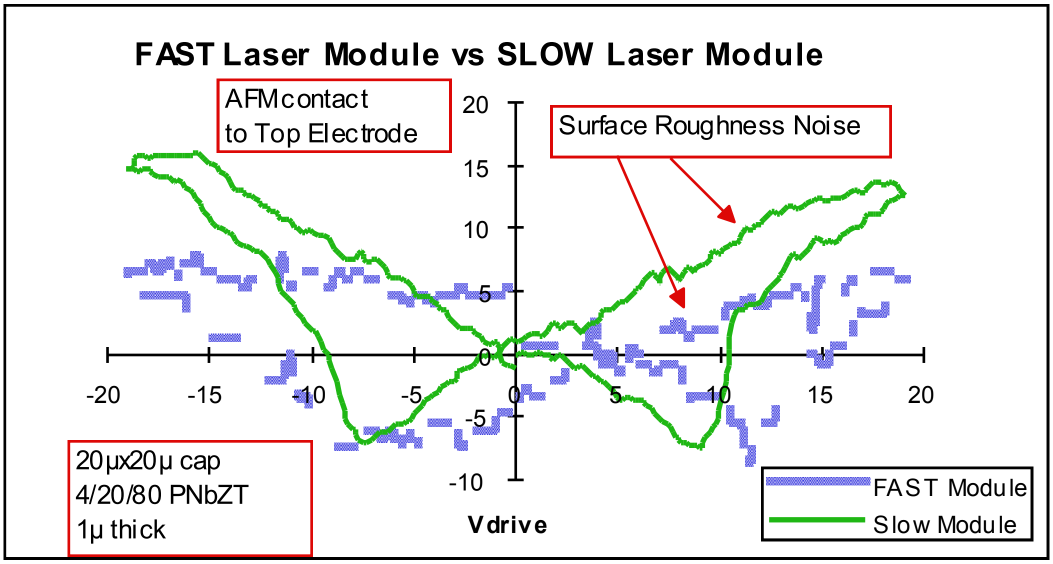

Figure 8
Surface Roughness Noise as a Function of the Microscope Head Model

Positioning Motor Backlash
All motors have backlash. Backlash occurs when a motor rotates backwards a small amount when stopping after being intentionally moved forward. The motor armature has to "settle" into a new stable position. Thus, whenever the tip or chuck is moved by the operator, the actual position of the tip horizontally and vertically on the sample will drift for about a minute or two. If a measurement is taken while the sample is drifting, then the slope of the surface at that point will add an error to the piezoelectric actuation of the sample. The problem is mitigated somewhat by using the large spherical tips described in the previous section. However, it is best to wait a few minutes for the drift to stop. The drift is most noticeable when moving the vertical motor on the AFM to keep the **Hi Z signal** within the ± 20 volt input range of the MCh1 channel.

Voltage Protection for the Multichannel
The **Hi Z** analog output of the **Signal Access Module** can put out up to ±220V. This voltage will damage the multichannel amplifier input. A clipping circuit must be placed between the multichannel input and the **Hi Z** output to prevent the voltage input to the multichannel from exceeding ±20V. However, the 1KΩ resistor shown in Figure 1 is only 1/4 watts so a clipping circuit will blow out the resistor due to the current flow through it if the assigned voltage is 220V and the clipping circuit holds the other side of the resistor to 20V. A resistor between **Hi Z** and the clipping circuit will prevent this problem. The recommended resistor value is 100KΩ, which will lead to about a 1KHz filter effect on the measured values when coupled to the input capacitance of the multichannel. The protection circuit is shown in Figure 9.

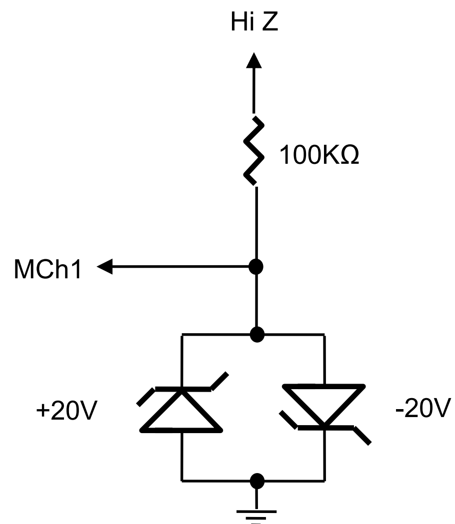

Figure 9
Recommended Protection Circuit for Use with Hi Z.

Radiant will build and supply this circuit to customers at their request.

Recommended Operation for the Digital Instruments Dimension 3000
In consideration of the factors described above, the following is the configuration required to make PIEZO measurements using a Digital Instruments AFM.

1. The sample must be prepared so that the bottom electrode can be connected to the RT6000 by some method. The sample should be as smooth as possible.

2. The AFM must have the **Signal Access Module** attached.

3. The AFM jumpers must be set according to Figure 2 above.

4. The microscope should be mounted in a Digital Instruments VT103 isolation chamber or some other chamber that provides very good acoustic and mechanical noise rejection.

5. Ideally, a spherical tip cantilever should be used. Barring that, a standard 225µ metallized cantilever, model **MESP**, will work but with higher surface roughness noise and drift noise.

6. The AFM operator must conduct a noise analysis test on the AFM and reduce the ambient noise to 0.5Å rms. The noise level can also be checked by looking at **In 0** or **Hi Z** with an oscilloscope. The normal amplification range is ~1000Å per volt on **In 0**

and ~100Å per volt on **Hi Z**. The noise should be less than 20mV on **Hi Z** when the system is properly quiet.

8. The operator should calibrate the AFM and set it to contact mode.

9. The RT6000 earth ground should be thoroughly grounded to the VT103 isolation chamber and the AFM inside.

10. A protection module should be attached between the Hi Z and the multichannel to be used.

11. The following connections should be made:

    A. DRIVE => **ANA 2** (Signal Access Module)
    B. RETURN => Sample
    C. MCh1 => **Hi Z** (Signal Access Module)

12. Set the AFM **Feedback Amplification** gain to 2.00.

13. Look up the AFM vertical calibration and insert it into the Multichannel setup window for the gain factor. (In our experiment, it turned out to be 137Å/volt for **Hi Z.**)

14. The operator should lower the cantilever tip to the top electrode surface and adjust the surface force to cause the **Z-Center Position** to be 0V.

15. Execute the F5 Setup menu on PIEZO normally.

16. Take data! But wait for 2 minutes after moving either the tip or the chuck before making the measurements.

Usable Range
From the results, the film thickness from which measurable displacements may be acquired on an AFM are from 2000Å to 20µ.

Information
For more information or assistance, please contact Radiant Technologies, Inc.

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