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Large Signal Displacement Measurement with an Asylum SA Atomic Force Microscope

Rev B

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Introduction:

At the invitation of Dr. Roger Proksch, I visited Asylum Research in Santa Barbara, CA on Tuesday, October 21 and Wednesday, October 22, 2008. Asylum has an experimental lab with an SA Atomic Force Microscope for use in developing hardware and software for its products. I connected a Precision Premier II to the AFM and captured the butterfly loop of a 1µ thick PNZT capacitor at 1Hz. The Asylum instruments may be found at www.asylumresearch.com.

Test Setup:

I brought to Asylum a 100V Precision Premier II for acquiring the measurements. Using its built-in USB communications port, I ran the tester from my laptop. Roger programmed the SA to route the output of the quad-cell detector for the AFM to an external BNC connector on the AFM. I connected that signal to the SENSOR input of the Premier II. Roger calibrated the output of the AFM Z displacement to a sensitivity of 70nm/V or 700Å/V. The Premier II SENSOR input has a resolution of 76µV/bit and a single pass noise floor of 1mV. With a 1mV noise floor on a single pass, the Premier II with the Asylum SA AFM sensitivity has a useful displacement resolution of approximately 0.7Å. The effect of the noise floor is reduced by a factor of four or more using averaging. (See the document "Displacement Sensor Operations" from Radiant Technologies, Inc. for more information.)

The resolution limit calculated in the previous paragraph is that of the Premier II alone. The sensor, in this case an AFM, adds its own noise to the measurement. This particular AFM generated about 15Å to 20Å of noise peak-to-peak, a value much larger than expected for an Asylum AFM. No doubt, the use of this instrument as a test bed by Asylum contributed to its out-of-specification noise level. Radiant has a Premier II at its office abused in a similar manner. Roger attached a Stanford Research Systems programmable filter on the output of the AFM set for 1KHz (www.thinksrs.com). This modification significantly reduced the noise level of the AFM signal to 3Å peak-to-peak. Averaging x10 of the 1Hz measurement reduced the noise level to approximately 1Å peak-to-peak.

The Precision Premier II and Precision LC generate a 5V SYNC signal when they are capturing data. (Again, see the document "Displacement Sensor Operations" from Radiant Technologies, Inc. for more information.) The SYNC signal is accessible at a BNC on the rear face of the testers. With some sensors, the SYNC signal can be used to coordinate the operations of the tester and the sensor. While the SA AFM does allow controlling digital inputs, it was not feasible to use the SYNC signal to control the Z integration function on the SA for our measurements this time. We thus had two options:

1) Put a PAUSE task in the program at every point where the AFM must be manually manipulated prior to a measurement loop being executed or

2) Free the AFM prior to starting the test program on the Premier II with the knowledge that the AFM output will remain stable for the duration of the program execution.

The integrator on the SA AFM was stable long enough for us to take 10 sequential loops under program control on the Premier II so we chose option 2.

Sample Description:
The data in this report were acquired from a Radiant Type AC capacitor. The Type AC die has 1µ of 4/20/80 PNZT on a 1500Å platinum bottom electrode. The top platinum electrode is 1000Å thick. It is covered by 800Å of PNZT followed by 400Å of TiOx and 2300Å of silicon dioxide. The metal connections are in chrome/gold. The dimensions of the targeted capacitor were 80µ x 125µ. Both of these dimensions are less than the 550µ thickness of the silicon substrate so there should be little bending moment of the substrate to contribute to an amplified piezoelectric piston movement.

> NOTE: 1µ thick 4/20/80 PNZT will bend a 4” diameter, 550µ thick <100> silicon wafer 1000Å at a distance of 5 millimeters from the center of the capacitor. Bending moments are important to consider when measuring converse piezoelectric constants.

This particular die had a well etched under the left-hand side of the die, creating a membrane capacitor. We measured the displacement of the $10{,}000\mu^2$ capacitor on the right-hand side of the die as marked by the red “dots” in Figures 1 and 2 below. The measured capacitor was on solid silicon.

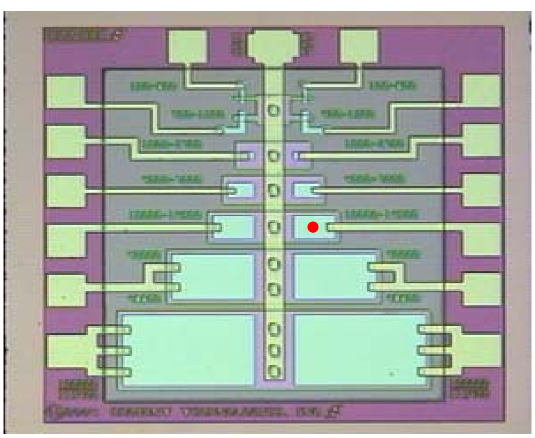


Figure 1
Type AC Die without Well

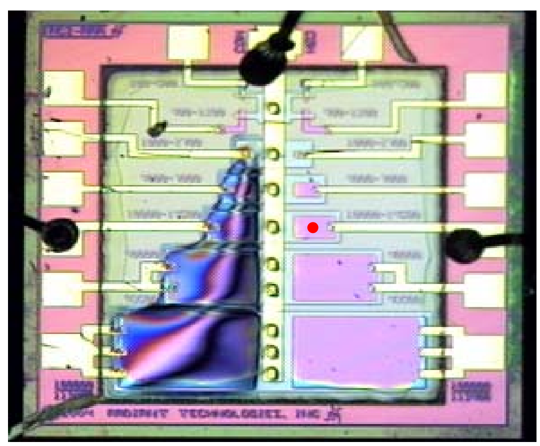
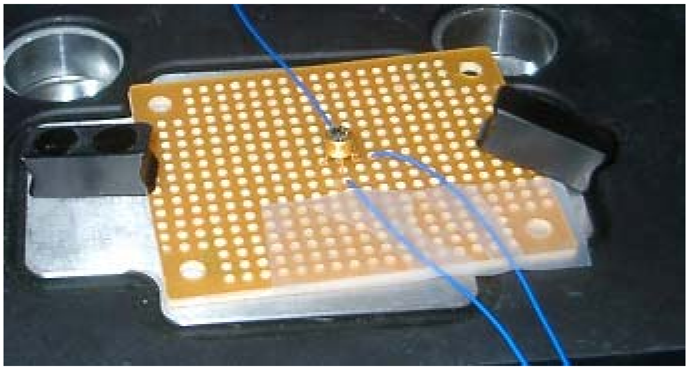


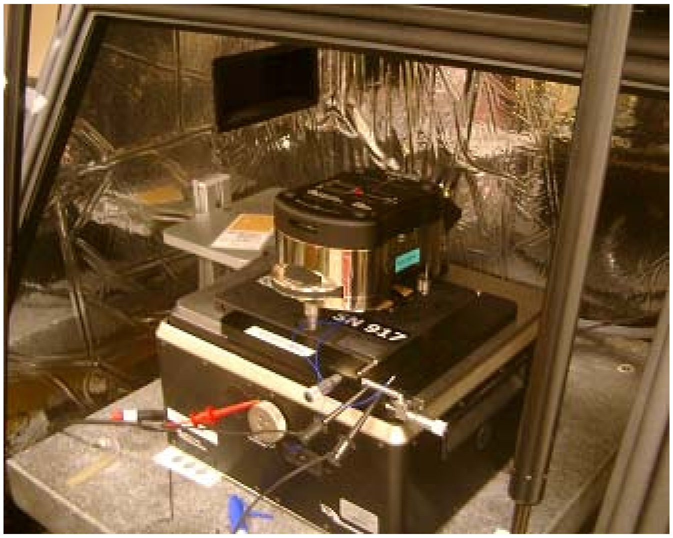
Figure 2
The Tested Die in Package

For the Asylum visit, Carl Montross at Radiant prepared the Radiant Type AC die on a TO-18 header without a lid. At Asylum, Roger Proksch mounted the package on a blank electronic perf board and soldered small-gauge wires to the package leads. He placed the board on the test frame of the AFM and placed the AFM probe head over the sample. I connected the capacitor leads to the Premier II using mini-grabbers at the end of coax cables. Photographs of the AFM with the sample are shown below.

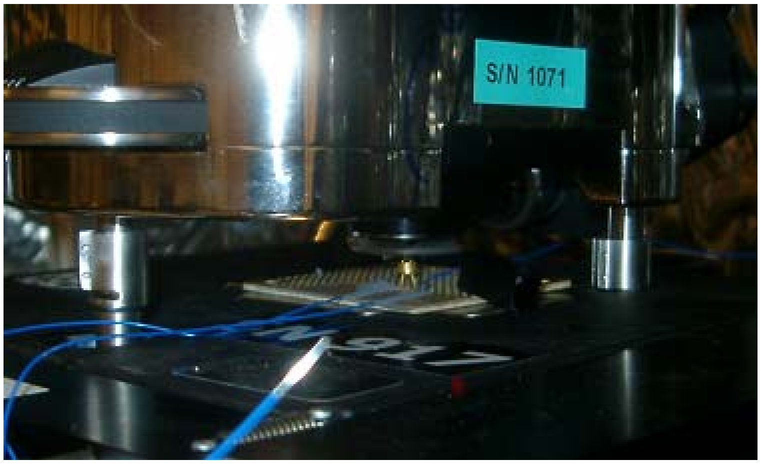
Perf Board and Sample on the AFM Base
Figure 3



Close-up of Sample on the Perf Board with Wires
Figure 4



The AFM Probe Head over the Sample
Figure 5



The Sample under the AFM Probe Head
Figure 6

Once the sample was in place and the AFM test fixture assembled, Roger used a built-in camera and motorized X/Y stage to position the AFM probe tip in the center of the capacitor to be tested.

Parasitics Affecting Atomic Force Microscopes:
Atomic Force Microscopes can measure extremely small displacements. Nevertheless, they are subject to several unique parasitic effects which can distort the results.

60Hz/50Hz/Control System Electrical Noise:
Atomic force microscopes are physically large systems with high-voltage control circuits. When connecting a Precision tester to a sample mounted in an AFM, it is highly likely that the RETURN lead will pick up 60Hz or 50Hz from the AFM power supply as well as EMF from the high voltage signal that controls the Z-position of the AFM stage. Coax cables should be used to connect the DRIVE and RETURN BNCs of the tester to the sample. The sample should be isolated electrically from the AFM chuck and the AFM chuck should be grounded. Finally, and most important, the shields of the coax cables from the tester must be connected to the frame ground of the AFM. This connection makes the EMF noise common mode for both machines, allowing the tester measurement circuits to subtract it out automatically. If all else fails, connect both the tester and the AFM to the same wall socket!

Mechanical Amplification
The purpose of this experiment was to measure the piston displacement of the surface of the thin ferroelectric film capacitor. The total displacement of the capacitor surface should be in the range of 20Å. One micron of PZT is capable of bending a 550µ thick silicon wafer, overwhelming the piston displacement of the capacitor. To avoid this effect, it is best to measure a capacitor with dimensions much smaller than the thickness of the substrate thereby reducing the length of the "lever arm" which the capacitor can bend. Additionally, placing the measurement point as close as possible to the geometric center of the capacitor top electrode where the capacitor "cups" further reduces amplification effects. You can have the AFM scan the capacitor surface to find the geometric center for you.

In this experiment, I measured the center of a capacitor with dimensions of 80µ by 125µ on a 550µ thick silicon substrate. The capacitor was large enough in dimensions that it could have shown slight amplification at the outer edges of the capacitor top electrode. An additional factor here was that the die was epoxied to the package header, further reducing its ability to move when the capacitor was actuated. Such clamping of the sample substrate helps reduce the lever arm of the mechanical amplification but it does not totally eliminate the possibility of wrinkling by the substrate while under restraint.

Were my purpose to determine the converse piezoelectric constant for the ferroelectric material, I would measure a much smaller capacitor, possibly 10µ x 10µ or even 5µ x 5µ. Since the ferroelectric film in this case is 1µ thick, measuring the displacement of a capacitor with the dimensions of 1µ on a side could lead to other errors because fringe electric fields between the electrodes could contribute to the sample response.

Starting Point Alignment
All test fixtures, not just AFMs, can have drift in the Z-position of the frame holding the test fixture. Consequently, the first point of each sequential measurement might end up at a different location in Z space, causing inaccurate reproduction of the measured signal when averaging multiple measurements. A solution to this problem is to mathematically zero the first point of each sequential measurement before averaging. A measurement is zero'd by subtracting the value of the first point from all points in that measurement, causing a translation along the Y-axis.

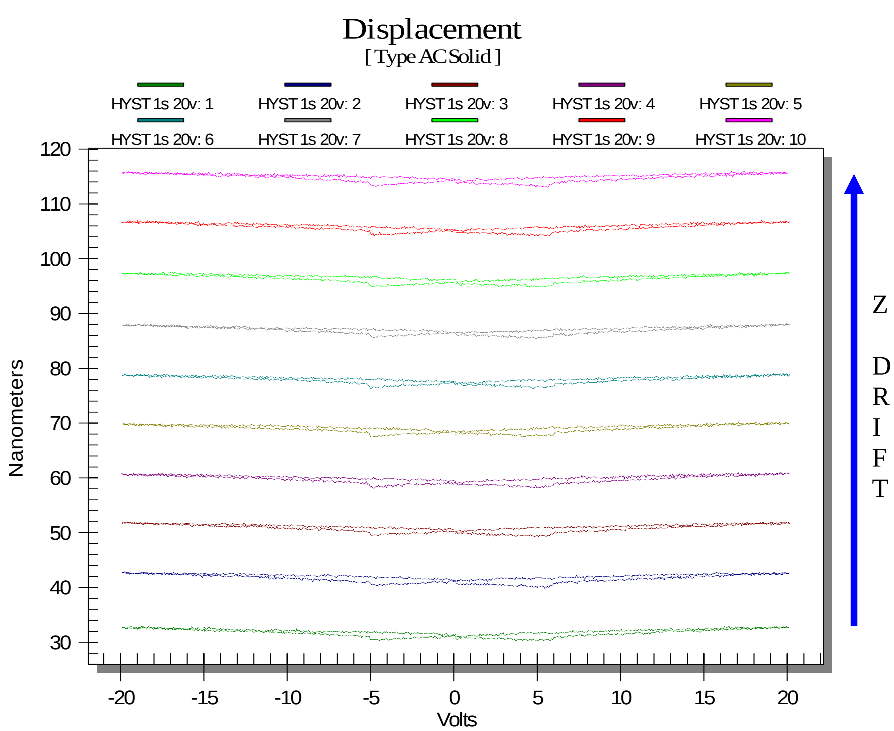
Maximum Sensitivity
Normally, AFMs acquire the location of the AFM probe tip in space and then *move the chuck* piezoelectrically to maintain the prove tip in a fixed position in space. The frequency response of this control circuit is usually in the hundreds of Hertz so measurements of the butterfly loop taken with the control signal active must be taken at 1Hz or slower. When in this mode, the *error signal* from the Z

control system is the desired source of information for the tester SENSOR input and it may need to be integrated using the Single-Trace Math filter in the Vision Library.

For maximum sensitivity and frequency response, disable the control system and feed the output of the position sensors directly to the tester SENSOR input. The position information is very fast, in the hundreds of kilohertz range, and it is a direct measure of the position of the AFM probe tip in space. Theoretically, this “free” position signal is the best one to use for measuring the large signal butterfly loop. The problem with this approach is that without the control system actively controlling the position of the chuck, the AFM chuck will begin a relatively constant-rate drift in one direction or the other until it hits an internal stop. Please note that all AFMs have this issue, not just the Asylum instruments. This drift is the source of the Z-drift error described next so if you use this mode of operation, be prepared to characterize the rate of Z-drift and subtract it from your results.

Z-drift

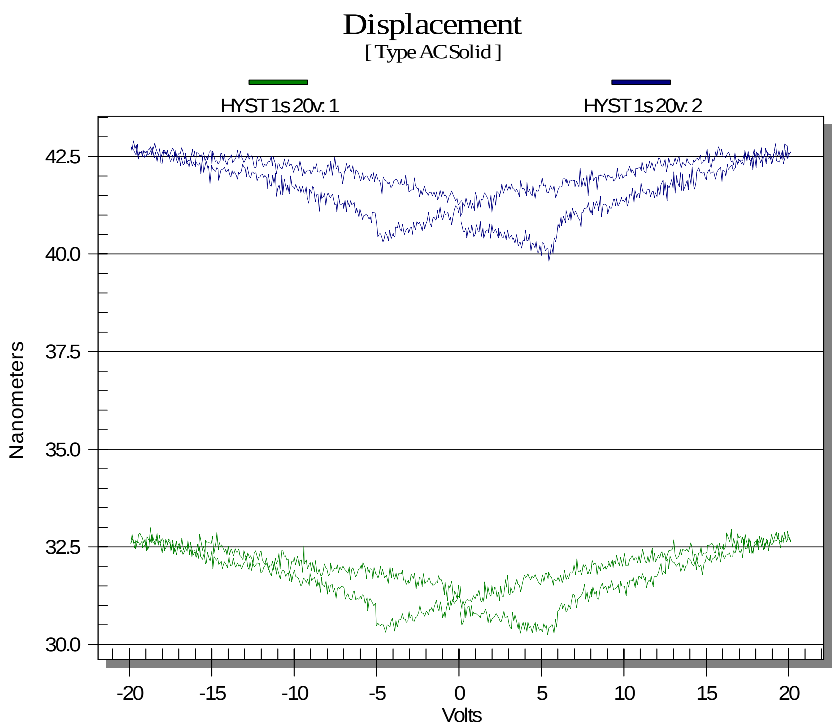
When using an AFM at maximum sensitivity in the free mode, the constant drift rate of the chuck will add to the displacement data. The amount of drift differs from machine to machine and from day to day. This effect will not only cause the starting point of each measurement to be different, as noted earlier, but will also cause a “slope” in the resulting measurement so the starting point and ending point of a butterfly loop do not line up. We captured a classic example of this effect on the SA AFM as shown in Figure 7 below. The data in Figure 7 is the same as that analyzed in the next section of this report. Figure 7 is a plot of the absolute measured values of 10 sequential 1-second butterfly loops on the SA AFM after the AFM control system was turned off.



Sequential Measurements of the Same Sample on the SA AFM

Figure 7

The drift rate was high enough that it introduced a slope to each butterfly. Two of the loops are shown in Figure 8 as raw data. The drift between the first and last points for each butterfly loop is readily apparent.



Two of the Loops from Figure 7 Showing Drift Effects
Figure 8

The distortion of the measured butterfly loops can be corrected by mathematically introducing a drift of equal magnitude in the opposite direction. To accomplish this requires careful characterization of the AFM over a time period equivalent to or longer than that required to capture all of the measurements to be averaged. Luckily, each measurement record in the Vision Archive contains a time stamp. I copied the time stamp for each measurement in Figure 7 into a spreadsheet along with the value of the first point of each loop in Figure 7. I found that the drift rate for this particular AFM was consistently 0.54 nanometers, or 5.4Å, on each test loop of 1 second. Each test consisted of 1017 points, a number also accessible from the Vision Archive, so the drift rate was 0.00053nm per measurement point. With this information, it was relatively easy in the spreadsheet to construct a sloped line to subtract from each measurement in order to eliminate the Z-drift distortion.

Correcting Systematic Distortions

At present, the PIEZO task in Vision will accept a Z-drift value from the operator and subtract that drift from the measurement before it is stored in the Archive. PIEZO will also plot individual measurements with the data translated so the first point is zero. The PIEZO SENSOR data cannot be readily submitted to a loop averaging task. Radiant is presently constructing two filters for the Vision Library that will allow the correction operations to be performed on PIEZO SENSOR data as well as on other measurement tasks before the data is sent to a loop averaging filter. The new tasks will also provide a smoothing filter.

Results:
The test consisted of 10 sequential 20V hysteresis loops in a row with 1s periods. The SRS programmable filter was connected between the AFM Z output and the SENSOR input. The filter cutoff frequency was set to 1kHz. Being 1000 times faster than the measurement period, the filter introduced negligible phase distortion to the final results. The absolute values of the ten measurements have already been shown above in Figure 7. I exported the ten loops to a spread sheet where I

1) moved each measurement vertically so that each starting point was “0”,

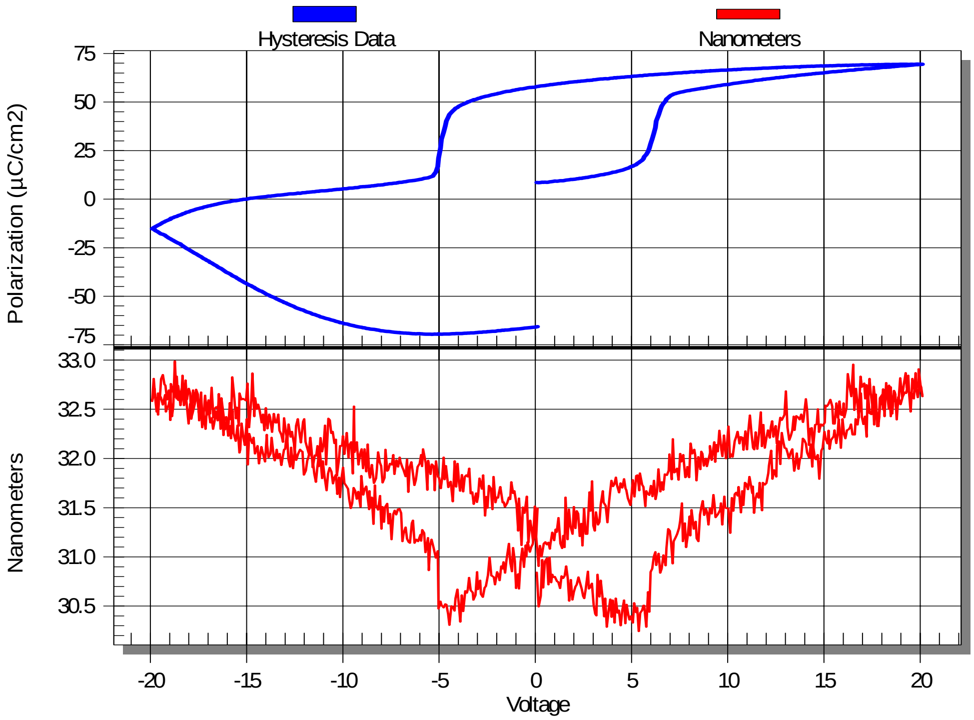
2) calculated the Z-drift rate and subtracted it from each loop,

3) averaged the ten loops, and

4) applied a smoothing filter to the averaged butterfly loop.

As described earlier in the “Test Setup” section of this report, we did not use the SYNC signal of the Premier II to coordinate the tester and the AFM operations. Instead, Roger disabled the control system and fed the output of the position sensors directly to the tester. Roger found that the chuck on the SA AFM would take several minutes to hit its stop after the control system was turned off, long enough for the Premier II to execute at least 10 measurement loops for averaging. Therefore, the procedure was for me to prepare to run the test program and, just prior to execution, for Roger to turn off the Z-position control system to release the chuck. After the program execution, Roger would turn on the control system in preparation for the next test.

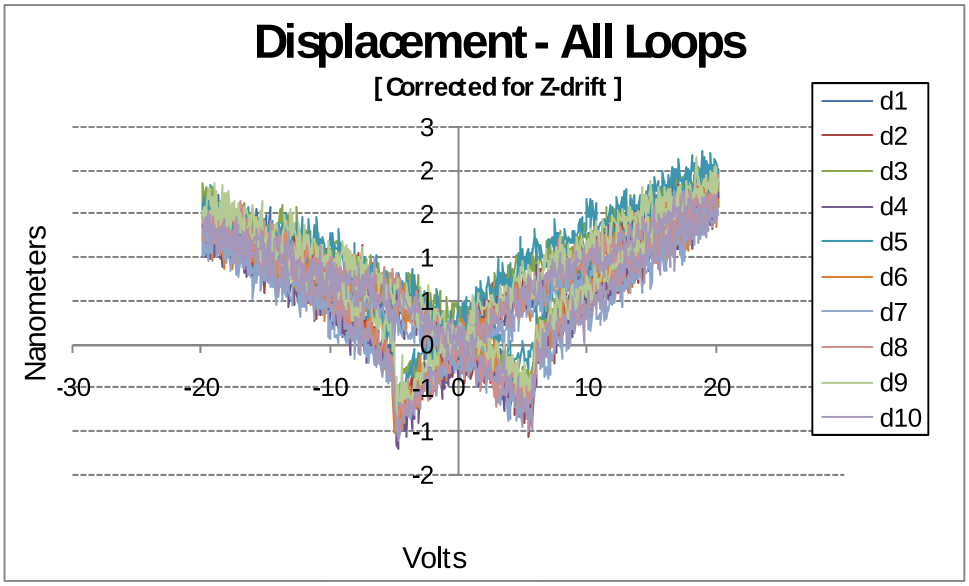
The raw polarization and displacement data for one of the ten measurements of this sample is shown below:



Raw Measurement of Polarization and Piston Displacement of Type AC Capacitor
Figure 9

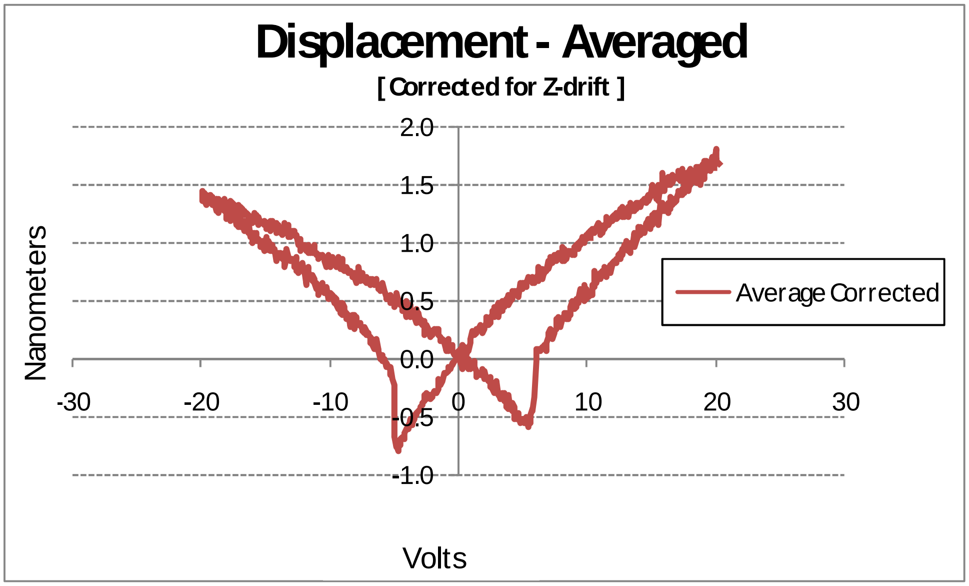
Note that the negative saturation point of the polarization loop shows leakage. The voltage at which the leakage started was three times the negative coercive voltage for the sample. This is not unexpected for a thin PZT capacitor when exposed to strong electric fields at the high illumination levels typical in an AFM. Also note that the leakage of the capacitor did not appear to affect the shape of the butterfly loop. Finally, you can see the discontinuity between the butterfly loop start/stop points due to the Z-drift.

The plot below shows the ten loops after zeroing and correction for Z-drift.



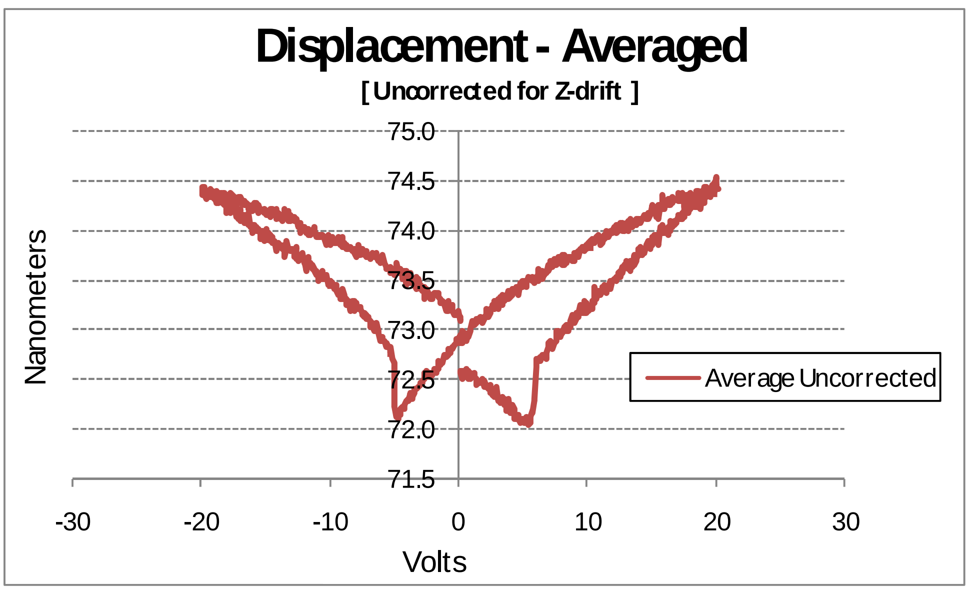
All Loops Zero'd and Corrected for Z-drift
Figure 10

Figure 11 shows the average of the loops in Figure 10.



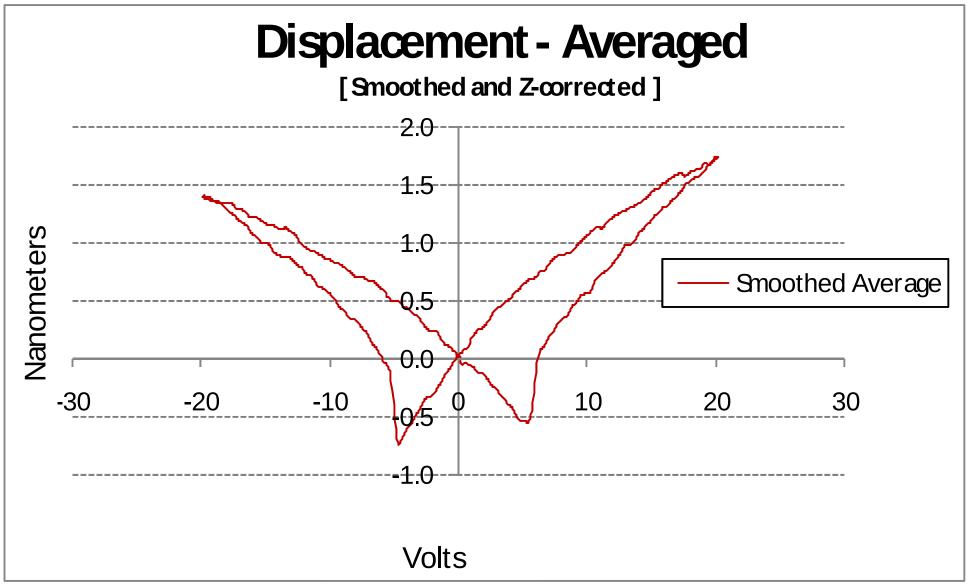
Averaged Butterfly Loop Corrected for Distortions
Figure 11

For comparison, I plot below the zero'd and averaged butterfly loop without the Z-drift correction.



Butterfly Loop Uncorrected for Z-drift
Figure 12

Finally, Figure 13, the lucky figure, shows the butterfly loop of Figure 11 after the application of a triangular 9-point smoothing filter.



The Butterfly Loop
Figure 13

Nice!

Conclusion

The piston-only butterfly displacement loop of a thin ferroelectric film capacitor can be measured accurately with an atomic force microscope. Given the resolution of AFMs, it is possible to capture the displacement of the surface of the capacitor to a resolution less than 1Å. The resolution can be further improved to almost 0.1Å with a smoothing filter. The parasitic effect of mechanical amplification may be significantly reduced by measuring capacitors with very small areas. Radiant is preparing new filter tasks for the Vision Library to be used in displacement measurements to correct acquired data for parasitic distortions from attached displacement sensors.