

Keysight Technologies
PFM Experiments with
High Voltage DC/AC Bias

Support Note



Introduction

In this technical note, we will briefly describe a number of different configurations for doing PFM measurement using the Keysight Technologies Inc. 5420/5500/5600 AFM systems, particularly about the configuration that allows the user to perform PFM experiments with high AC/DC bias with the help of an external high voltage amplifier.

Shijie Wu and John Alexander
Keysight Technologies

Piezoelectric force microscopy (PFM) has found major applications in the study of ferroelectric materials, particularly for high resolution imaging, domain switching, local hysteresis measurements, and switching of ferroelectric capacitors. For example, ferroelectric materials are used in many field effect transistor applications due to their high permittivity. With the demand of miniaturization of electronic devices, it becomes important to study the size effect of ferroelectric materials, i.e., to study the critical size range where significant deviation from bulk properties occurs. The high spatial resolution of PFM also provides a unique opportunity to study the fundamental process of domain switching, including the thermodynamics and kinetics of domain nucleation, growth, and relaxation. By analyzing local hysteresis loop measured by PFM, information about the piezoelectric properties of small ferroelectric domains can be obtained, including coercive voltages, nucleation voltages, forward and reverse saturation and remanent responses, as well as the effective work of switching defined by the area included in the hysteresis loop. Simultaneously recorded VPFM and LPFM nanoscale hysteresis loops were used to differentiate 900 domain switching from 1800 domain switching, and to study the dependence of PFM on crystallographic orientations.

Even though a wide range of materials can be studied by PFM with a relatively low AC/DC bias to the tip or sample, within +/-10 V typically, there is a demand for being able to apply higher bias, up to several hundred volts, in many cases. One reason for such a request is the need of high voltages for domain switching in some materials. In order to study the piezoelectric behavior of those materials, high enough voltages have to be applied to switch the polarization and to observe the hysteresis of the relaxation process. A second reason for the need of high voltage bias is to increase the signal-to-noise ratio of PFM experiments on materials of lower piezoelectric response. Since the PFM signal measured is proportional to the bias voltage, it will increase with the AC voltage of the applied modulation bias. Therefore, one can apply higher bias to study the piezoelectric properties of materials with lower piezoelectric coefficient, provided the material under test can sustain the voltage applied without damage and pertinent property change.

In this technical note, we will briefly describe a number of different configurations for doing PFM measurement using the Keysight Technologies Inc. 5420/5500/5600 AFM systems, particularly about the configuration that allows the user to perform PFM experiments with high AC/DC bias with the help of an external high voltage amplifier. The setup discussed in section A involves only the standard hardware including a MAC III controller. This configuration is the most direct approach, every system equipped with a MAC III controller can do PFM experiments with this setup. The discussion in section A also helps user to understand the various sources of input/output signals in the system, thus better understand the operational principle of PFM, However, this configuration is not the best configuration for doing PFM due to the possible coupling between signals. The approach in section B separates the modulation bias from the PFM signal by the use of a MAC III Signal Access Box, thus minimizes the coupling between the AC bias and AFM signals, and should be used as the recommended approach for low voltage PFM experiments. The configuration in section C shows a simple approach for PFM experiments with high voltage bias, using a commercially available stand-alone high voltage amplifier.

A. PFM setup for 5420/5500/5600 AFM system with default hardware

The default configurations for PFM experiments using the 5420/5500/5600 system are essentially all the same. The typical setup for PFM experiment is illustrated in a block diagram (excluding the shaded part) in Figure 1, involving the use of MAC III controller as both the function generator for AC modulation bias and the lock-in amplifier for PFM signal analysis. The MAC III has three dual phase lock-in amplifiers (LIA) converting the AC inputs to amplitude and phase. These digitally-controlled analog LIA have a broad bandwidth (up to 6 MHz) that covers the operation bandwidth of the photodetector employed in the microscope. The electric connection for a simple PFM experiment is similar to that of a current sensing AFM experiment, except that a contact/AAC mode nosecone is used instead of a CSAFM nosecone. The conductive cantilever is connected to tip bias through the scanner and spring clip on the nosecone, and the bias switch at the back panel of the Head-Electronics Box (HE) should be set to "Tip". The sample is connected to the sample bias

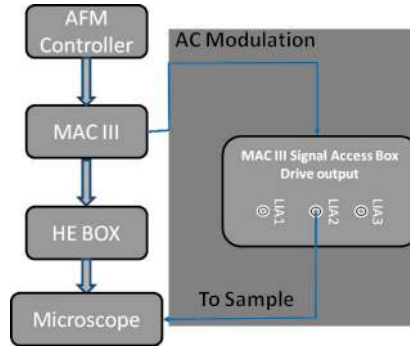


Figure 1. Block diagram showing the PFM setup with default hardware and with a MAC III Signal Access Box (shaded part of the diagram) for Keysight 5420/5500/5600 AFM system.

through the middle pin of the 3-wire EC cable that is connected to the base of the microscope. During an experiment, the DC bias and the AC modulation can be applied to either the sample or the conductive tip from the software control in PicoView. As shown in Figure 2, the DC bias is applied to the conductive cantilever when the "Apply Bias To" control is set to "Tip" from the Advanced tab of the "Servo" control window, and the sample is set to ground at the same time. Similarly, the DC bias can be applied to the sample by setting the "Apply Bias To" control to "Sample", and the tip will be set to ground in this case.

The AC bias is generated from the AC drive signal from the lock-in amplifier in MAC III; usually the AC drive from LIA

2 is used. The AC modulation can be applied to the sample or the conductive cantilever by choosing the proper settings in the "Advance AAC Mode Controls" window. Figure 3 shows an example setting for applying AC bias from LIA 2 to the sample. In the left side window of Figure 3, the AC bias is defined using the drive output of LIA 2, which in this case was set to a frequency of 10.123 kHz and a peak amplitude of 1V. Other adjustable parameters in the window include: the "Gain" to scale up the input signal if the piezoelectric effect is weak and the "Bandwidth" to reduce the signal noise by applying a band-pass filter. The "Input" signal can be set either to "Deflection" for VPFM or to "Friction" for LPFM. The "Phase Shift" shifts the relative phase angle of the AC drive signal against the internal reference signal of the lock-in amplifier. The "Phase Offset" adds a constant offset to the measured phase angle for proper display.

In the right side window ("Outputs") of Figure 3, the "Sample Bias" is set to "Drive 2", which directs AC bias signal from the drive output of LIA 2 to the sample. The "Sum" box beside the "Sample Bias" is checked in this case, thus the bias signal from the MAC III will be added to the bias from the sample bias DAC (if any) and delivered to the sample together. If this "Sum" box is

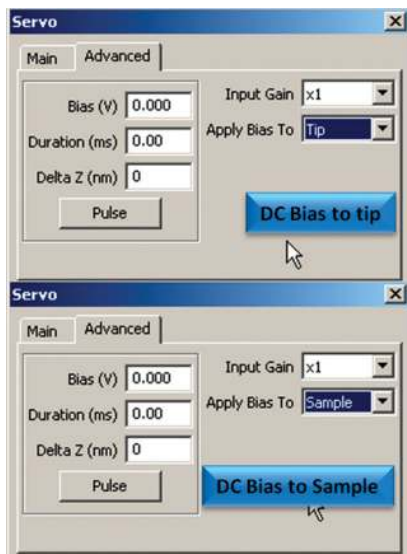


Figure 2. Software control window for setting DC bias to sample or to Cantilever.

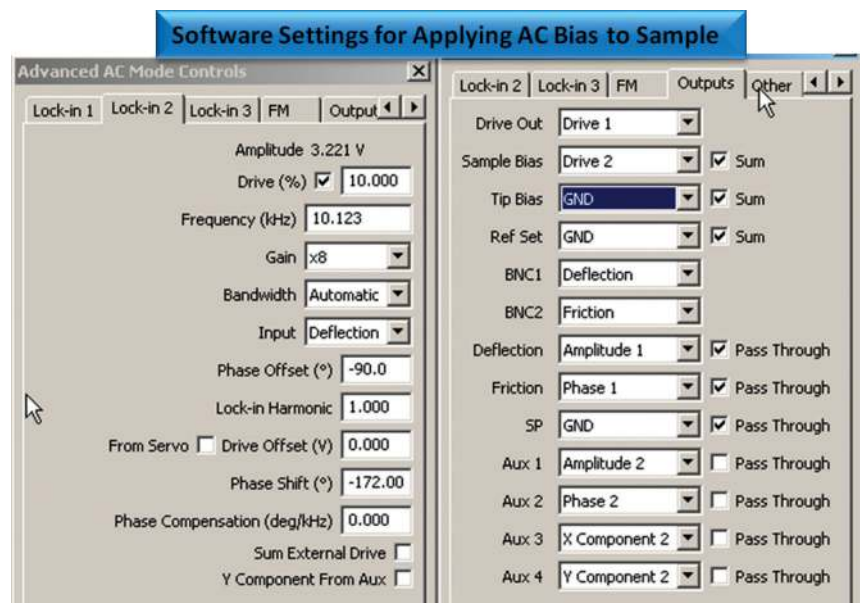


Figure 3. Software control windows for setting the AC bias to sample and the PFM signal output

unchecked, then the bias from the sample bias DAC is cut open, and only the bias from the MAC III will be sent to the sample. If the "Tip Bias" is set to "Drive 2" and the "Sample Bias" is set to ground ("GND") in this "Outputs" window, then the same bias signal from MAC III will be applied to the conductive cantilever. Again, this AC bias signal can be added together with the bias from the tip bias DAC if the corresponding "Sum" box is checked.

It is important to notice that the bias from the controller DACs, which is set in the "Servo" control window, and the bias from the MAC III controller, which is set in the "Advanced AC Mode Control" window, are from different sources and work independently. They can be applied to sample or tip together as one summed signal; or they can be applied separately, with one to the sample and another to the tip, and vice versa.

The outputs from the lock-in amplifier used to analyze the PFM signals are given in the form of Amplitude, Phase, X Component, and Y Component. These signals can be channeled internally to one of the four auxiliary channels Aux 1, Aux 2, Aux 3, and Aux 4 in MAC III, and then accessed by PicoView for display and processing. As shown in the "Outputs" window in Figure 3, the Aux 1, 2, 3, 4 channels are set to Amplitude, Phase, X, and Y Components from LIA2, respectively. Because the input signal to LIA 2 is deflection, thus all those four signals correspond to the piezoelectric response normal to the sample surface, i.e., VPFM. If LIA 3 is set to the same frequency as Drive 2 with its input set to friction, then the PFM signal given by LIA 3 will correspond to the piezo effect parallel to the sample surface, i.e., LPFM. If Aux1 and 2 are assigned to VPFM signals (signals from LIA 2 in this case) and Aux 3 and 4 are assigned to LPFM signals (signals from LIA 3 here), then VPFM and LPFM can be measured simultaneously for the sample under test.

B. PFM setup with MAC III Signal Access Box

In PFM experiments because of the huge disparity in the signal size between the drive signal and the tiny deviations in the deflection signal, maintaining separation is one of the serious requirements for successful measurements. With the default setup described above, however, there is a possibility that the applied AC modulation bias will interfere with other electric signals in the system, particularly those in the same frequency range. In order to avoid coupling between the PFM bias and other AFM signals, and to minimize the noise level in the AC bias and the PFM signal detected, it is necessary to isolate the pathway of the AC bias from the rest of the signals.

As described in Reference 2, the isolation of the PFM bias from the rest of the signals can be done with the help of a MAC III Signal Access Box. The drive outputs from MAC III are accessible through the MAC III Signal Access Box, which is shown in the shaded block of the diagram in Figure 1. The "clean" bias voltage is supplied to the sample with a separate shielded cable connecting the Drive Output of LIA 2 from the Signal Access Box and to the sample stage. The connections between the components are supported by the software that controls them using Advanced AC Controls window and allows monitoring of the signals via the Aux Channels I/O window. Since the drive output from the MAC III Signal Access Box bypasses the lines and control switches inside the MAC III controller and connects to the sample stage directly, the settings for "Sample Bias" in the "Outputs" window shown in Figure 3 are no longer relevant. It can be set to "GND" just for safe practice. With the AC bias from the MAC III

directly applied to the sample, one still has the choice to apply a DC bias from the controller DAC to the tip, similar to what have been discussed in the previous section.

By using the MAC III Signal Access Box, it is convenient to setup the PFM experiment with AC bias supplied to the sample. If it is necessary to apply the AC bias from Drive 2 directly to the tip with an external cable, then one need to use a specially modified nosecone which is discussed in the next section.

C. High voltage PFM with an external amplifier

The controller DACs and the drive outputs from the MAC III all have a range of +/-10V, thus limiting the experiments to materials of relatively high response and low switching potential. When the materials under test become less sensitive and need high voltage for polarity switching, then high voltage AC modulation and DC bias will be required. High voltage bias can only be applied to the sample through external cabling because the increased voltage will also increase the magnitude of interference with other AFM signals. Since the default voltage range from the system is +/-10V, a high voltage amplifier is needed to amplify the voltage to the desired value. Because the AC modulation usually runs from several kHz to several hundred kHz in frequency, the amplifier used has to have a relatively high speed as well.

The high speed, high voltage amplifier used in this experiment is the Keysight 33502A unit. The Keysight 33502A is a two-channel, isolated amplifier. Both channels can either propagate a signal directly or with 5x amplification. It also allows the user to select whether the channel should be AC or DC coupled. The bandwidth of the unit is > 300 kHz (-3dB) for small signal and 100

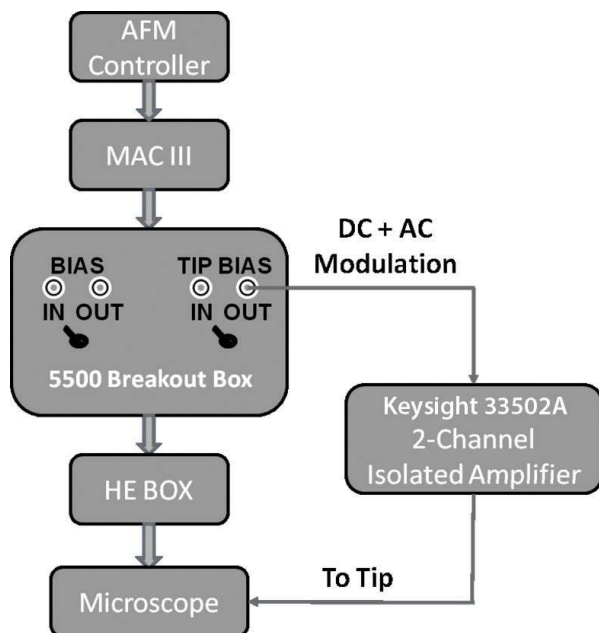


Figure 4. Block diagram illustrating the setup for PFM with high voltage AC/DC bias using a Breakout Box and an external high voltage amplifier.

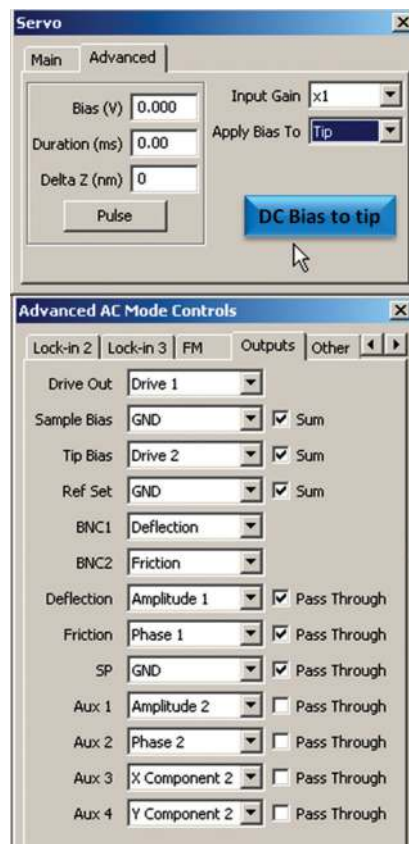


Figure 5. Software settings for generating the low voltage AC/DC bias to be amplified by the high voltage amplifier.

kHz @50Vpp for full power output. The Keysight 33502A has a very low noise, < 40nV/rt-Hz @ 1 kHz. The maximum output voltage is 50Vpp at maximum power. The use of the Keysight 33502A here is a simple choice of convenience. There are other commercial units available for this application as well. For example, the Falco Systems WMA-280 high voltage amplifier and the Trek Model PZD350 M/S piezo driver/amplifier are all suitable candidates. It is up to the user to decide on the one with the right speed and the right output voltage for their experiments. The user should also take proper safety precautions when using high voltage amplifiers.

The high voltage amplifier is connected to the system via the Breakout Box for the 5420/5500/5600 system, as illustrated in the block diagram in Figure 4. The Breakout Box is connected to the MAC III using a short 44-pin cable to minimize signal coupling in the line; and is connected to the HE with a regular 44-pin cable. As parameter

settings shown in Figure 5, when the "Apply Bias To" control in the "Servo" window is set to "Tip", and the "Tip Bias" control in the "Outputs" window is set to "Drive 2" with the "Sum" box checked, the AC drive signal from LIA2 of the MAC III and the DC tip bias from the controller DAC are mixed by a sum circuit inside the MAC III controller and sent to the "TIP BIAS"/"OUT" BNC connector on the Breakout Box. The "OUT" BNC connector is connected to the input of the high voltage amplifier using a regular BNC cable. There is a toggle switch on the Breakout Box beneath the "TIP BIAS" connectors. This toggle switch should be pushed towards the "IN" connector, in which case the voltage from the MAC III will not be sent to the tip through the 44-pin cable anymore, rather serves only as the input signal to the high voltage amplifier. The output from the high voltage amplifier gives the desired high voltage bias signal for PFM. This high voltage bias signal can be connected directly to the cantilever through an external cable. The sample needs to be connected to

the microscope using the 3-pin EC cable to close the circuit, i.e., to be grounded or biased by the sample bias DAC.

The regular nosecone is connected to the tip bias DAC through the steel spring clip and the metal core of the multipurpose scanner. In order to supply the bias from the amplifier directly to the cantilever via external cabling, the nosecone has to be modified. The modification includes 1) the cutting and bending of the tail of the spring clip so that it will not contact the metal core of the scanner, i.e., the electric connection between the cantilever and the internal tip bias DAC is broken open; 2) a 3-pin connector is soldered to the metal spring clip to connect the cantilever to the output of the high voltage amplifier. A photo picture of the whole setup including the MAC III, the Breakout Box, the high voltage amplifier, the microscope and the scanner with the modified nosecone is shown in Figure 6 (next page). The red wire with the 3-pin connector soldered to the nosecone is long enough to avoid any mechanical stress to the scanner.



Figure 6. Photos showing the hardware components for high voltage PFM, particularly the modified nosecone with electric lead to connect to the output of the high voltage amplifier.

Examples

Periodically poled lithium niobate (PPLN) is a domain-engineered lithium niobate crystal with its ferroelectric domains pointing alternatively to the +c and the -c direction. It is obtained by electrical poling with periodically structured electrode. The sample used here has alternating domains of 15–20 μm wide strips, as shown in Figure 7. Since the surface of the PPLN is polished without etching to expose the alternating domains, the topography image is fairly smooth without noticeable structures. The PFM images (amplitude and phase),

on the other hand, clearly reveals the existing alternating c+ and c- domains. Because the two domains differ only in the direction of polarization, the PFM amplitudes shows only small difference between them, and a minimum at the domain interface due to the cancelation of opposite movements of c+ and c- domains. The 180° phase angle (+/-5V, with 18°/V) clearly indicates the totally opposite polarization direction of the neighboring domains. As stated earlier, the PFM signal measured will increase with the AC voltage of the applied modulation bias. The effect of the modulation bias is illustrated in Figure 8,

where two line profiles from the PFM amplitude images obtained using a 20V peak-to-peak modulation and that using a 40V peak-to-peak AC modulation are compared together. The increase in PFM amplitude with AC bias voltage is quite obvious from these data. Figure 9 (next page) shows the results from a poling experiment on PVDF film deposited on a Si substrate. PVDF thin film is typically 50-60% crystalline. When poled, PVDF is a ferroelectric polymer. The piezoelectric coefficient of the poled PVDF thin film is about 6–7 pC/N, which is about 10 times larger than that observed for other polymers. Unlike other popular piezoelectric materials, such as PZT, PVDF has a negative d33 value. Physically, this means that PVDF will compress instead of expand or vice versa when exposed to the same electric field. In this experiment, a -25V DC bias was applied to the AFM tip and an area of 8 x 8 μm was first scanned in contact mode. Then the tip bias was switched to +25V and a 4 x 4 μm area was scanned in the center of the 8 x 8 μm region. Finally, PFM imaging was done over a 12 x 12 μm area to visualize the effect of poling. It is evident from the PFM amplitude image that there exist some crystalline phases in the film that shows stronger piezo effect after poling. There are also some domains that show minimum piezo effect and are affected little by the applied electric field. The PFM phase image also clearly shows a 180° phase difference between the -25V poled region and the +25V poled region, indicating that the polarization direction can be switched

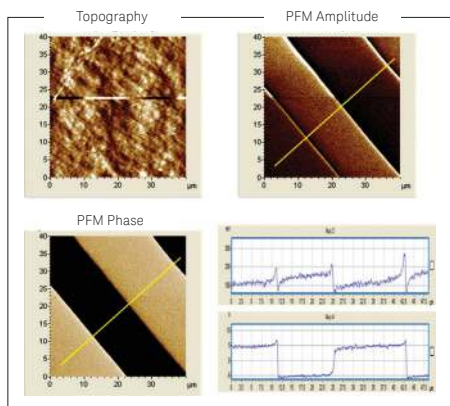


Figure 7. PFM imaging of PPLN with a 20kHz and 40V p p ac modulation.

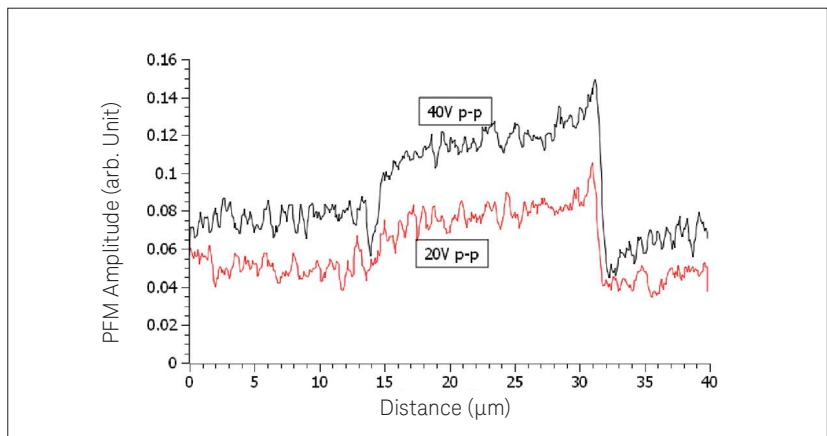


Figure 8. Increase of the PFM amplitude with the increase of AC modulation voltage.

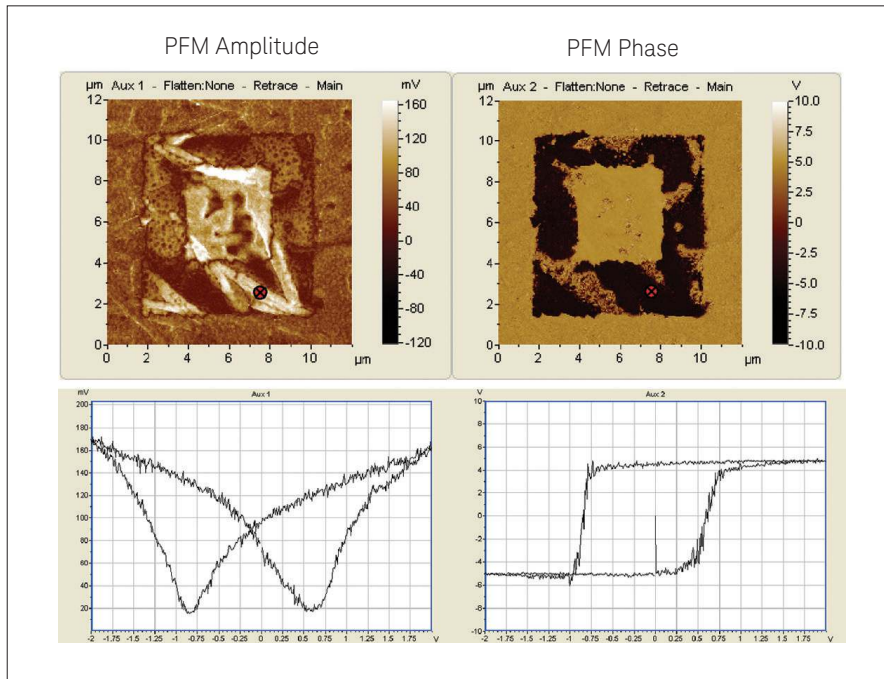


Figure 9. Poling of PVDF film and the hysteresis curve at the position indicated by the red marker. The film was poled with a -25V tip bias for an $8\text{ }\mu\text{m}$ area, then a $4\text{ }\mu\text{m}$ area in the middle was poled with a $+25\text{V}$ tip bias, and finally a $12\text{ }\mu\text{m}$ area was scanned to reveal the poling effect. The hysteresis curve was obtained between $+10$ and -10 Volts, the axis shows $\pm 2\text{V}$ that was amplified 5x by the external amplifier before being applied to the tip.

when the applied electric field change direction. This switching process can be studied by the hysteresis curve. The hysteresis curve presented in Figure 9 is collected at the point indicated by the red arrow tail marker on the image, which is located on one of the piezo active phases. The voltage applied has been amplified 5x by the external high voltage amplifier (Keysight 33502A), therefore the reading of 1V on the x axis of the hysteresis curves corresponds to 5V in reality. From the phase curve, it is

seen that the film will start switching polarization at about $\pm 3.5\text{V}$ (the coercive field). The shift of the center of the hysteresis curve from 0 volts suggests the possible existence of internal bias field in this aged sample. Even though the coercive field is much lower than the applied poling voltage, which means the polarization switching starts at a much lower bias, the application of a higher poling voltage increases the switching rate and the completeness of the process

Summary

A number of PFM configurations using the Keysight AFM systems are described and the key parameters involved in PFM experiments and their appropriate settings in the software are discussed. The necessity and possible approach of doing high voltage PFM are demonstrated with samples like PPLN and PVDF thin film deposited on Si substrate.

AFM Instrumentation from Keysight Technologies

Keysight Technologies offers high-precision, modular AFM solutions for research, industry, and education. Exceptional worldwide support is provided by experienced application scientists and technical service personnel. Keysight's leading-edge R&D laboratories are dedicated to the timely introduction and optimization of innovative and easy-to-use AFM technologies.

www.keysight.com/find/afm

For more information on Keysight Technologies' products, applications or services, please contact your local Keysight office. The complete list is available at: www.keysight.com/find/contactus

Americas

| | |
|---------------|------------------|
| Canada | (877) 894 4414 |
| Brazil | 55 11 3351 7010 |
| Mexico | 001 800 254 2440 |
| United States | (800) 829 4444 |

Asia Pacific

| | |
|--------------------|----------------|
| Australia | 1 800 629 485 |
| China | 800 810 0189 |
| Hong Kong | 800 938 693 |
| India | 1 800 112 929 |
| Japan | 0120 (421) 345 |
| Korea | 080 769 0800 |
| Malaysia | 1 800 888 848 |
| Singapore | 1 800 375 8100 |
| Taiwan | 0800 047 866 |
| Other AP Countries | (65) 6375 8100 |

Europe & Middle East

| | |
|----------------|---------------|
| Austria | 0800 001122 |
| Belgium | 0800 58580 |
| Finland | 0800 523252 |
| France | 0805 980333 |
| Germany | 0800 6270999 |
| Ireland | 1800 832700 |
| Israel | 1 809 343051 |
| Italy | 800 599100 |
| Luxembourg | +32 800 58580 |
| Netherlands | 0800 0233200 |
| Russia | 8800 5009286 |
| Spain | 800 000154 |
| Sweden | 0200 882255 |
| Switzerland | 0800 805353 |
| | Opt. 1 (DE) |
| | Opt. 2 (FR) |
| | Opt. 3 (IT) |
| United Kingdom | 0800 0260637 |

For other unlisted countries:
www.keysight.com/find/contactus

(BP-09-23-14)