Piezoresponse imaging

In piezoresponse mode (PR) a conducting AFM probe is brought into contact with the sample surface c.f. contact mode imaging. An applied AC bias between the tip and sample yields a piezoelectric response of the sample and may be detected as a first harmonic component of the deflection signal. The applied AC bias should be at a frequency that is too high to affect the deflection signal used by the feedback system to measure topography. PR mode provides information on the polarisation through local piezoresponse; however, contrast in PR images may include contributions from Maxwell stresses, polarisation switching below the tip, etc. Despite the difficulties related to the quantification of PR imaging it is readily used qualitatively to determine the out-of plane component of local polarization, i.e., domain orientation.

Piezoresponse imaging may be carried out using a basic Dimension® or MultiMode® series AFM with a NanoScope® IV controller, or a NanoScope® IIIa controller with a Quadrex® phase extender without the need for additional hardware. You should use NanoScope® software version 5.30xx or later. PR mode is available after selecting “Piezo Response” under the “Microscope Mode” parameter from the “Other Controls” panel. This will configure the hardware and the “Feedback” panel to allow access to the required parameters necessary for PR experiments.

Set up the AFM as for contact mode using a conducting, or metal-coated probe. The softer platinum-iridium coated SCM-PIC probes work well for PR mode but due to their inherently low resonance frequency of around 10 KHz they will not perform well in Tapping® mode. If a stiffer lever is required then the SCM-PIT levers may be used. These have a resonance frequency of around 70 KHz and may therefore also be used for Tapping® mode imaging.

For PR mode the deflection setpoint should be set approximately 2 Volts above the free-air deflection for SCM-PIC cantilevers but reduced to around 0.2 Volts when using the PIT levers. The system should be engaged on the surface with the AC/DC biases disabled (set to zero) and a good topographical image obtained. This will allow the imaging force, via the deflection setpoint, as well as the feedback gains to be adjusted so as to reduce to a minimum any tip/sample wear. When the height data is satisfactory the biases required for PR imaging may be applied.

N.B. Obtaining a good topography image with Tapping® mode prior to attempting PR may reveal areas of interest whilst minimising damage to the sample that may occur with contact mode.

The experimental set-up for PR mode is shown schematically as figure 1 below. The AC excitation bias may be applied to either the tip (left) or the sample (right). The standard electrical configuration for a NanoScope® IV or NanoScope® IIIa/Quadrex® system is shown as figure 2.

N.B. Although the AC or DC bias may be applied either to the tip or the sample, these configurations are mutually exclusive when in PR mode. If the AC bias is applied to the tip then any DC offset must be applied to the sample and vice versa.

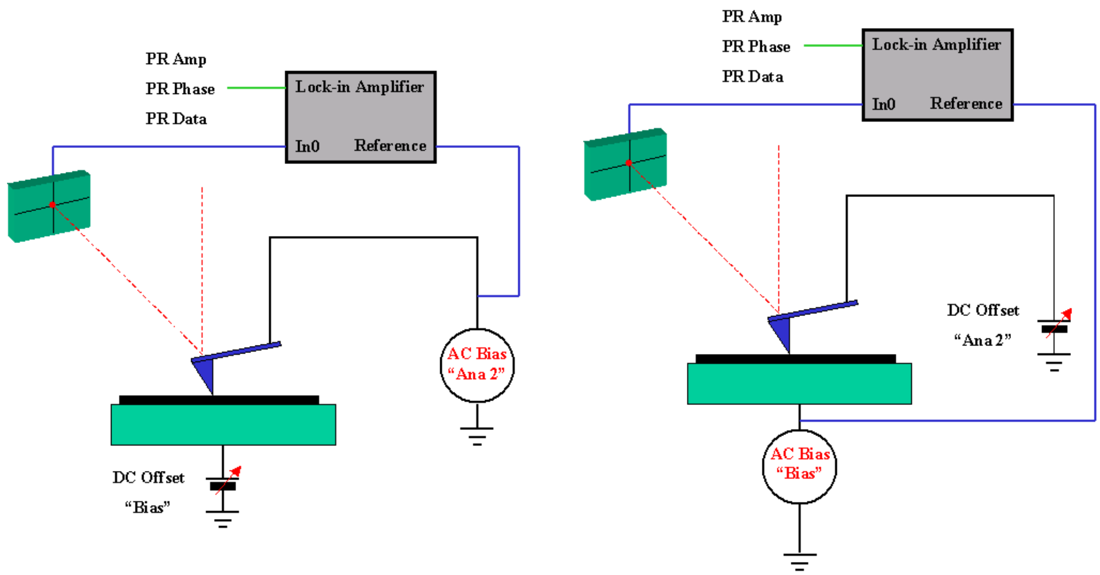

Figure 1. Piezoresponse imaging can be done with the AC Bias applied either to the tip (left) or the sample (right).

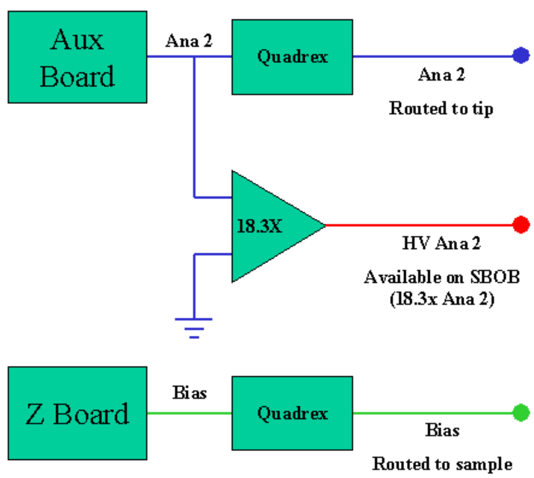

Figure 2. The tip and sample bias with the AFM in it's default configuration NanoScope® IV or a NanoScope® IIIa/Quadrex® system.

The tip may be biased with up to ±12 Volts using the Ana2 signal line and the sample to ±10 Volts with the Bias line. In contact and tapping modes these biases may be applied simultaneously giving an available tip/sample potential difference of up to 22 Volts. Further, within the NanoScope® controller the Ana2 line is split. The default path passes through the Quadrex® extender, integrated into the NanoScope IV, and is connected directly to the tip. The second path forms the input to a high voltage amplifier located on the Aux board within the controller. The gain of this amplifier is fixed at 18.33x, and hence yields ±220 Volts with the ±12 Volts available on the Ana2 DAC. If DC voltages in excess of this range are required the HV Ana2 line may be used. This signal line however, is only available to users with a signal access module (SAM). The BNC connector associated with the HV signal line is on the bottom row of the SAM and is labelled "Ana2" in red. It should not be confused with the Ana2 low voltage signal line on the third row labelled in black. To apply this HV signal to the tip it will be necessary to connect the HV Ana 2 "Output" BNC to the low voltage Ana 2 "Input" with a suitable BNC to BNC cable and set the Ana 2 low voltage selector switch to “EXT→MIC”. Alternatively the HV may be applied to the sample by way of the “Bias” connector on the SAM. For further details refer to the SAM support note.

The AC bias frequency and amplitude will depend greatly upon the sample being studied. It is beyond the scope of this manual to determine the bias frequency and amplitude required for individual samples. However, it is recommended that the bias frequency chosen be away from the mechanical resonance frequency of the cantilevers being used.

With the AC bias either disabled or applied to the sample you have direct access to the analog 2 parameter and hence to HV analog 2. When analog 2 is set to a DC level the HV analog 2 line is automatically set to 18.33x that value, figure 3 below. If more accurate control of the HV bias is required then it may be set directly using the “High Voltage” parameter. This will set the analog 2 line to the correct value to give the desired high voltage setting, figure 4 below.

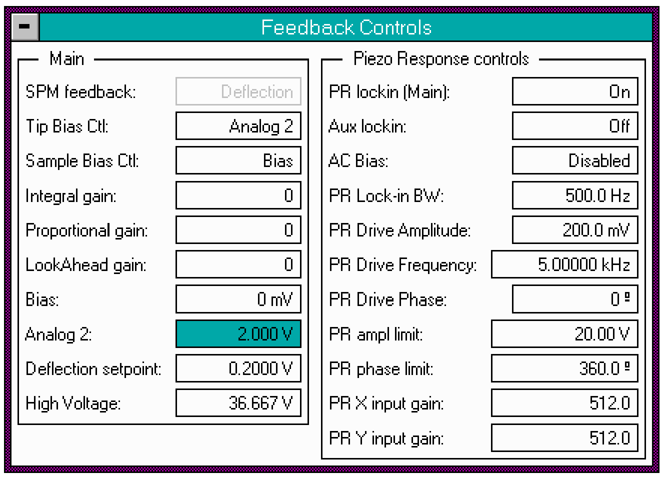

Figure 3. The HV signal line is scaled to 18.33x that of the analog 2 line.

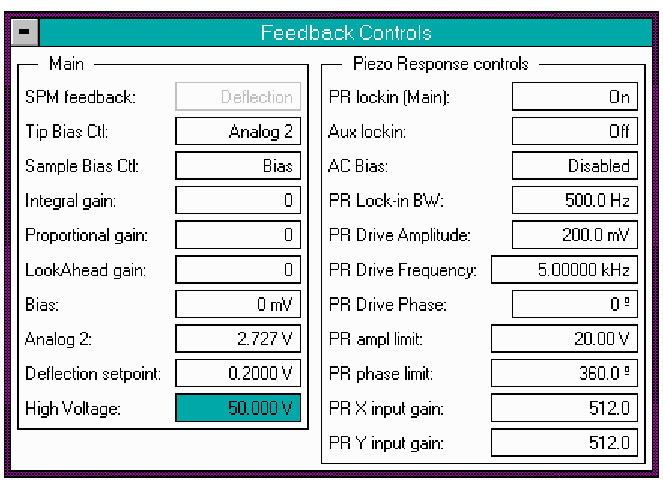

Figure 4. Direct access to the high voltage parameter gives more accurate control of the applied bias.

If the AC bias is applied to the tip however, the "Tip Bias Ctl" parameter will be deleted, analog 2 will be set to zero and greyed out preventing further access to the tip bias control, figure 5. This will result in the high voltage also being set to zero due to architectural limitations of the controller. In this configuration the low voltage analog 2 DC signal does not propagate beyond the extender electronics. However, it is still possible for the analog 2 DAC within the NanoScope® controller to generate a bias and hence for the high voltage amplifier to be available. In order to overcome this apparent shortcoming, analog 2 prior to the extender electronics, is set to the same value as the "Bias" parameter, figure 6. Although low voltage Ana 2 is not available at the tip, the high voltage amplified signal may still be accessed via the SAM. This configuration will limit the high voltage available to ±183.33 Volts; the bias line is only able to supply ±10 Volts. As in the previous case the value of the high voltage may be set explicitly using the "High Voltage" parameter.

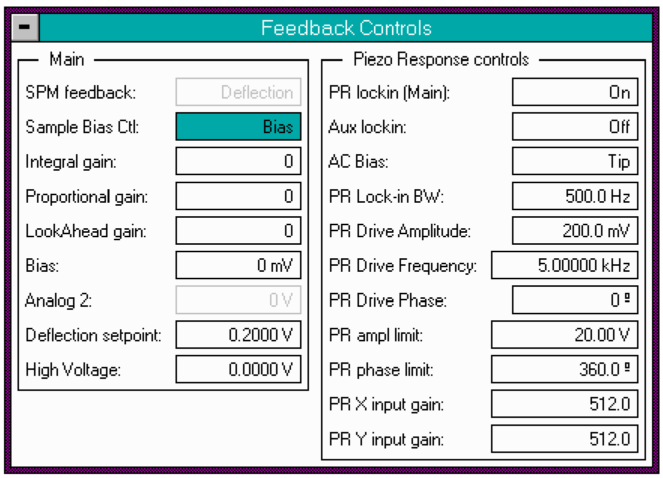

Figure 5. The "Tip Bias Ctl" is removed when the AC Bias is routed to the tip.

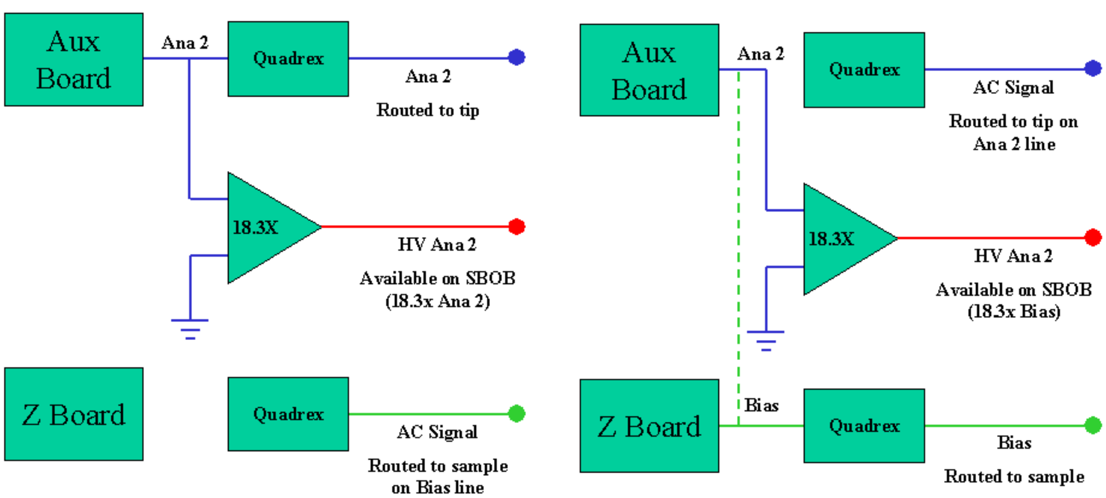

Figure 6. With the AC bias routed to the sample there is a direct correlation between the Ana 2 and HV Ana 2 signal lines (left). However, the low voltage Ana 2 signal is not available once the AC bias is routed to the tip (right). In this situation the Ana 2 DAC on the aux board is set to mirror the value of the Bias DAC yielding the HV signal.

Software Control

All of the PR parameters required for imaging are available to the user via the "Feedback" panel. The deflection signal is set as the input to the primary lock-in amplifier identified as the "PR lock-in" in the feedback panel. Therefore, when using PR mode the primary lock-in should be turned on, figure 7 below. The secondary lock-in amplifier is not used during PR mode and no signals are routed to it. This should therefore be turned off for imaging in this mode.

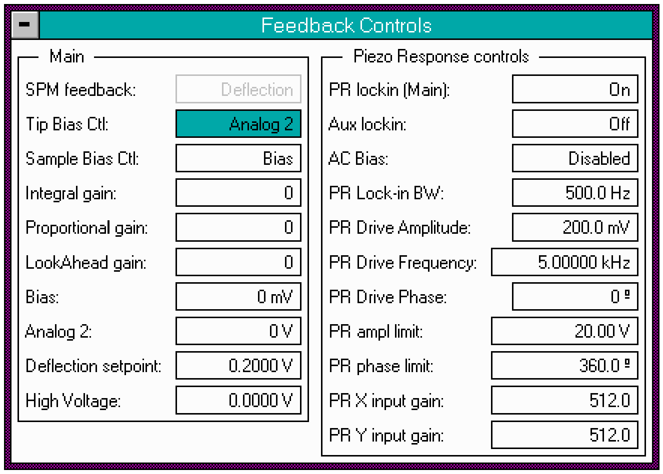

Figure 7. Ensure that the primary lock-in is turned on.

PR lockin (main)

Turns the primary lock-in amplifier **On**/**Off**. This lock-in amplifier is used for PR mode and must be set to **On** whilst collecting data. If the primary lock-in amplifier is set to **Off** then the PR data types will not be available. The Aux or secondary lock-in is not used and settings associated with it are not important for PR mode imaging.

AC Bias

Selects whether the AC bias is applied to the tip, the sample or is disabled. The AC bias must be applied to either the tip or sample in order to collect any data from a PR sample.

PR Lock-in BW

Sets the bandwidth of the primary lock-in amplifier. This should be kept low (≤ **500 Hz**) in order to avoid topographical artefacts being displayed with the PR data.

PR Drive Amplitude

Sets the amplitude of the applied AC bias. This should be kept low whilst imaging to prevent switching of domains within the sample. The precise value of this parameter will be sample dependent and is beyond the scope of this manual.

PR Drive Frequency

Sets the frequency of the applied AC bias. The precise value of this parameter will be sample dependent and is beyond the scope of this manual.

PR Drive Phase

Sets the drive phase of the AC bias.

PR ampl limit

Sets the limit for the ADC on the amplitude data type. Reducing this value will result in less pixelation for data with a low amplitude response.

PR phase limit

Sets the limit for the ADC on the phase data type. Reducing this value will result in less pixelation for data with a low phase response.

PR X input gain

Gain applied to the X data from the lock-in amplifier prior to that data being passed to the DSP for amplitude/phase calculation. **4096** represents a gain of **1, 2048** a gain of **2** etc. This should be reduced to increase the overall gain of the lock-in system when looking at samples with extremely weak PR response. It is anticipated that this parameter be changed in parallel with the PR Y input gain.

PR Y input gain

Gain applied to the Y data from the lock-in amplifier prior to that data being passed to the DSP for amplitude/phase calculation. **4096** represents a gain of **1, 2048** a gain of **2** etc. This should be reduced to increase the overall gain of the lock-in system when looking at samples with extremely weak PR response. It is anticipated that this parameter be changed in parallel with the PR X input gain.

Tip Bias Ctl

Selects the bias connection to the tip. In order to apply a DC bias to the tip this should be set to Analog 2. The parameter will not be available when the AC bias is applied to the tip.

Sample Bias Ctl

Selects the bias connection to the sample. In order to apply a DC bias to the sample this should be set to Bias. The parameter will not be available when the AC bias is applied to the sample.

Bias

DC bias applied to the sample.

Analog 2

DC bias applied to the tip.

High Voltage

The voltage output of the HV Ana2 line on the signal access module if installed. Required voltages in excess of those normally found on the Bias/Analog 2 lines may be typed directly into this panel. This parameter is present even if the SAM is not connected. However, in this case the HV Ana 2 signal cannot be accessed.

PR mode specific data types

When the software is configured for PR mode, the PR specific data types described below will be available. These data types may be selected from the Channel 1, 2 and 3 panels in the usual manor. With a NanoScope® IIIa/Quadrex system it is only possible to collect data of a single PR data source at any one time. However, with the NanoScope® IV it is possible to collect data from one or more PR data channels simultaneously.

PR Amplitude

Amplitude output from the lock-in amplifier as calculated by the DSP. Amplitude data is always positive. PR amplitude yields information about the magnitude of the polarisation of the sample at the AC driving frequency but not it's direction.

PR Phase

Phase output from the lock-in amplifier as calculated by the DSP. Phase data may be positive or negative. Yields information about the direction of the polarisation of the sample at the AC driving frequency. Phase data contains no information about the magnitude of the polarisation.

PR Data

A hybrid data channel that combines data from both the PR Amplitude and PR Phase channels. The amplitude data is multiplied by the sign (±) of the phase. This data type therefore contains information about both the magnitude and direction of the sample polarisation at the AC driving frequency.