Kelvin Probe Module II

User Manual

Version 4

08 / 2018

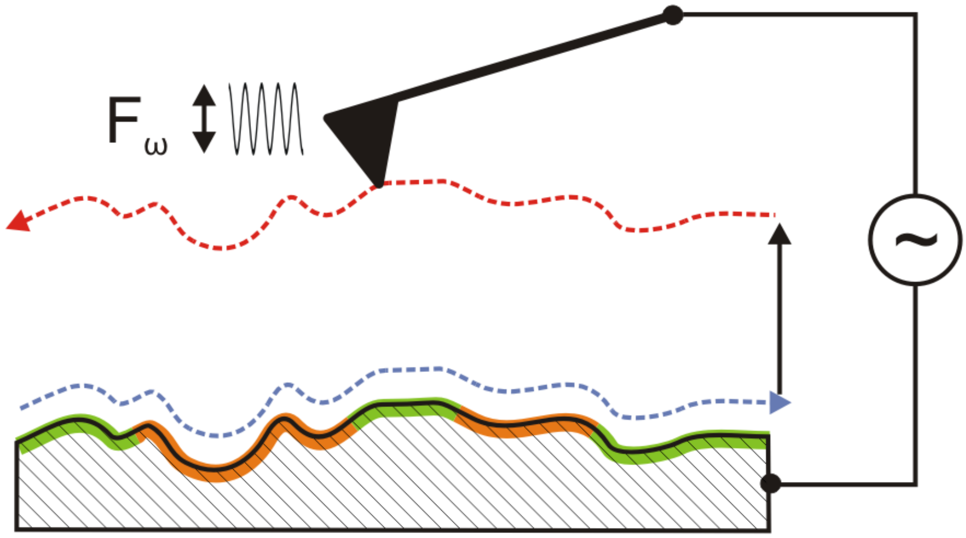


Table of Contents

§ 0 Introduction

The KelvinProbe Module is designed for use with the JPK NanoWizard® AFM.

§ 1 Safety instructions and warnings

The KelvinProbe Module is designed for use with AFMs of the JPK NanoWizard® Series.

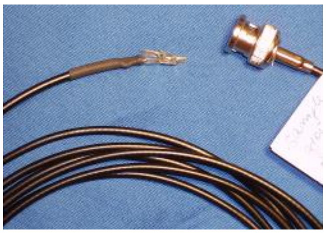
This manual is supplementary to the main NanoWizard Series user manual. All safety instructions and warnings of the NanoWizard Series user manual also apply here.

Please read this manual and all manuals belonging to the supplied components carefully.

Please contact JPK for assistance (++49 30 726243 500) support@jpk.com.

§ 2 Components and installation

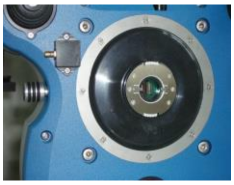
2.1 Components

	Cantilever holder fixed spring with electrical tip connection and corresponding cable.
	T-type BNC branch connection
	SMB-Harwin adapter with 2 screws (might be already attached to AFM head)
	1 cable with 2 SMB plugs, one side has an SMB-BNC adapter attached
	1 cable for sample grounding (crocodile clip and BNC)
	1 connection cable BNC-BNC

2.2 Hardware Setup

There are minor alterations in the electrical connection between the *standard* AFM controller (without signal access module) and *advanced* AFM controller (with signal access module). In addition, a special stress relief has to be mounted to the JPK NanoWizard® head.

2.2.1 Mechanical setup



The plastic connector, called SMB-Harwin adapter, is fixed to the AFM head with 2 screws.

2.2.2 Electrical Connection to the advanced AFM controller

1. Connect T-type BNC branch connector to Precision 5 (red circle)
2. From one end of the T-type connector connect a BNC cable on Axis 8 Out (white circle). For some system configurations this connection should be made to a different axis output (e.g. Axis 4 Out). When the KPM panel opens (see section 3.2), it will display which axis is used.
3. Use the SMB to BNC cable to connect the other end of the T-type connector with the Harwin-adapter attached to the AFM head
4. Connect the BNC end of the BNC to crocodile clip cable to Precision 6 (grey circle) and use the crocodile clamp to ground the sample

The front panel of the controller should now look like this:

2.2.3 Electrical connection to the standard AFM controller

For controllers without signal access module use the following connections.

1. Connect T-type BNC branch connector to Precision 5 (red circle)
2. From one end of the T-type connector connect a BNC cable on Axis 4 Out (white circle).
3. Use the SMB to BNC cable to connect the other end of the T-type connector with the Harwin-adapter attached to the AFM head
4. Connect the BNC end of the BNC to crocodile clip cable to Precision 6 (grey circle) and use the crocodile clamp to ground the sample

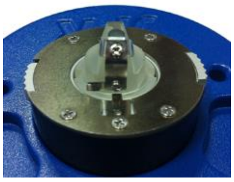
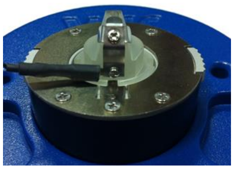
2.2.4 Cantilevers

Electrically conductive cantilevers for intermittent contact mode are required for Kelvin Force experiments. Cantilevers with a resonance frequency of 75 kHz and more can be used for KPM experiments. Standard AC mode or force modulation mode cantilevers made from doped silicon possess sufficient conductivity. An aluminum coating on the detector side is unnecessary.

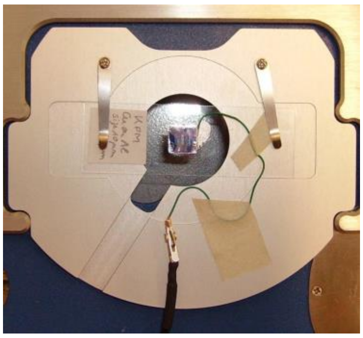
We recommend Pt coated force modulation cantilever e.g.:

- Multi75E-G (BudgetSensors), EFM (NanoWorld)
- Tap300E-G (BudgetSensors), NCHPt (NanoWorld)

2.2.5 Connecting the Electrically Conductive Cantilever

	**1** If the fixed spring glass block is used, mount the cantilever as usual and tighten the fixation screw carefully.
	**2** Attach the connector wire to the contact on the cantilever spring of the glass block
	**3** Connect the SMB plug to the SMB-Harwin adapter fixed to the AFM head.

2.2.6 Grounding the Sample



Always ground the samples or ground the metal support of the sample.

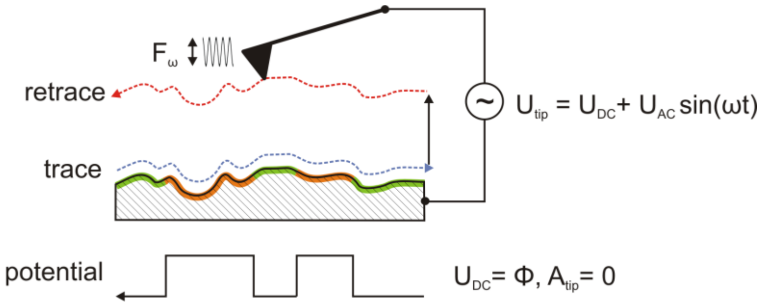
In case of non-conductive samples, stick them on a conductive support and connect it to the crocodile clip on the grounding cable. A piece of mica (non-conductive) could be glued onto a metal stub. The stub then should be grounded.

To avoid a ripple pickup use one of the BNC connectors of the SPM Control Station as ground. Note: Only the outer circular part of the BNC connector is grounded.

§ 3 Kelvin probe microscopy

3.1 Principle

KPM is usually carried out in HoverMode. In a first pass (trace) over the sample the topography is measured, and on the return pass (retrace) this height information is used to maintain the cantilever at a constant offset height above the surface. The cantilever needs to be conductive and has a connection to an external circuit to control the tip potential U_{tip}. This potential U_{tip} consists of dc component U_{DC} and a small ac component U_{AC}, modulated at a frequency ω which is normally set to the resonance frequency of the cantilever oscillation without any mechanical driving oscillation being applied directly to the cantilever.



If there is a difference between the surface-potential Φ and the applied tip potential U_{tip} a capacitive force F_{cap} will act on the cantilever. Since U_{tip} is modulated by U_{AC} this capacitive force is also modulated and causes an oscillation of the cantilever with amplitude A_{tip} (see figure below). Thus, if the potential U_{DC} applied to the tip differs from the surface potential Φ the cantilever oscillates with an amplitude $A_{tip}>0$.

$$F_{cap}(x,y) = -\frac{\partial C}{\partial z}\left[\frac{1}{2}\left(U_{DC} - \phi(x,y)\right)^2 + \frac{U_{AC}{}^2}{4}\right]$$

$$F_{\omega}(x,y) = -\frac{\partial C}{\partial z}\left(U_{DC} - \phi(x,y)\right)U_{AC}\sin(\omega t)$$

- F_{cap} capacitive force between tip and sample
- F_{ω} oscillating force at the frequency ω
- U_{DC}, U_{AC} dc and ac terms of the applied voltage
- $\phi(x,y)$ surface potential
- C tip-surface capacitance

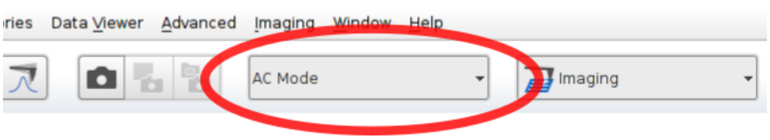
Therefore, during the hover pass over the surface, the feedback is used to vary the dc potential applied to the cantilever tip to minimize the cantilever oscillation A_{tip}. This uses the X channel of the Lock-in signal rather than the normal oscillation amplitude, to find the zero point stably (ideally the phase contribution goes from +1 to -1 as the amplitude goes through zero). When the cantilever oscillation is zero, the applied voltage U_{tip} is equal to the surface potential difference Φ and hence this U_{tip} can be saved as the surface potential difference "image".

3.2 Software Setup

This is a step-by-step tutorial to the first KPM image. It assumes that the KPM connections have been made as described in section 0.

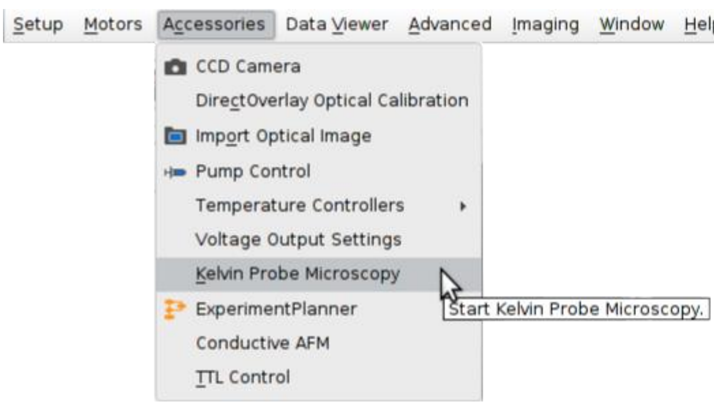
The outputs of the lock-in amplifier are lock-in 1x and lock-in 1y which are physically amp cos phi and amp sin phi.
The channels for recording the surface potential difference and the KPM-feedback used in the experiment are as follows:

	JPK SPM Control Station
Surface potential difference	Precision 5
KPM-feedback	Lockin 1 X / Amp Cos Phi



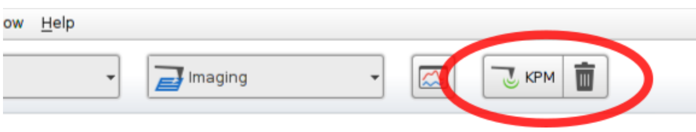
1

Start SPM and select AC Mode.



2

Choose Kelvin Probe Microscopy from the Accessories menu.



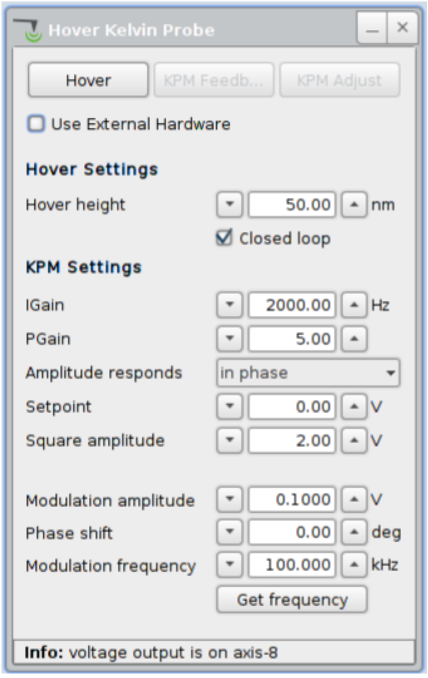
3

Two new buttons will appear in the Toolbar. The “KPM” button opens the KPM-window, while the 2nd button resembling a trash can removes the buttons and closes the window.

4

The Kelvin probe window contains all parameters necessary for the KPM experiment:

As long as you are not clicking somewhere there will be info at the bottom of the appeared panel about the selected output channel. Please check whether this connector is used.



Hover: switch on the hover mode, which always has to be done for a KPM-experiment

KPM Feedback: switch on the feedback to control the dc voltage for the KPM measurement

KPM Adjust: to adjust the KPM gain settings a rectangular test waveform can be applied to the tip to simulate a variation of the surface potential (see below)

Hover height: the height above the surface at which the tip is held during the retrace segment of the AFM scanning

Closed loop: if checked, the height measured channel will be used for recording the height profile in trace and the same channel will be used to control the hover on the retrace

I-Gain: integral gain of the KPM feedback

P-Gain: proportional gain of the KPM feedback

Amplitude responds: The user should determine whether the oscillation amplitude of the cantilever responds *in phase* or *out of phase* with the variation of the surface potential during the KPM-measurement in retrace. This should be set correctly before the KPM feedback is enabled (see below).

Setpoint: amplitude setpoint of the KPM-feedback, in most cases this value is zero

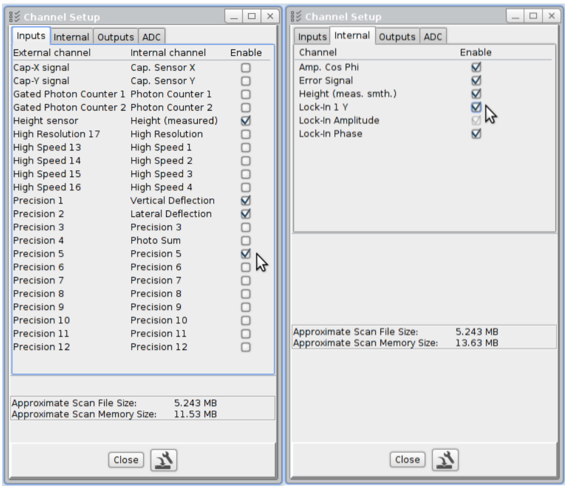
Square amplitude: amplitude of the test waveform (see “KPM-adjust”)

Modulation amplitude: modulation amplitude of U_{tip} applied between tip and sample (U_{AC})

Phase shift: phase offset for the lock-in detection of the cantilever oscillation

Frequency: modulation frequency of U_{tip}, usually set to the resonance frequency seen in the ac mode tuning window

Get Frequency: clicking on this button will import the frequency currently applied for ac mode imaging



5

Make sure that the channel **Precision 5** is enabled in *Setup* → *Channel Setup*. While using the **JPK SPM Control Station** also **Lockin 1 X / AmpCosPhi** has to be enabled for the use in the KPM-feedback.

The channel **Precision 5** will record the surface potential difference during retrace.

Additionally, enable **Precision 5** for the retrace in *Setup* → *Saving settings* to save the KPM-image

3.3 Setting up the KPM-Experiment

This section describes the procedure for selecting the feedback settings for the KPM image. The familiar feedback settings displayed in the standard *Feedback* window of SPM only influence the “normal” topography imaging settings during trace. The feedback settings displayed in the KPM-window only influence the KPM amplitude during retrace. The KPM feedback settings for I- and P-gain cannot be compared with those values for “normal” imaging I- and P-gain.
The procedure for optimizing the gain settings for KPM-imaging involves applying a test waveform to the cantilever. This test waveform is a rectangular with amplitude set by the Square Amplitude parameter to simulate steep changes in the surface potential. Then the feedback is optimized to respond to these steep changes.

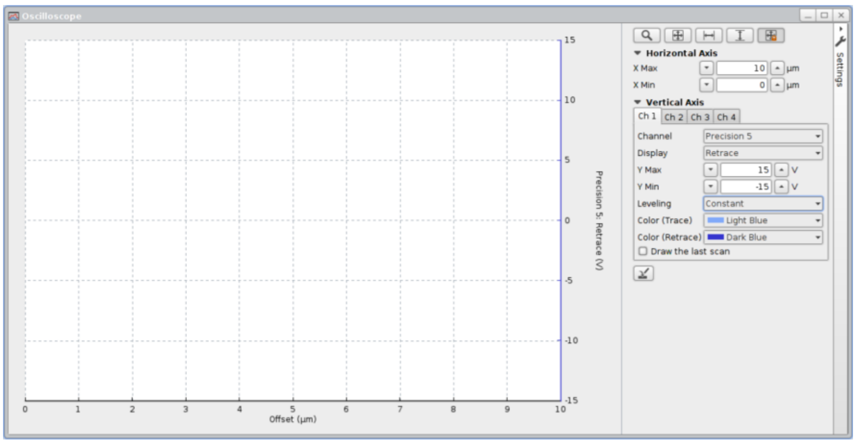
This is the procedure for optimizing the KPM feedback settings:

1

Select an image size of 1 nm x 1 nm

2

Open the oscilloscope from the tool bar in the SPM program At the top of the SPM program select the Oscilloscope button, a window named Oscilloscope will open. When setting up the Oscilloscope, enter the following settings:

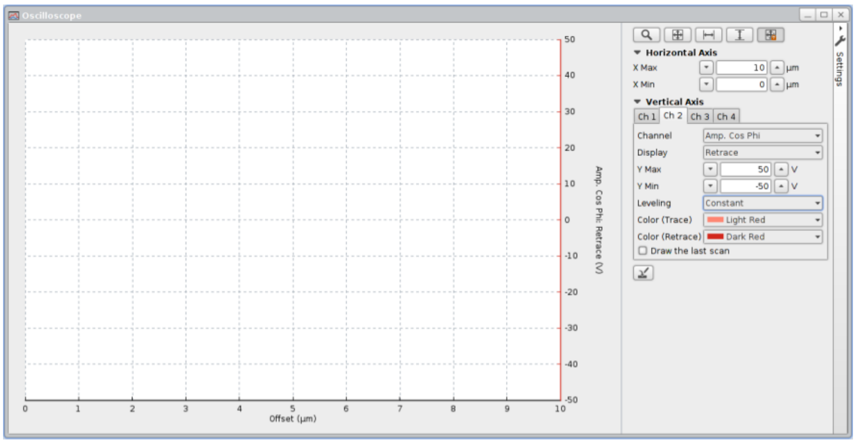


3

Modify the channels to be displayed to:

Channel 1 (Ch1)

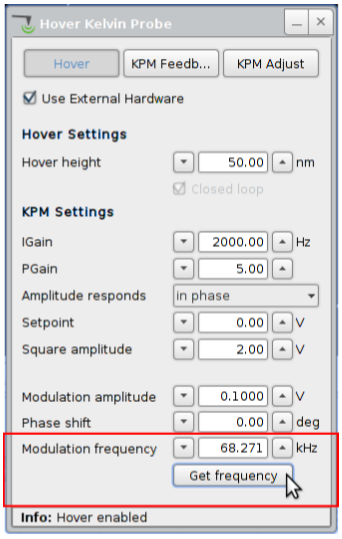
- select **Precision 5**
- select *retrace*
- levelling to *constant*



4

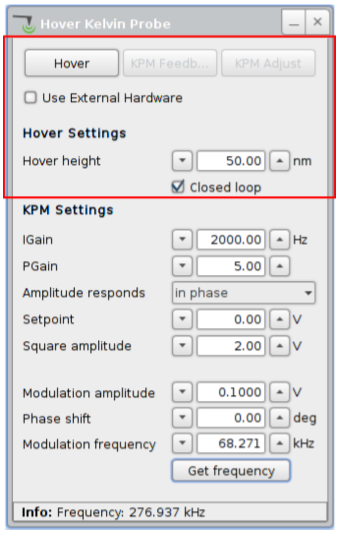
Channel 2 (Ch2)

- select **Lockin 1 X / AmpCosPhi**
- select retrace
- set Levelling to constant



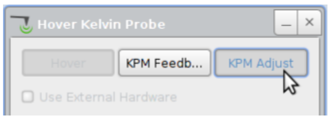
5

The modulation frequency will be automatically set to the value selected for the cantilever excitation during the cantilever tuning. If the excitation frequency was changed after the Hover Kelvin Probe window was loaded, pressing the *Get frequency* button will update the frequency.



6

Set the *Hover height* to 50nm, enable the *Closed loop* and activate the *Hover* mode. If hovering is enabled the closed loop settings cannot be changed. To change this setting disable hover mode, change the closed loop settings and re-enable hover mode.



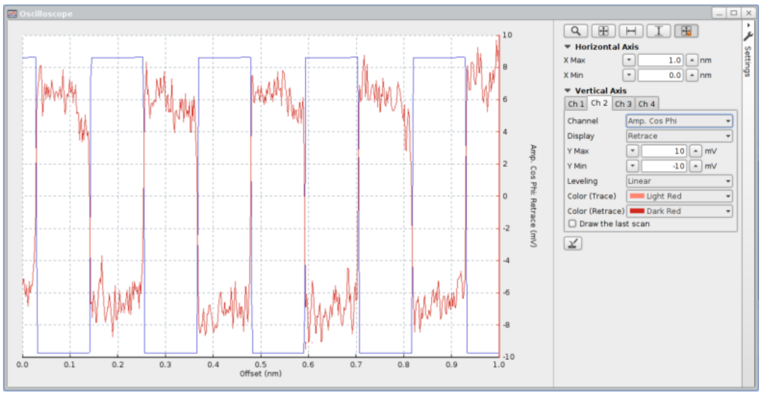
7

To help with adjusting the KPM feedback settings a square wave has to be applied to the tip by activating *KPM Adjust.*

8

Approach cantilever.

Press “Run”. This causes the AFM to begin to scan the sample. The instrument now continuously scans an image of 1 x 1 nm in size.

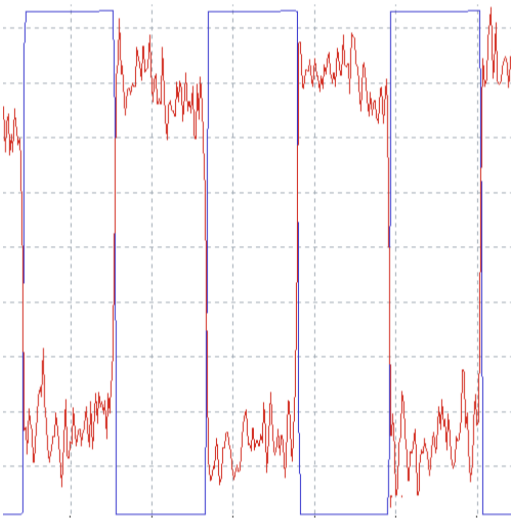


9

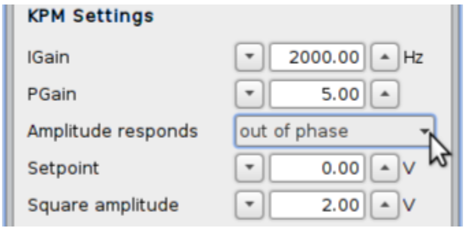
You should see something as displayed on the left: blue rectangles (**Precision 5**) and more “noisy” red rectangles (**Lockin 1 X / AmpCosPhi**).

10

Increase the value for the *Phase shift* and observe the oscilloscope. During increasing / decreasing the response of the **Lockin 1 X / AmpCosPhi** should change. Stop the *Phase shift* when the red rectangles have maximum amplitude. Alternatively, observe the channel **Lockin 1 Y** and adjust the *Phase shift* for minimum amplitude in this channel before switching back to **Lockin 1 X / AmpCosPhi**, which is then maximized as a result.

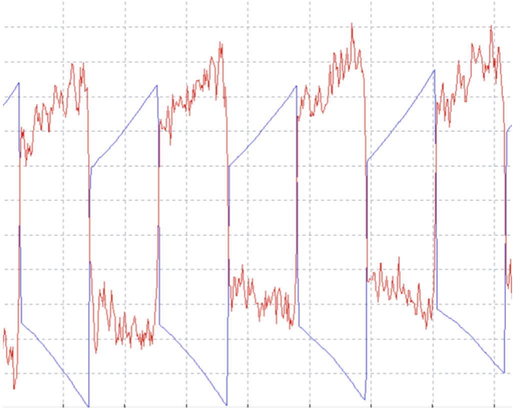


Observe whether the blue rectangles and the red response are “in phase” or not and select in the *Amplitude responds* combo box accordingly.



In our case (see screenshot on the left) the signals are “out of phase”.

If the amplitude response is low and too noisy, it is always good to work with *Modulation amplitude* values of 2.0 V or higher (maximum value 10.0 V). Ideally this parameter should be set as small as possible.
The value for square amplitude does not have to be changed, usually.

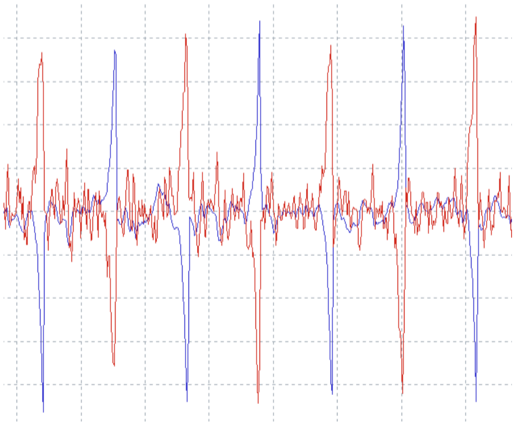


11

Enable the KPM-feedback by pressing the *KPM-Feedback* button.

Now increase *I-Gain*. Typical values are between 100 and 1000 Hz. Increase *I-Gain* until you see something like it is displayed on the left: A **spiky blue Precision 5** curve.

Now, increase *P-Gain* until you see oscillations in the blue **Precision 5 signal**. If there is oscillation, reduce *P-Gain* to half of its value.



12

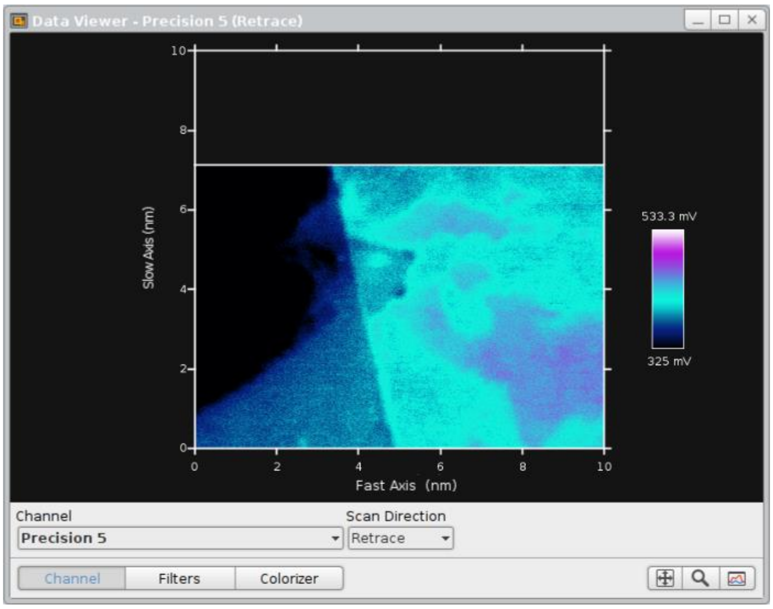
The image on the left represents the optimal settings for the feedback control.

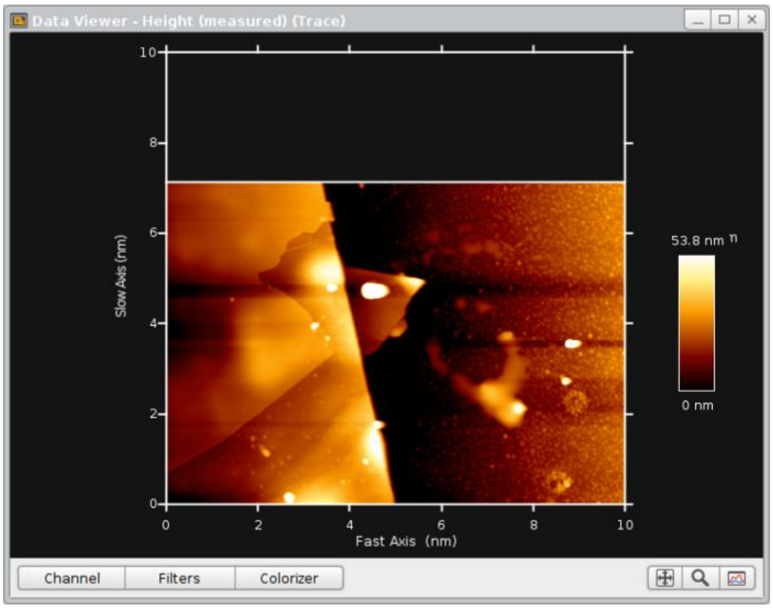
The average of the blue **Precision 5** (U_{DC}) curve represents the value for the surface potential difference Φ at the 1 x 1 nm^2 square.

Finally disable *KPM Adjust* while keeping *KPM-Feedback* activated. The KPM is now ready for the experiment.

3.4 Performing the KPM experiment

For KPM imaging, a suitable scan-range can now be selected and the regular imaging can be started.





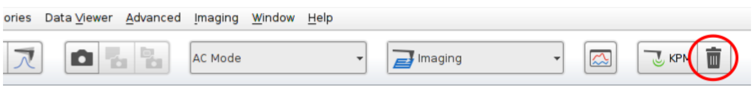
1

Open two data viewers, one for the height image (trace) and the other for the KPM surface potential difference recorded on **Precision 5** (retrace).

If the precision 5 signal is rather noisy, it may help to lower the KPM gains or increase the modulation amplitude.

The hover height also determines the image quality. It should be as low as possible, but sufficiently high. The overall sample roughness determines the hover height. If it is too low, the cantilever will run into the sample during the retrace scan. Usually this happens first on steep edges and can be observed as an artefact in the channel lock-in amplitude retrace.

During image saving make sure that you save the **Precision 5** retrace signal.



2

When the KPM experiment is finished, switch off the KPM-Feedback and the Hover mode and close the KPM script by pressing the close button in the toolbar and disconnect the connections made in 0.

3.5 Troubleshooting

Problem	Possible cause	Correction
There is no Kelvin Probe Microscopy option in the Accessories menu.	Old software version.	Update the software: download the current software version for your instrument at https://customers.jpk.com/ and follow the instructions for software update. If that doesn't help contact support@jpk.com.

§ 4 Appendix

4.1 KPM Feedback parameters

Feedback-formula for KPM feedback control is:

$$DAC = P \times (ADC - Sp) + I \times \int (ADC - Sp) dt$$

were *ADC* is the voltage U_{DC} applied to the tip (read back into Precision 5), *Sp* is the setpoint (normally set to *Sp=0*) and *P* are the integral and proportional gains (*I-Gain, P-Gain*) and *DAC* the voltage U_{DC} outputted on Axis 4 and applied to the tip. The KPM *I-Gain* values are "true" Hz values.

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