ELECTRIC FORCE MICROSCOPY (EFM)

KELVIN FORCE MICROSCOPY (KFM)

OPERATION INSTRUCTIONS

Agilent 5500 Scanning Probe Microscope equipped with the MACIII accessory:

Electric Force Microscopy (EFM) and Kelvin Force Microscopy (KFM)

by John Alexander and Sergei Magonov

Outline

Foreword

New progress in Atomic Force Microscopy (AFM) is expected with the development of multi-frequency measurements. Multi-frequency studies were enabled with an Agilent 5500 scanning probe microscope after the introduction of the MACIII accessory. This technical note describes the essential features of the electronic controller and the MACIII accessory as well as their main functions. In addition we provide practical instructions for setting Electric Force Microscopy (EFM) and Kelvin Force Microscopy (KFM) experiments. The new instrumentation will help in development of novel capabilities for the examination of local mechanical and electromagnetic properties. It is obvious that a level of complexity in multi-frequency measurements is much higher as compared to the basic AFM procedures, and it will take some time for develop the most efficient experimental protocols that allow a full realization of the capabilities of such studies. During the past year, we were exploring multi-frequency methods for AFM-based studies of local electric properties and, particularly, in electric force microscopy (EFM) and Kelvin force microscopy (KFM). Our accumulated experience in implementing EFM and KFM studies of different samples is summarized below.

Microscope Controller and MACIII accessory

A block diagram of the Agilent 5500 scanning probe microscope, **Figure 1**, demonstrates how the MAC III accessory is incorporated between the electronic controller and head electronics module as well as connecting to a host computer. 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. The MACIII 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 auxiliary inputs and drive outputs are accessible through the MACIII signal access box. The software, which is flexible in routing signals back to the microscope controller, supports two servo systems related to these LIA. One LIA is used for AM tracking of sample topography with the probe peak-to-peak amplitude or its X-, Y- vector components used for feedback. This LIA is also provided with a Q-control function. The 2^{nd} LIA can be applied in the AFM-based electrostatic tip-sample force

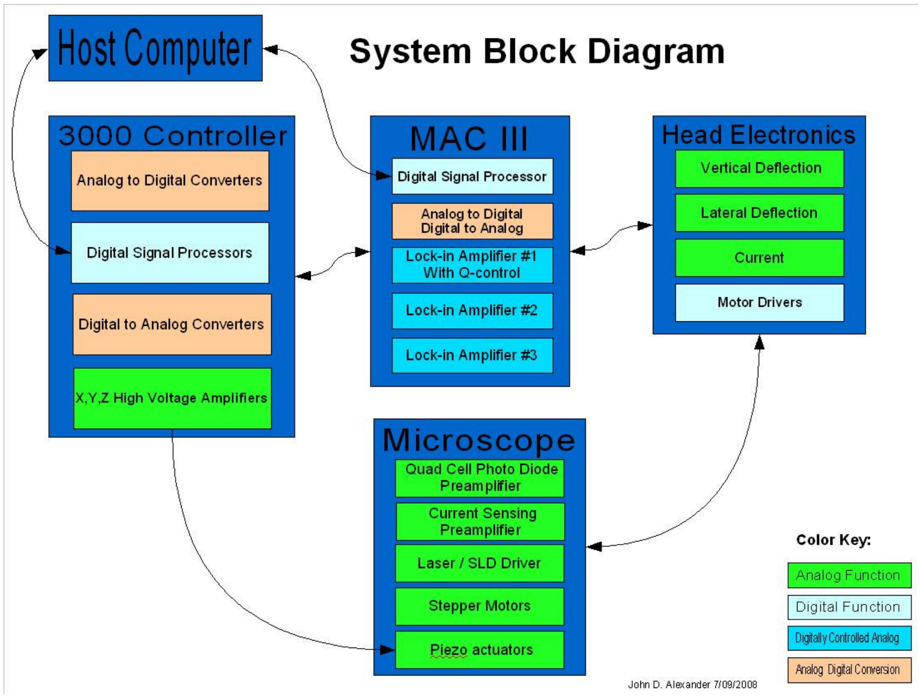


Figure 1. Block diagram of main components of Agilent 5500 scanning probe microscope.

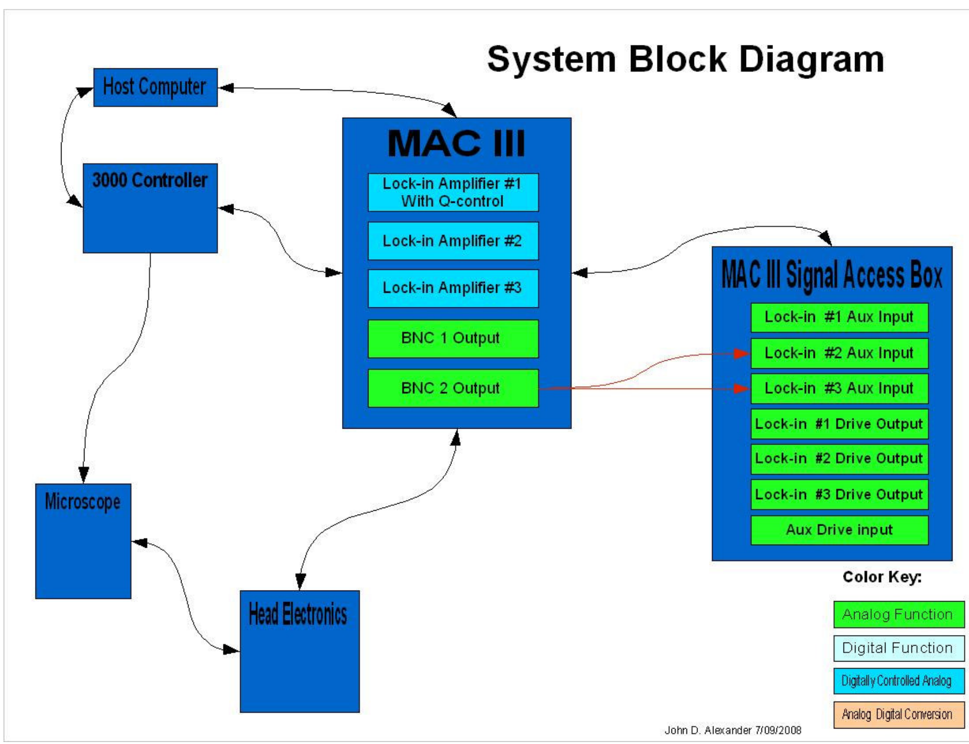


Figure 2. Block diagram showing the main components of the MACIII accessory and its Signal Access Box.

studies as shown below. The 3rd LIA can be set for monitoring various signals and can be applied for the adjustment of operation parameters. The MACIII Signal Access Box (**Figure 3**) provides a customer with the LIA's Inputs and Drive Outputs that might needed for the design of novel experiments. Particularly, the routing of Aux input channels of 2nd LIA and 3rd LIA to the BNC2 connector is used in one of the practical applications of KFM. In this case, the Aux input of the 2nd LIA brings in the phase signal of the 1st LIA, when the BNC2 connector is set to Friction from within the software. A T-shaped connector might be used to connect both LIA to the BNC2 of the MACIII box. In other cases, 2nd LIA might be connected to different signals, for example those that are delivered by Aux2 channel. In one of the KFM implementations, Aux 2 is set to the Y-vector component of 1st LIA and this signal is used in the KFM servo loop.

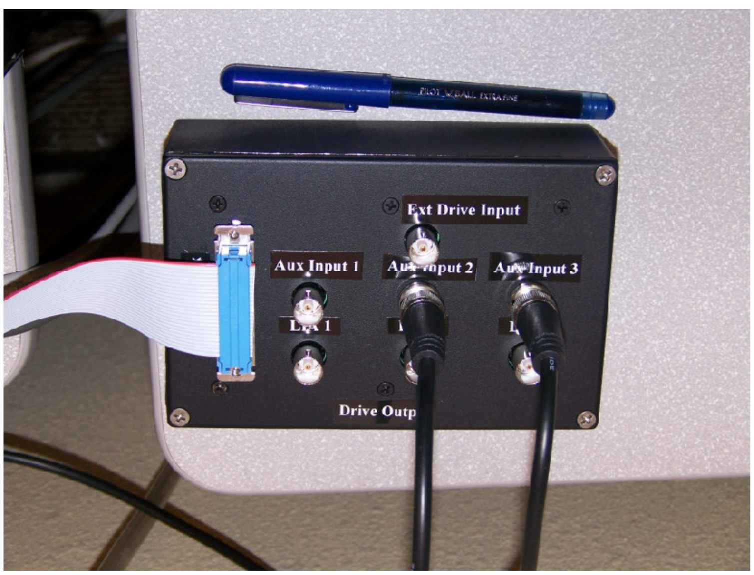


Figure 3. The MACIII signal access box with eth connection required for the KFM operation.

EFM and KFM Measurements: Set-ups, Samples and Probes.

EFM and KFM have been introduced almost 20 years ago, and these are two complementary techniques. In these techniques, an AC electric voltage is applied between a probe and a sample. If there are surface locations with different electric properties such as surface charges, doping level, dielectric constants etc they cause different electrostatic forces between the probe and these regions in the sample. These forces induce changes in the oscillation parameters (amplitude, phase) of the probe. In order to differentiate the probe response caused by mechanical and electrostatic force interactions, the latter are measured either at different frequencies (ω_{mech} and ω_{elec}) or at the same frequency (usually ω_{mech}) in normal and lift operations. The probe response due to the electrostatic forces is usually presented as a map/image that reflects spatial variations of the surface electrical properties. This is the essence of EFM. In KFM, the only difference is the use of an additional DC voltage, which is applied to the probe with the purpose of nullifying the effect of the electrostatic interactions. This is practically realized using an additional servo loop and the obtained DC voltage, which is equivalent to the surface potential, is measured in different surface locations and presented as surface potential image in KFM. In contrast to EFM, which is generally a qualitative method that requires complex analysis to convert the probe response into the quantitative electric properties, KFM directly provides the quantitative surface potential data. More about these techniques and their applications you can found in our Application Note.

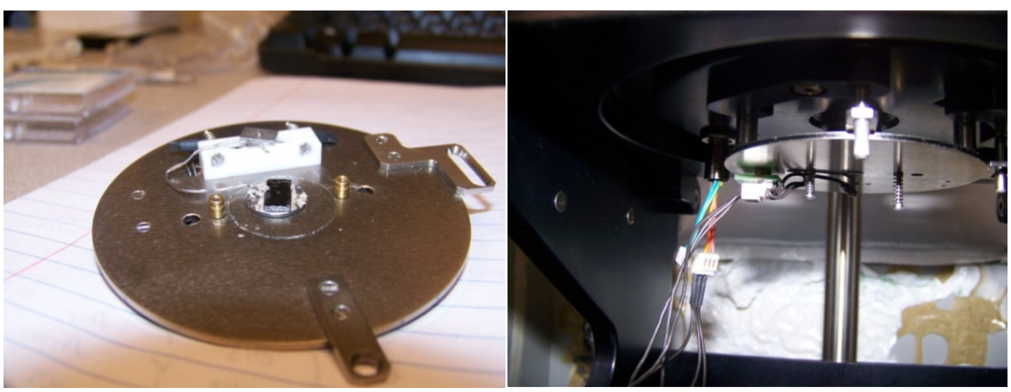


Figure 4. A typical installation of the sample for EFM and KFM measurements. Left – a piece of graphite with organic layer as a sample with grounding wire attached to the sample with a drop of conducting silver glue. Right – a grounding connection under the sample plate.

A sample for KFM measurements is placed on the sample plate and a wire connecting the sample to ground is fixed with a drop of conducting silver glue, **Figure 4** (left). Don't forget to connect the grounding cable to the bottom of the sample plate as shown in **Figure 4** (right). We found out also that for some samples EFM and KFM operation can be performed without grounding the sample, yet this operation might be less stable than the one with the grounded samples or substrates. In these experiments, we applied several types of conducting probes typically Pt-coated Si probes with cantilever stiffness in the 2-5 N/m range, which are made by different manufacturers (Olympus, NanoSensors and MikroMasch). TEM micrographs of representative probes (**Figure 5**) showed that their apex has a diameter in the 20 - 40 nm range. Our experience showed that the probes made by Olympus are generally sharper and their apex's dimensions are most consistent from probe to probe. In addition to the conducting probes, the

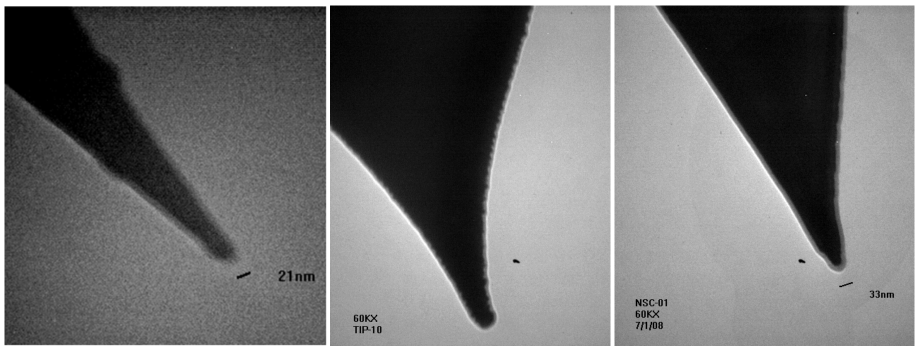


Figure 5. TEM micrographs of the conducting AFM probes made by Olympus (left), NanoSensors (center) and MicroMasch (right). The micrographs are courtesy of Bernard Mesa (MicroStar Technologies).

KFM and EFM measurements can be performed with regular Si probes, which usually have a conductivity level sufficient for these operations. However, the signal-to-noise ratio depends on a conductivity level therefore the Pt-coated probes are most suitable for the AFM-based electrostatic force measurements.

As we are showing here, the KFM measurements can be performed in a variety of different ways. The same is true for EFM studies, which does not require using the electric servo loop needed in KFM. KFM can be performed in two main operations, which we will name as AM-AM and AM-FM. In both cases, the amplitude modulation is applied for topography tracking with 1st LIA and the electrostatic measurements with 2nd LIA. For these studies 2nd LIA employs either the amplitude of the probe oscillation at ω_{elec} (AM-AM) or the phase changes at ω_{elec} (AM-FM). In addition, the phase signal can be also substituted by the Y- vector component of the amplitude at ω_{elec} that actually defines the phase changes. The block diagrams of KFM in the AM-AM and AM-FM operations are presented in **Figures 6** and **7**. The differences between running experiments in the AM-AM and AM-FM are clearly seen from the block diagrams. In the AM-AM configuration the 2nd LIA senses the photodetector signal in parallel with 1st LIA whereas in the AM-FM the 2nd LIA is connected in series with 1st LIA, and therefore, 2nd LIA processes the side band signal at $\omega_{mech} + \omega_{elec}$, which originates from modulation of the mechanical resonance with ω_{elec}. This modulation can be further enhanced by damping the quality factor (Q) of the probe at the mechanical resonance as shown below.

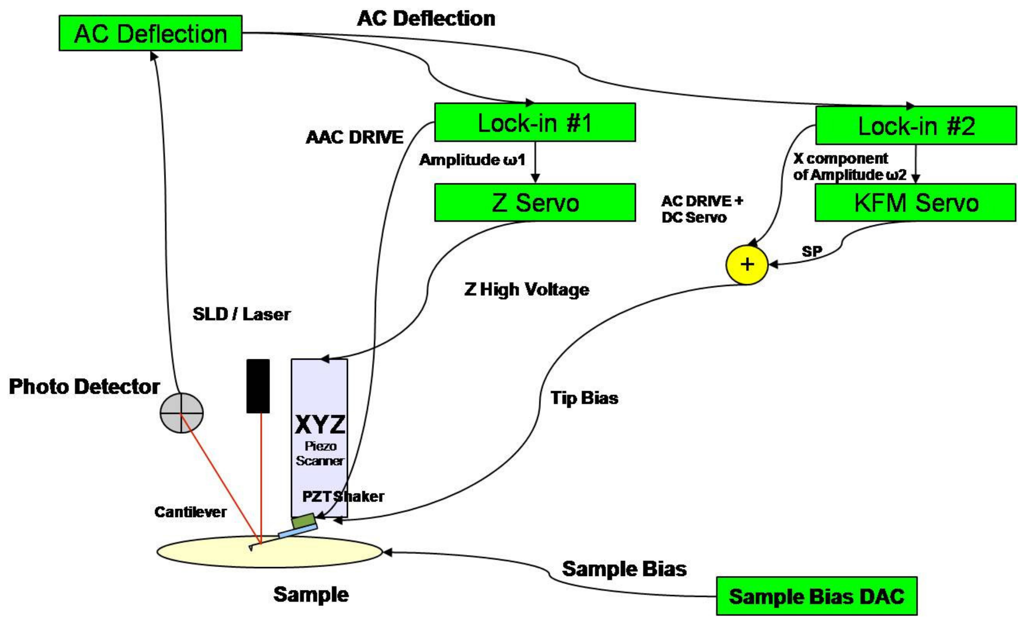


Figure 6. The block diagram of the AM-AM operation with the Z servo operating at $\omega_1 = \omega_{mech}$ and the 2nd KFM servo using the X- component of amplitude at $\omega_2 = \omega_{elec}$ for this purpose.

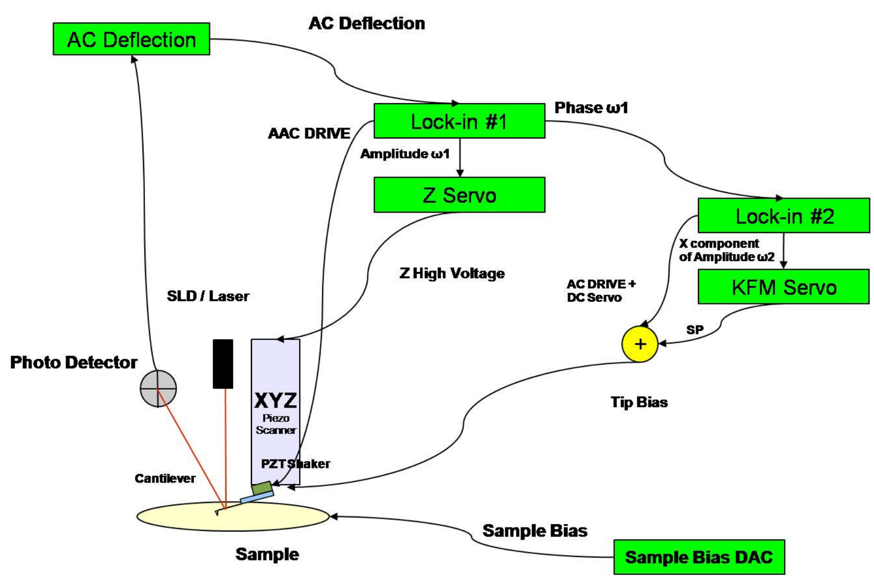


Figure 7. The block diagram of the AM-FM operation in KFM with the Z servo operating at $\omega_1 = \omega_{mech}$ and the 2nd KFM servo using the phase signal at $\omega_2 = \omega_{elec}$ for this purpose.

Tuning Procedures in AM-AM operation of KFM

First of all, we are describing an adjustment of the experimental parameters for the AM-AM operation using either the low frequency (say 10 kHz) or the 2nd flexural mode. It is worth noting that particular KFM is defined by the parameters of the 2nd LIA, Outputs and Other panels of the Advanced AC Mode Controls window. The parameters related to the performance of 1st LIA for tracking of surface topography are practically identical for all EFM/KFM operations. All of these measurements were performed in the intermittent contact regime with the parameters specified below. As the KFM probe, we chose a conducting NanoSensors probe. This probe has the following two signals in its frequency spectra: one around its 1st flexural resonance (58.75 kHz) and the other one of the 2nd flexural oscillation (374.4 kHz), which are shown in **Figure 8**.

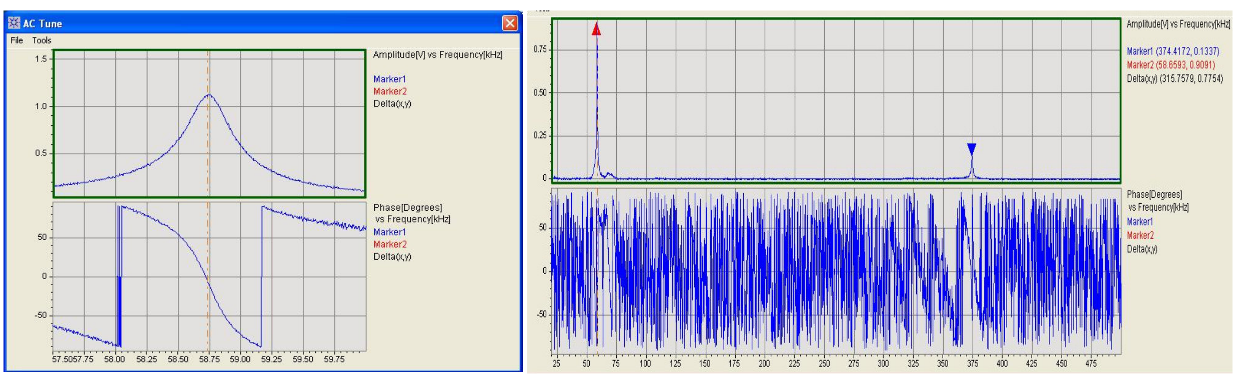


Figure 8. The frequency spectra of a conducting NanoSensors' probe in two spectral regions.

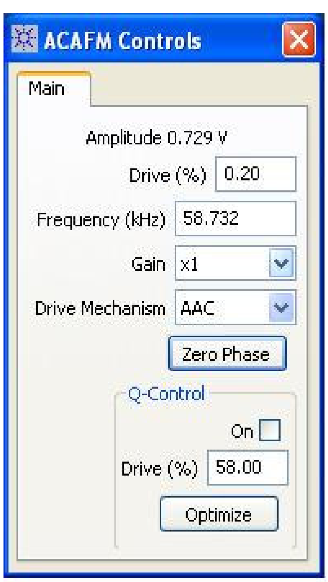
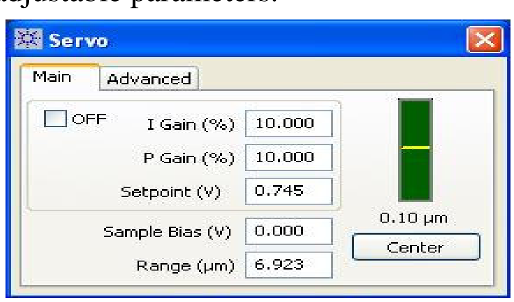
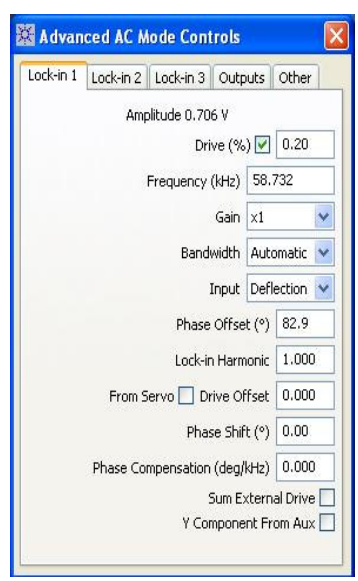


Figure 9. ACAFM Controls (left) and Servo (center) windows, which present the parameters employed for surface imaging. The 1st LIA panel of the Advanced AC Mode Controls' window (right) shows a broader army of user adjustable parameters.





The imaging parameters, which are used for topography of a sample surface, are shown below in the regular AC AFM Controls and Servo windows and some of them identically appear in 1st LIA panel of the Advanced AFM Controls window, which has also additional important parameters related to Phase, Input type, Bandwidth, etc. (**Figure 9**).

During the tuning procedures we are using also other panels of "Advanced AC Mode Controls". The parameters of the Outputs, 2nd LIA and Other panels adjusted for the AM-AM operation are shown in **Figure 10**. In the Outputs panel one should choose Tip Bias as the "Drive 2" signal (it

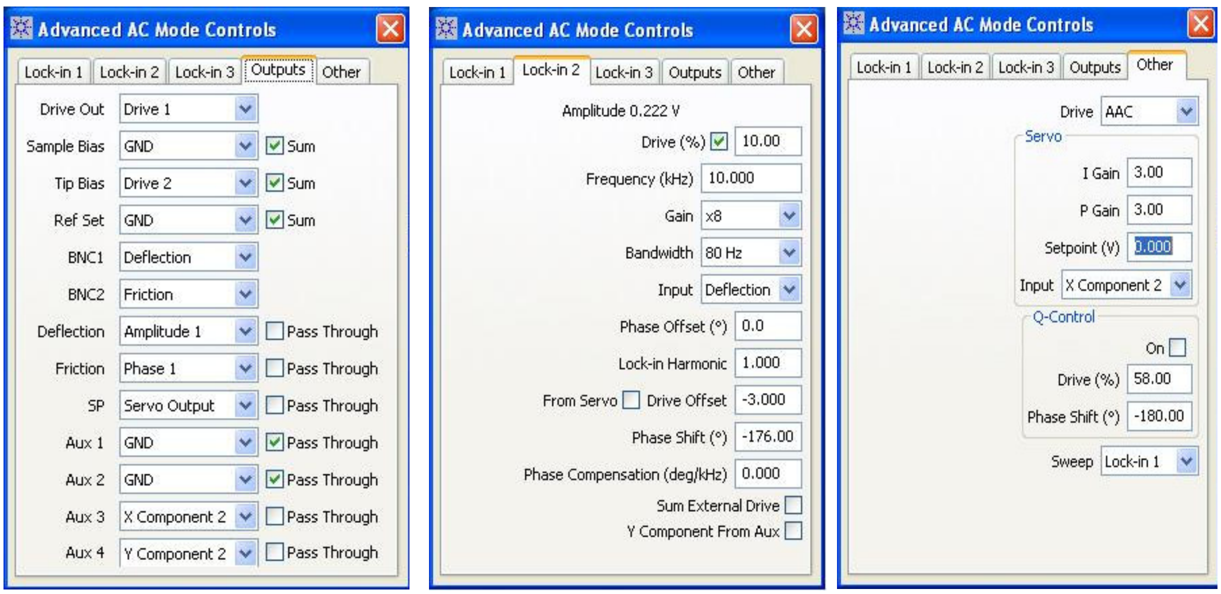
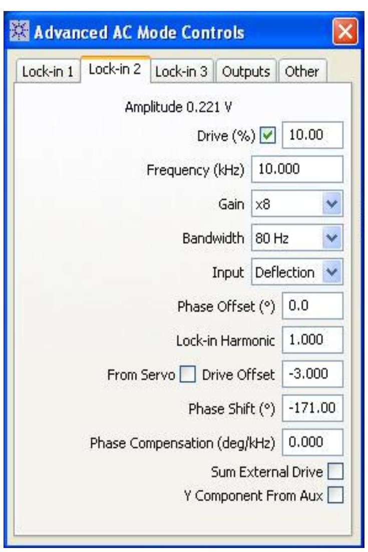


Figure 10. The panels of Advanced AC Mode Controls window for the AM-AM operation.

does not matter if the nearby "sum" box is checked or unchecked); SP as "Servo Output" with the unchecked "pass through" box nearby; Aux 3 and Aux 4 as the X Component 2 and Y Component 2, respectively, both with the unchecked "pass through" boxes. These settings define the outputs channels on the Outputs panel, **Figure 10** (left), to help monitor the adjustment of the phase of the 2nd LIA that should maximize the X-component 2 and minimize Y-component 2 of the 2nd LIA. This optimization is performed with the amplitude signal of the 2nd LIA (top of the 2nd LIA panel) typically above 0.1 V. This is achieved by chosing an appropriate drive voltage (in our example 10%, as seen from the 2nd LIA panel), gain (8 in our case), a low bandwidth (80 Hz in our case) and Drive Offset (-3V in our case). The Drive Offset voltage increases the electrostatic tip-sample force for tuning purposes, and choosing it at -3V level limits a possible influence of this voltage on the sample and tip. After these parameters are optimized so that the amplitude reaches some reasonable level (0.222V in our case), the researcher adjusts the Phase Shift to make the Aux 3 = X2 signal at the amplitude level and, respectively, the Aux 4 = Y2 signal around zero. As one sees from the 2nd LIA panel in **Figure 10** and Aux IO Channels in **Figure 11**, this condition was achieved with Phase Shift of -171 degrees (**Figure 12,** left).

Figure 11. The Aux IO Channels window showing values of Aux 3, Aux 4 and SP important for KFM adjustment and operation.

After the phase adjustment is done one should switch the Bandwidth in 2nd LIA panel to 1-2 kHz, **Figure 12** (center). Once this state is achieved the microscope is ready to operate in EFM, which can be realized by imaging the X-component 2 or Y- component 2 signals in different sample locations. This can be done by choosing additional image channels as Aux 3 = X2 or Aux 4 = Y2, respectively. One can also choose the imaging channels as Amplitude 2 and Phase 2 that provides identical information. All aforementioned parameters belong to 2nd LIA.



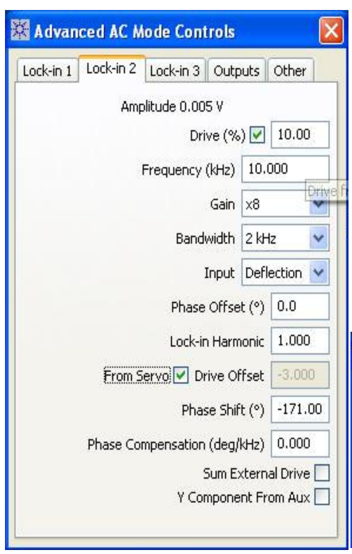
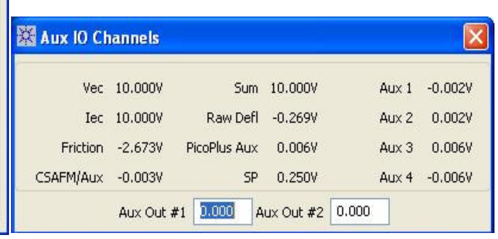


Figure 12. Left - the Advanced AC Mode Controls with the parameters optimized for EFM imaging. Center - the Advanced AC Mode Controls after switching on the KFM servo. Right - the Aux IO Channels panel in KFM mode.



The transition from EFM to KFM is made by checking the box "From Servo", **Figure 12** (center). The important parameters of the KFM feedback are the I and P gains, which are shown in the Other panel in **Figure 10** (right). Gains from 1 to 3 are typical for the feedback in the AM-

AM operation. In the same panel one can find the Input, which defines the signal (typically X2 component) employed by the servo loop, the Sweep – for choosing the LIA for sweep purpose, as well as the Setpoint for the following adjustment. Due to a potential exposure to external charges (say surface charges occasionally formed on a probe substrate or a nose cone window) the value of surface potential can be shifted or can depend on the probe-sample separation. The effect might be compensated by adding Setpoint in the Other panel, see **Figure 10**, right. This check is shown in **Figure 13**, where SP versus Z plot is recorded together with Amplitude versus Z curve. For this purpose, the SP channel should be chosen in the Advanced panel of Spectroscopy window (**Figure 13**, left), and amplitude-versus-Z and SP-versus-Z sweeps can be performed continuously (**Figure 13**, center) until the adjustment of the Setpoint (typically numbers are small, below 0.1 but can be positive or negative) makes the SP-versus-Z plot more or less straight. In our case the situation was fine (**Figure 13**, right) without adjusting the offset, but in other implementation of KFM (see below) the situation will be different.

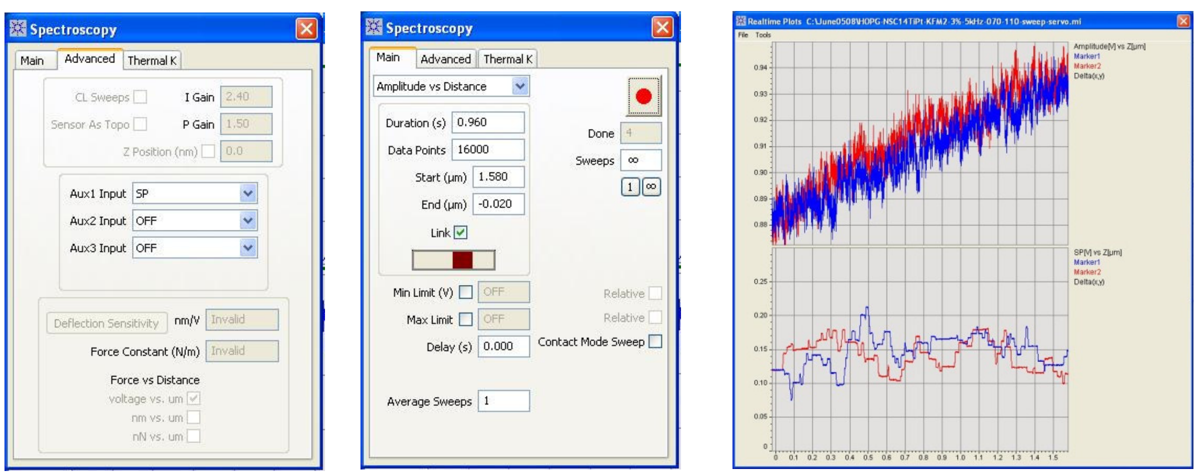


Figure 13. The windows needed for finding an appropriate voltage offset for the surface potential measurements.

At this point, the microscope can operate in the KFM mode and the imaging channel SP should be added to map the variations in surface potential. Fine adjustments can be continued further by tuning Bandwidth in the 2nd LIA panel, scanning rate, feedback gains in the Other panel, etc. An additional check of the efficiency of the KFM operation can be done with a use of 3rd LIA. It is the best to perform this check when scanning is not activated. First, one should choose the 3rd LIA in the Sweep window of the Other panel (**Figure 14**, left). Then, in the 3rd LIA panel (**Figure 14**, center) 80 Hz Bandwidth should be chosen. The Input should be set to

Deflection, which means that the sweep will provide the amplitude signal of the photodetector. The sweep Start/End points should be something like 2 kHz and 25 kHz (**Figure 14**, right) to define the frequency range where the electrostatically-driven signal at ω_{elec} = 10 kHz and at $2\omega_{elec}$ = 20 kHz will be displayed. The idea is to see how large the signal is related to the electrostatic tip-sample interactions and how optimal is the KFM feedback in nullifying these interactions by applying DC voltage to the probe. In other words, the frequency sweeps should be performed when the KFM servo is in "off" and "on" states. If the amplitude signals (i.e. the amplitude at 10 kHz in the sweep made in the "off" state, is not large enough, it can be adjusted larger or if the same amplitude in the "on" state in not small enough it can be adjusted smaller) are not satisfactory then one can make the necessary adjustments, for example, by increasing the drive voltage in the 2nd LIA panel or by optimizing the gains of the KFM feedback in the Other panel.

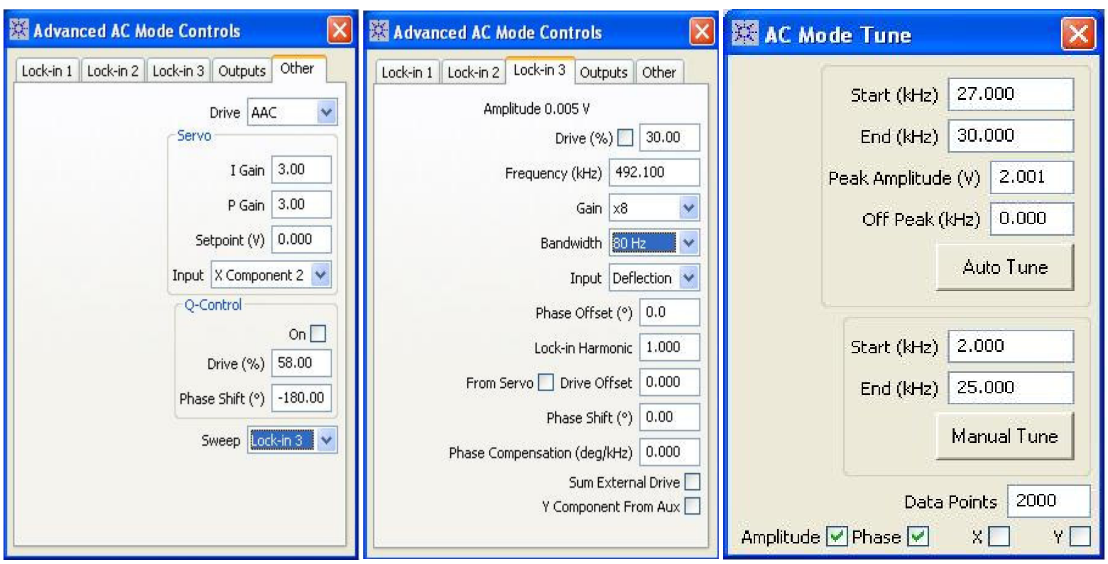


Figure 14. (left, center) The Other and 3rd LIA panels with the parameters needed for the check of the KFM performance. Right – the Start/End sweep points in the AC Mode Tune window.

The amplitude-vs-frequency curve obtained with the sweep made in the "off" state of the 2nd LIA (**Figure 15**, left) shows the strong signal at 10 kHz and very small one at 20 kHz. The amplitude-vs-frequency curve in the "on" state shows only a minor signal at 10 kHz with the signal at 20 kHz being larger. This sweep was made with the Gain parameters of the KFM servo loop equal

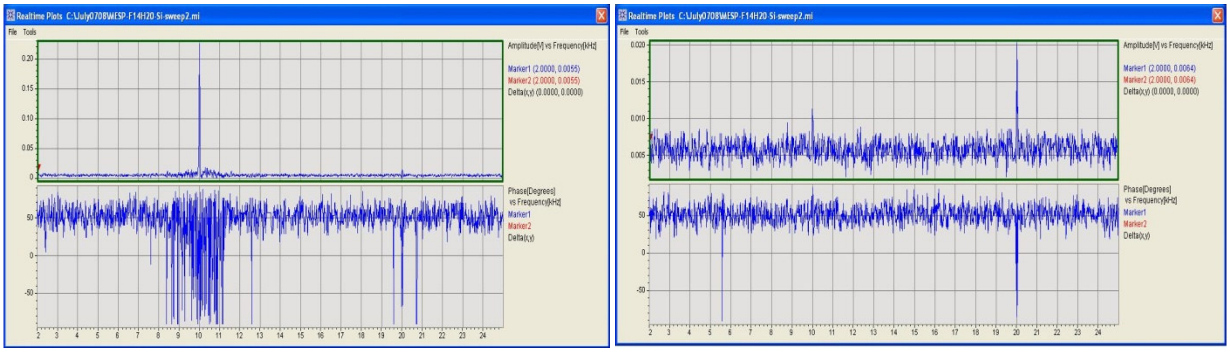


Figure 15. Left – The amplitude and phase spectra in the "off" state of the KFM servo loop. Right – The amplitude and phase spectra in the "on" state of the KFM servo loop. Note that the vertical amplitude scale in the right spectra is ~ 10 times smaller than in the left spectra.

to I_{gain} = 3 and P_{gain} = 3. These gains appeared to be slightly high that leads to a noticeable peak at 10 kHz. After the Gains were reduced to I_{gain} = 1 and P_{gain} = 1 (**Figure 16**, left) the amplitude peak at 10 kHz became less noticeable, **Figure 16** (right).

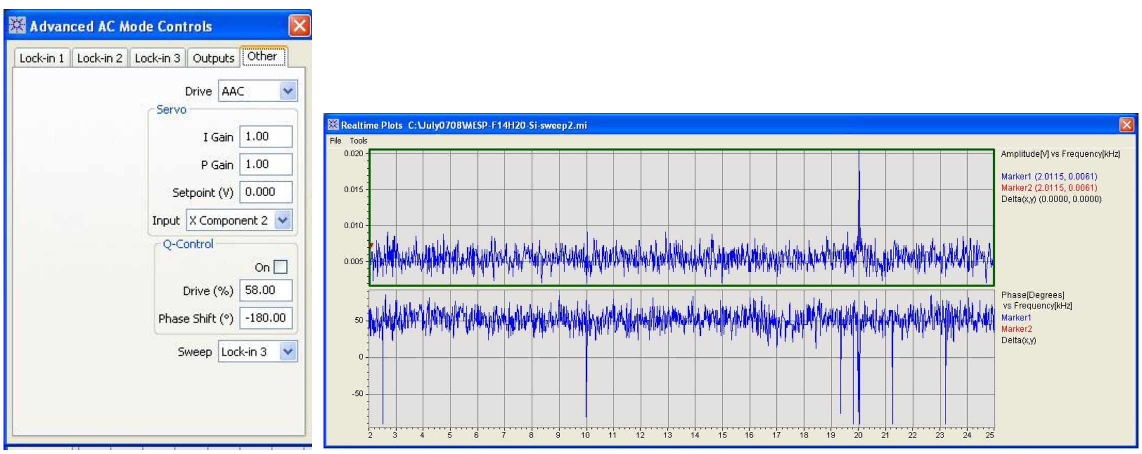


Figure 16. Left – the Other panel with smaller gains. Right – the amplitude and phase spectra with the KFM servo I_{gain} = 1 and P_{gain} = 1.

This was the final adjustment of the AM-AM operation with ω_{elec} = 10 kHz. If one want to use the 2^{nd} flexural mode instead of the low frequency 10 kHz then the adjustments are quite similar. The difference in 2^{nd} LIA was the Drive voltage, which was smaller (2% versus 10%) to get the amplitude value ~ 0.2V, **Figure 17**. In this particular and not general case there was no need for the Phase Offset adjustment because the X2- and Y2 - components were optimal with -171 degrees offset used for the 10 kHz case.

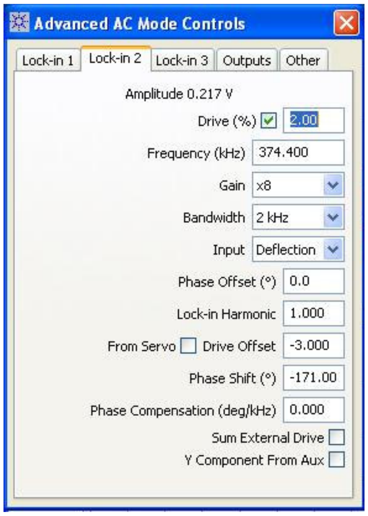


Figure 17. The 2nd LIA parameters for EFM/KFM operation tuning using the 2nd flexural mode for the electrostatic force measurements.

The check of the SP versus Z curve showed that it was not straight (**Figure 18**, left) and the Setpoint of 0.009 (**Figure 18**, center) was required to correct this dependence (**Figure 18,** right).

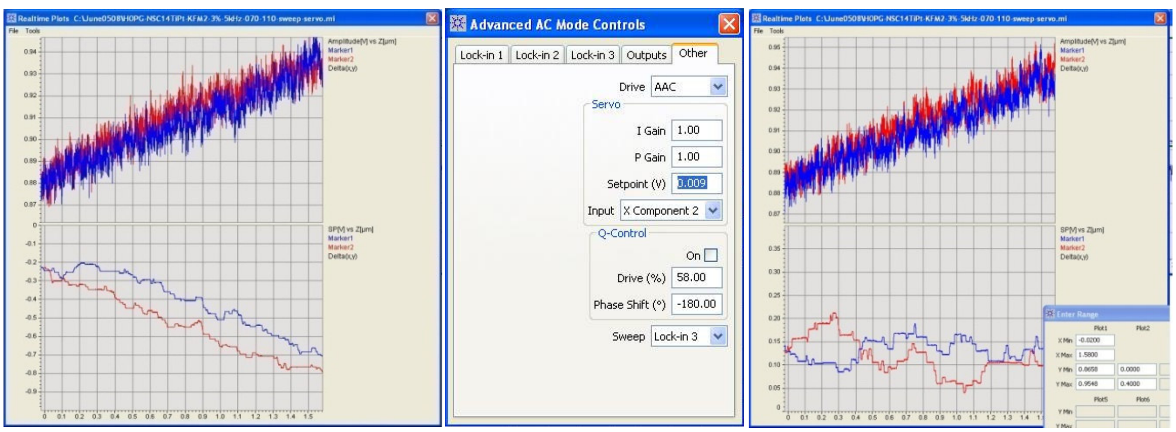


Figure 18. Left and right - the amplitude-versus-Z and SP-versus-Z curves before and after the adjustment of the Setpoint voltage to 0.009V (center).

The final check was related to the amplitude-versus-frequency spectra obtained in the "off" and "on" state of the KFM servo loop. After the appropriate choice of the Start/End frequencies around the 2nd flexural resonance, the spectra that demonstrate the proper choice of the parameters were obtained, **Figure 19** (left, right).

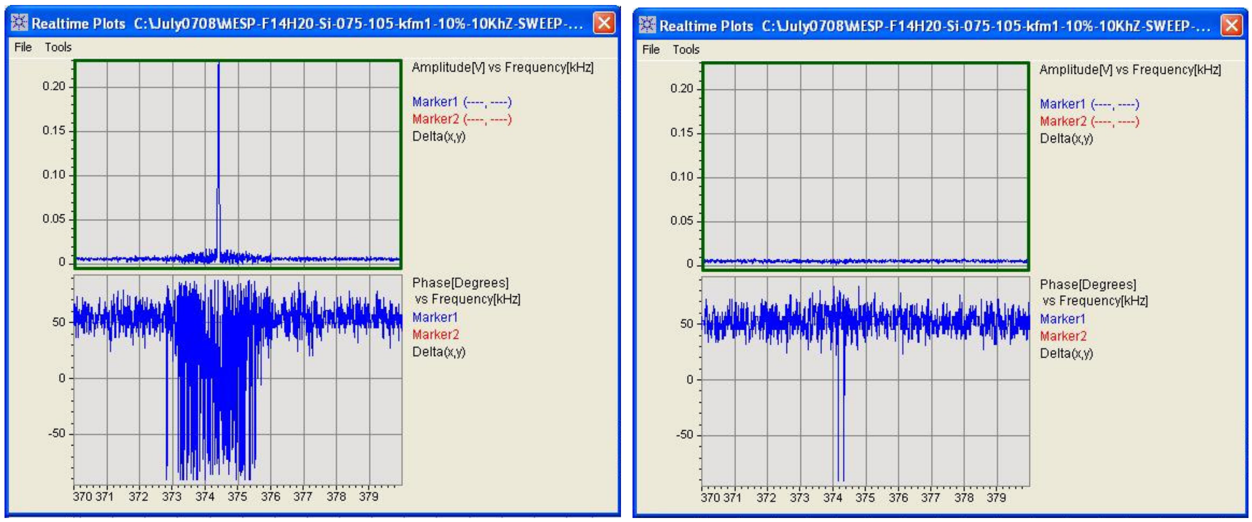


Figure 19. Left and right – the amplitude-versus-frequency and phase-vesus-frequency spectra obtained in the "off' and "on" states of the KFM servo loop, respectively.

Tuning Procedures in AM-FM operation of KFM

The AM-AM operation does not rely on the use of the signal access box, but the latter is essential for the AM-FM. The Aux Inputs of 2nd and 3rd LIA of the box should be connected to BNC2 connector of the MACIII (**Figure 3**). For the AM-FM operation BNC2 should be chosen as Friction in the Outputs panel; that brings the Phase signal to the 2nd LIA. In the 2nd LIA panel one should chose Aux as the Input and the driving frequency set at 5kHz (**Figure 20**, left). The

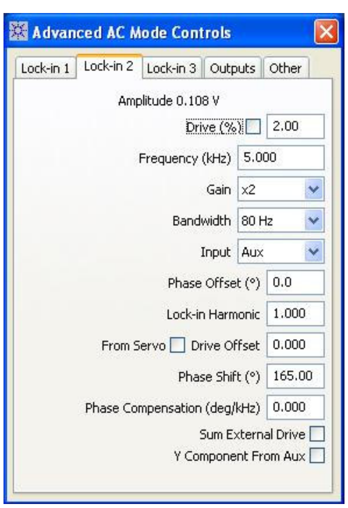
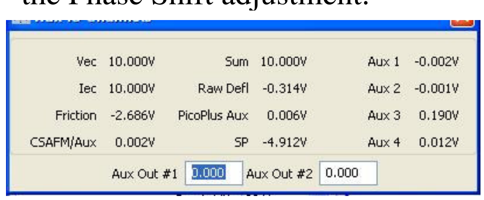
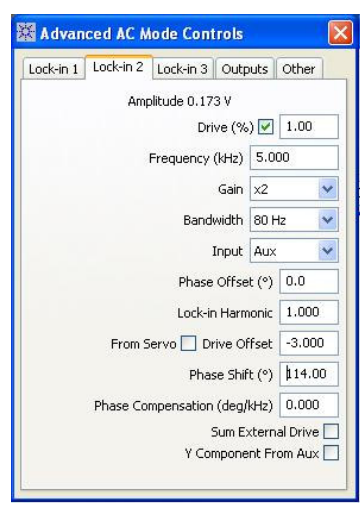


Figure 20. Left and right - the 2nd LIA panel of the Advanced AC Mode Controls before and after the Phase Shift Adjustment. Center – the Aux IO Channels window after the Phase Shift adjustment.





appropriate values of X2- and Y2- components (**Figure 20**, center) were obtained at 114 degrees Phase Shift (**Figure 20**, right). The Drive voltage (1%) is typically smaller than in the AM-AM operation. Before the switching on the KFM servo loop, the Bandwidth was changed to 1 kHz (**Figure 21**, left) that brought SP to a stable level around 0.2V (**Figure 21**, center). The KFM servo gains for the AM-FM operation are typically small (I_{gain} = 0.1-0.3, P_{gain} = 0.1-0.3), **Figure 21** (right).

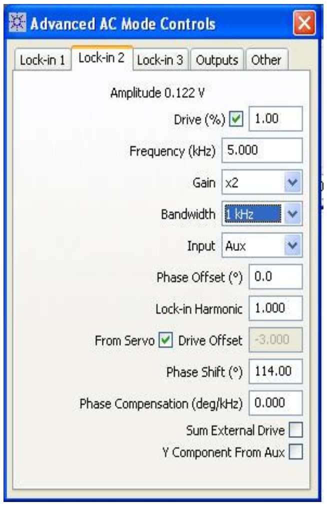
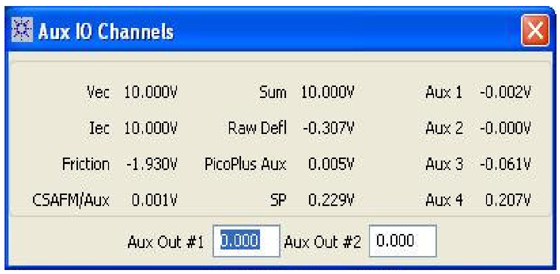
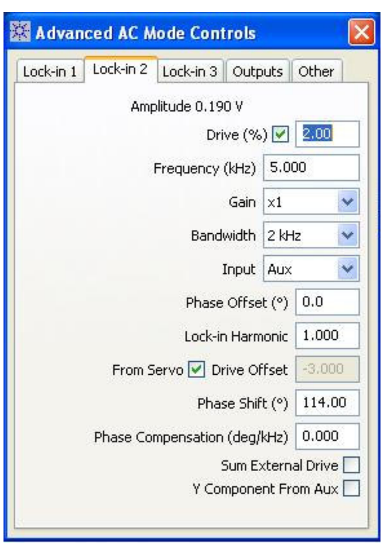


Figure 21. The parameters for the AM-FM operation in KFM: left and right - the 2nd LIA and Other panels of the Advanced AC Mode Controls. Center – the Aux IO Channels window.



The changes of the Drive voltage from 1% to 2% and the changes of the Gains from ×2 to ×1 (**Figure 22**, left) were made to improve the signal-to-noise ratio. The KFM servo loop performance was checked by sweeping 3rd LIA with Aux as the Input (**Figure 22**, right).



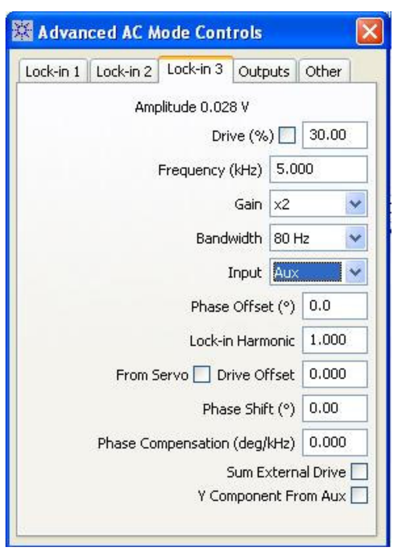


Figure 22. Left – the 2nd LIA panel with the optimized parameters. Right – the 3rd LIA panel prepared for the check of the performance of the KFM servo loop.

The results of the sweep in the "off" and "on" states of the KFM servo, which are shown in **Figure 23** (left, right), demonstrate that the adjustments of the parameters were appropriate.

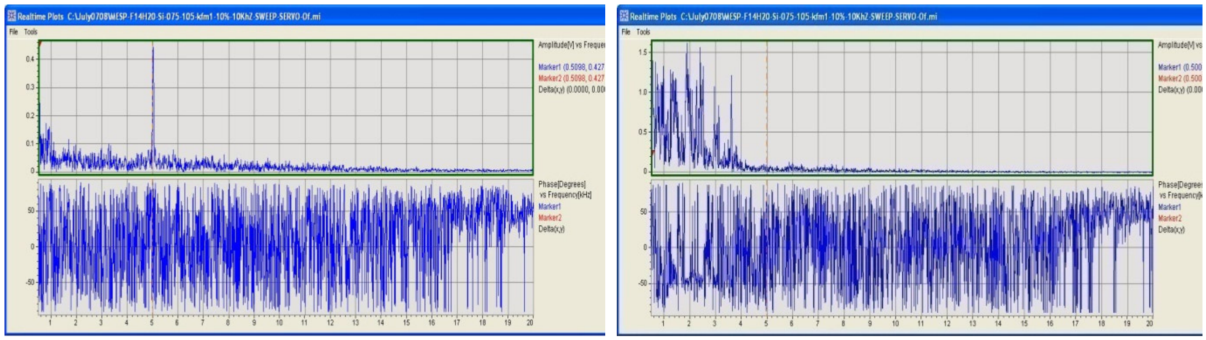


Figure 23. Left and right – the amplitude-versus-frequency and phase-versus-frequency spectra obtained with the KFM servo loop in the "off" and "on" states, respectively.

At this stage the AM-FM operation is properly adjusted for KFM imaging. The SP imaging channel should be added for mapping of the surface potential.

Instead of using the phase of the probe modulated by the electrostatic forces at ω_{elec}, the researcher can use the Y-vector component of the 1st LIA (or in other words, the Y-vector compenent 1) for the KFM servo, especially when the amplitude of the 1st LIA is equal to the X-vector component 1. The latter can be achieved by a proper adjustment of the phase of the 1st LIA. Our Application Note presented the data showing that using the Y- compoenent one can get the surface potential images with higher signal-to-noise ratio compared to the use of the phase. For the practical realization of the AM-FM operation with the Y- vector component 1 employed by the KFM servo loop one should define in the Outputs panel Aux1 as X1, Aux2 as Y1, and set BNC2 to Aux 2 (**Figure 24**, left) and then perform two phase adjustments. The phase of the 1st LIA should be chosen to make Aux 1 as high as possible and Aux2 close to zero, and the phase of the 2nd LIA should be chosen to make Aux3 as high as possible and Aux4 close to zero. This situation is illustarted in **Figure 24** (right). Naturally, the adjustment of the phase of the 2nd LIA will be easy when the Bandwidth will be set to the lowest value – 80 Hz, yet don't forget to switch it back to ~ 1 kHz for imaging of the surface potential. In principle, after the phase adjustments the microscope is ready for KFM measurements. If one wants to check the performance of the KFM servo loop, it can be done by sweeping the Aux signal in the 3rd LIA around 5 kHz similar to what have been done in case of the AM-FM operation with the phase as the KFM servo loop signal. Practically, the Aux of the 3rd LIA of the signal access box (**Figure 3**) should be connected to BNC2 of the MACIII and BNC2 is set to Aux2=Y-component 1.

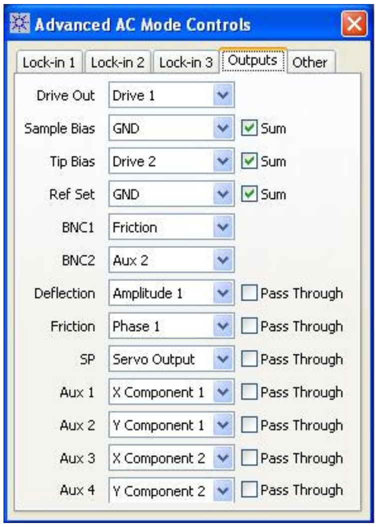


Figure 24. Left – the Outputs panel setting for KFM operating in AM-FM regime with the Y-vector component employed for the KFM servo loop. Right – the Aux 1 to Aux 4 signals after optimization of the phase of the 1st and 2nd LIAs.

There is also a possibility to increase the Y-component 1 signal by decreasing the Q-factor of the probe oscillation at ω_{mech} using the Q-control capability of the 1st LIA. This is the further expansion of KFM in the AM-FM mode, and currently we are checking have valuable is the use of the Y-component with low Q.

Conclusion

In this technical note we introduced 5 different procedures to perfom KFM measurements. In two procedures, named as AM-AM operations, the electrostatic tip-sample forces were measured at low frequency of 10 kHz ($\omega_{elec} < \omega_{mech}$) and at the 2nd flexural mode ($\omega_{elec} > \omega_{mech}$) and the probe amplitude at ω_{elec} was employed for the surface potential measurements. The AM-FM operations were based on the detection of the probe phase or the Y – vector component of the amplitude at mixed $\omega_{mech}+\omega_{elec}$ (ω_{elec} = 5 kHz) frequency. Additionally, the enhancement of the Y- component signal was suggested with the lowering of the Q-factor of the probe. At the moment, we have performed a limited number of the KFM experiments with these modes and the results are summarized in the Application Note. They indicate that as compared to the AM-AM procedures, the AM-FM measurements lead to he surface potential data with higher signal-to-noise ratio and better spatial resolution of 2 nm. We don't like to make the final conclusions regarding validity of different KFM modes and hope that further studies of various materials with these modes will shed a light on this question and demonstrate the power of AFM-based characterization of local electrical properties of diffferent materials.