RHK Technology
Application Note:

Kelvin Probe Force Microscopy with the RHK R9

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Introduction

Kelvin-probe force microscopy (KPFM) is an operation mode based on non-contact atomic force microscopy (nc-AFM). In nc-AFM mode the frequency of an oscillating cantilever is measured via a phase locked loop and kept constant by the topography feedback loop. The frequency shift with respect to the resonance frequency far away from the sample provides a measure of the force gradient as apparent between tip and sample. Simultaneously to topography measurement, KPFM allows the determination of local contact potential differences. In Kelvin probe force microscopy, a sinusoidal modulation plus a dc-bias voltage are applied between tip and sample. Due to the non-linear voltage dependence of electrical forces between tip and sample,

$$F_z^{el} = \frac{1}{2}\frac{\partial C}{\partial z}\left[U_{cpd} - U_{dc} + u_{mod}\sin\left(\omega_{mod}t\right)\right]^2, \quad (1)$$

the detection of force components at the modulation frequency ω_{mod} allows to fully nullify any contact potential difference U_{cpd} between tip and sample, by properly adjusting the dc-bias U_{dc}. The contact potential difference U_{cpd} originates in the work function difference between tip and sample as well as electric potentials due to bound charges, polar domains etc.

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This Application note summarizes and compares different techniques for doing Kelvin probe force microscopy, combined with nc-AFM in an ultra high vacuum environment.

KPFM Techniques

AM Kelvin [3] [4]

Basics

Figure 1 shows the configuration of AM KPFM measurements. A Phase Locked Loop (PLL in Figure 1) traces the resonance of the cantilever. This Topography PI controller (Topo PI) will move the Z piezo so that the df signal is kept constant. The idea of AM KPFM is to apply an AC component to the tip-sample bias voltage and to directly measure the amplitude of that frequency in the cantilever oscillation using a Lock-in amplifier (LIA). The LIA is synchronized to the Bias Oscillation frequency f_{Bias}. This frequency is usually chosen to be different from the cantilever fundamental resonance that is used for topography measurements. Using the resonance of a higher harmonic can improve the signal-to-noise ratio (SNR) at the cost of scanning speed. It is also possible to measure at the fundamental cantilever resonance in a multipass mode.

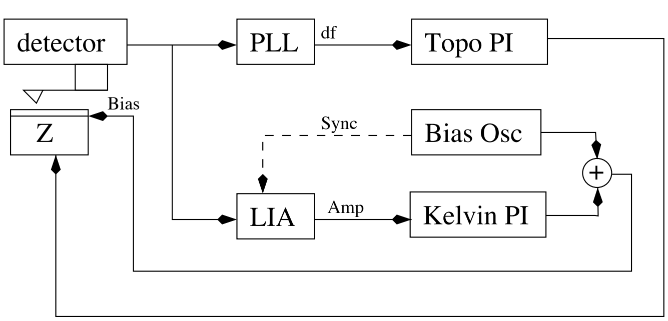

Figure 1 AM KPFM configuration

Strengths

When a cantilever resonance is used the strength of AM Kelvin method is a high sensitivity. Only a very small Bias modulation amplitude is needed for a signal with good SNR. This is an advantage for measuring semiconductors where the small amplitude avoids undesired band bending.

Weaknesses

AM Kelvin measurements have the fundamental problem that the capacitance of the whole tip pyramid and even the cantilever cause an electrostatic signal that is superposed to the high resolution components caused by the tip apex [5]. Nevertheless atomic scale measurements have been published.

When the tip interacts with the sample then a higher harmonic might move it's resonance, even though the fundamental frequency is kept constant by a Z loop. This will couple topographic signal into the Kelvin signal. The problem will get worse as the resonance gets narrower.

Another problem with amplitude detection is that with narrower peaks the detection time constant increases which requires slow scanning.

Furthermore the measurement is restricted to just the frequency of the resonance. Often, on higher modes, this frequency exceeds the bandwidth of standard preamplifiers.

FM Kelvin principle [2]

The electrostatic interaction between tip and sample has a frequency of f_{Bias}. Our cantilever resonance frequency will thus move up and down at a frequency of f_{Bias}. This is called a frequency modulation. An FFT plot of a frequency modulated signal will show the fundamental frequency (f_0 in our case) , surrounded by two small peaks at a frequency of f_0 - f_{Bias} and $f_0 + f_{Bias}$ as shown in Figure 2. There are two ways to measure this frequency modulation:

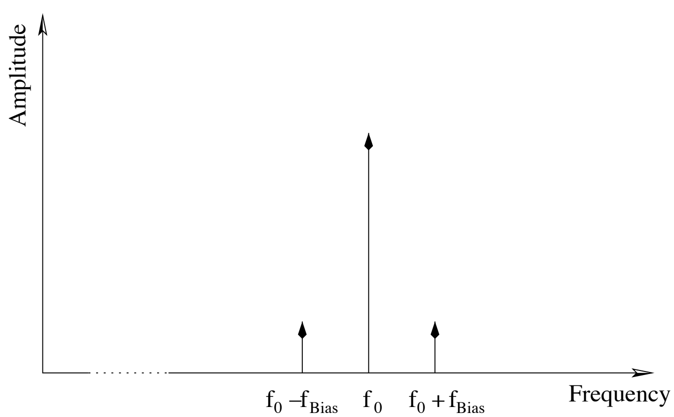

Figure 2 FM Kelvin Sidebands

1. Measuring the amplitude of oscillation in the df channel. This is called Sequential FM Kelvin and described in the next section.

2. Measuring the amplitude of the signal at $f_0 + f_{Bias}$. This is called Sideband FM Kelvin and described on page 4.

Sequential FM Kelvin

Basics

This is the classic approach to FM Kelvin. The tip-sample Bias voltage is modulated at a frequency f_{Bias} in the range of 0—5kHz. The PLL needs to be tuned fast enough ($f_{BWPLL} > f_{Bias}$) so that the Bias modulation shows up in the df signal. A Lockin Amplifier (LIA) looks at the df output of the PLL. The LIA is synchronized to the Bias modulation frequency f_{Bias}. The Kelvin PI controller is fed by the X output of the LIA.

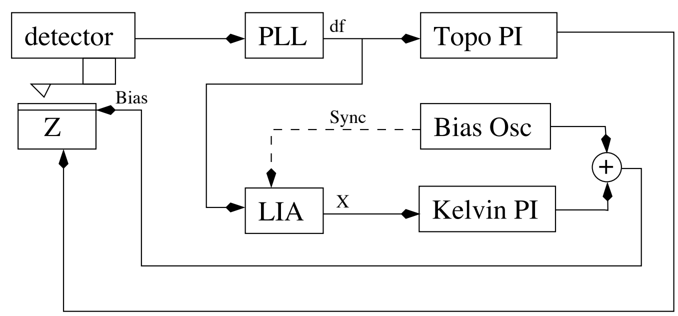

Figure 3 Sequential FM KPFM configuration

Strengths

The FM Kelvin method detects the derivative of the electrostatic interaction force. The detected interaction range therefore is much more localized to the tip apex which is more suited for high lateral resolution in the Kelvin images. [5]

In Sequential FM Kelvin the error signal on the Kelvin PI controller does not depend on the Bias modulation amplitude. The signal to noise ratio of the Kelvin measurement, however, does.

Some people find this method easier to understand and to debug, compared to the Sideband detection technique.

Weaknesses

The Sequential FM Kelvin method requires the PLL to be fast enough to follow the Bias modulation frequency. This causes several problems:

- Widening the PLL bandwidth will increase the noise in the df channel and in the Topography signal. The noise can only be lowered by an additional filter in post-processing.
- This additional noise limits usability for simultaneous Magnetic Force measurements (MFM) and KPFM
- The Bias modulation signal can show up in the df image. A post-processing filter will be needed to block the Bias oscillation frequency out of the df channel.
- The Topo PI controller needs to be tuned slower than optimal in order to filter out the Bias oscillation frequency. It is not recommended to low-pass filter the df signal before it enters the Topo PI controller as the signal propagation delay of that filter will drastically slow down the Topo PI loop performance.

Running the Sequential FM Kelvin method requires a signal path from the df data channel to the Lockin Input. On many nc-AFM controllers this requires an analog output that gets re-digitized again, leading to a loss in signal quality. The R9.5 controller has a signal route from any signal source to the lock-in inputs which allows us to keep the signal in the digital domain.

FM Sideband Kelvin

Basics

Figure 4 shows the PLL and the LIA both measuring the detector output. Again the Bias is modulated at a frequency f_{Bias} in the range of 0—5 kHz.

If we want our LIA to measure the signal at the frequency $f_0 + f_{Bias}$ then we have the problem that f_0 changes all the time as the tip interaction between tip and sample changes during measurement. The solution is to synchronize the LIA to the PLL and the Bias oscillation and run it at a frequency of $f_{PLL} + f_{Bias}$. This way the sideband peak is directly detected.

In this configuration the PLL needs to be slow enough to not follow the df modulation at f_{Bias}.

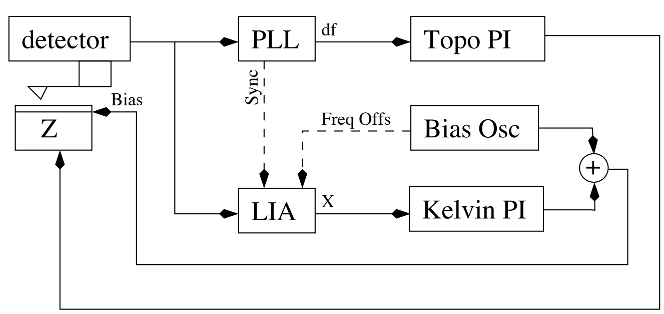

Figure 4 Sideband FM KPFM configuration

Strengths

As this is an FM Kelvin technique that measures the derivative of the electrostatic interaction, we only detect the signal from the very front of the tip apex.

The PLL bandwidth is lower than the Bias modulation ($f_{BWPLL} < f_{Bias}$). We therefore do not see spurs of the Bias modulation frequency in the df signal. The Bias can be modulated at a higher frequency which results in faster scanning speeds.

Weaknesses

The amplitude of the sideband signal depends on the Bias oscillation amplitude. For a good Signal-to-Noise ratio substantially higher Bias modulation amplitudes are necessary.

Comparison Measurements

The Test Sample

The test sample is a thin film of lead zirconate titanate, $Pb(Zr_{1-x}Ti_x)O_3$ (PZT), a widely used ferroelectric perovskite type material known for a high remanent polarization and a large piezoelectric coefficient d_{33}. In the lead titanate rich composition (0.48 < x) it is found with a tetragonal lattice below the curie temperature (350°C - 490°C) as also shown in Figure 5, which allows for six different polarization orientations. In the ferroelectric phases these unit cell dipoles align and build polarization domains. Domains with the polarization directions perpendicular to the surface are called c-domains, while domains with polarization parallel to the surface are called a_x and a_y. The typical configurations for tetragonal PZT thin films are the second-order polydomain and the cellular polydomain patterns shown in Figures 5d-e. Test experiments were performed with epitaxially grown $Pb(Zr_{0.2}Ti_{0.8})O_3$ thin films on 1at%-Nb doped STO obtained from Phasis Sàrl (www.phasis.ch) with a film thickness of 100 nm. The niobium doping induces conductivity of the STO substrate. The test sample shows the cellular polydomain pattern.

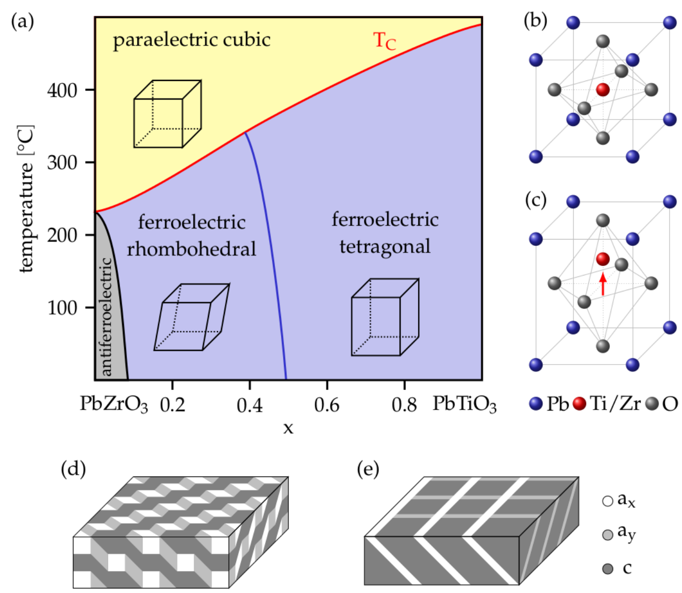

Figure 5 (a) Simplified phase diagram of $Pb(Zr_{1-x}Ti_x)O_3$ after [1]. Below the composition dependent Curie-temperature T_C (red line) the material becomes ferroelectric or antiferroelectric for very low $PbTiO_3$ concentration. At the morphotropic phase boundary (blue line) the structure changes between ferroelectric rhombohedral and ferroelectric tetragonal with eight and six possible polarization directions, respectively. (b) Schematic drawing depicting the unit cell of $Pb(Zr_{1-x}Ti_x)O_3$ in the paraelectric cubic and (c) the ferroelectric tetragonal phase. Due to the displacement of the central atom from the symmetry centre of the unit cell an electric dipole builds up. (d-e) In the ferroelectric phases the unit cell dipoles align to their neighbors and polarization domains build up. The typical configurations for tetragonal PZT thin films are (d) the second-order polydomain and (e) the cellular polydomain patterns.

AM Kelvin measurement

In the original plan of this document this section was supposed to show test measurements using AM Kelvin. We tried to use the 1st harmonic (6.2 x fundamental) of the cantilever resonance for the Bias modulation.

The frequency of that was around 420 kHz.

We had to use the original microscope manufacturer's interferometer preamplifier which did not have enough bandwidth to detect that signal.
An alternative is to use the cantilever fundamental frequency in a multipass KPFM scheme. This is possible in the R9, it requires some change on our IHDL experiment description.

The main problem with this method is that AM detection on a cantilever with a Q-factor of 300000 will be very very slow. This cantilever has a transient decay time constant of more than 4 seconds.

Comparison of Sequential FM Kelvin and Sideband FM Kelvin measurement
As apparent from Figs. 6 and 7 both methods can be used to measure the contact potential differences caused by the polarization domains. Yet, the image obtained with the sideband FM Kelvin method appears less stripy, i.e. there is less low frequent noise in the measurement as compared to the sequential method. Also, due to less stability in the topography loop caused by the low frequent bias modulation, the scan speed had to be set lower than in the sideband FM Kelvin method. The better SNR in the sideband method allows to observe smaller features in the image yielding a virtually higher lateral resolution.

Figures 8 and 9 show cross section lines, taken from the Kelvin measurements. Also here it becomes visible that the Sideband Kelvin measurement has a higher signal-to-noise ratio.

Why We Used Sideband Detection
Which KPFM mode to use is a question of personal favour. Direct sideband detection FM-KPFM is rather simple to use, allows for relatively fast scanning, and is quite stable. Additionally, as any FM-KPFM mode, it provides high lateral resolution, and especially interesting in material science it can be performed simultaneously to magnetic force microscopy.

Why we use the RHK R9

- The R9 is a fully integrated one-box solution with fully synced PLLs, lockins, and built in interferometer feedback detection, which replaced our multi-box solution. The effect of this simplification is shown in Figure 10.
- The R9 offers high flexibility.
- The R9 additional lockins could be used to simultaneously measure 2*f_{Bias} and 3*f_{Bias} for dC/dZ and dC/dV signals.
- R9 enables one-click switching between the different KPFM modes, no reconfiguring external cables.
- R9's LockGuard for the PLL provides a fast 5µs response to protect the AFM probe if the PLL becomes unlocked.
- The R9 allows to measure many channels of data simultaneously.
- The R9 has very low intrinsic noise.

Kelvin Probe Force Microscopy with the RHK R9

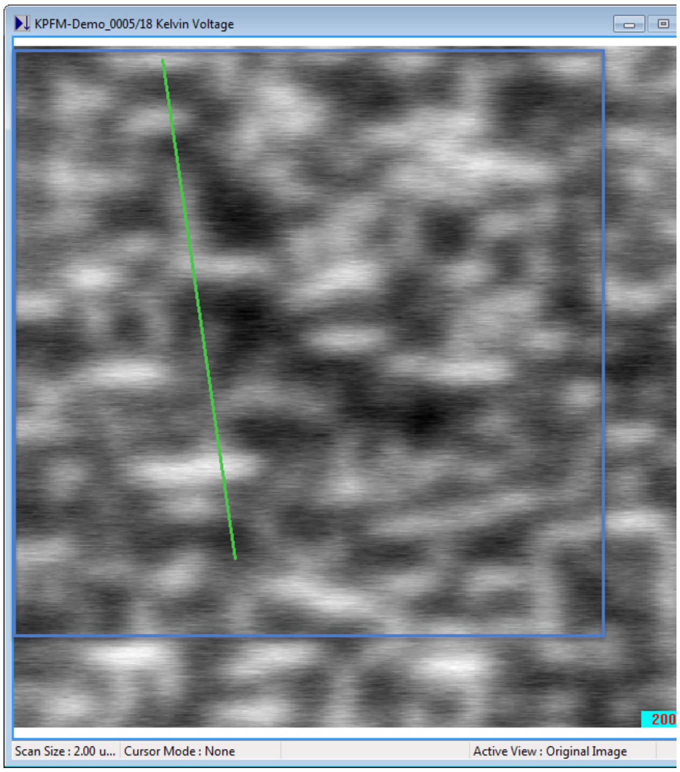

Figure 6 Sequential FM Kelvin result. The blue box marks the common scan area, the green line marks the position of the line scan in Fig. 8

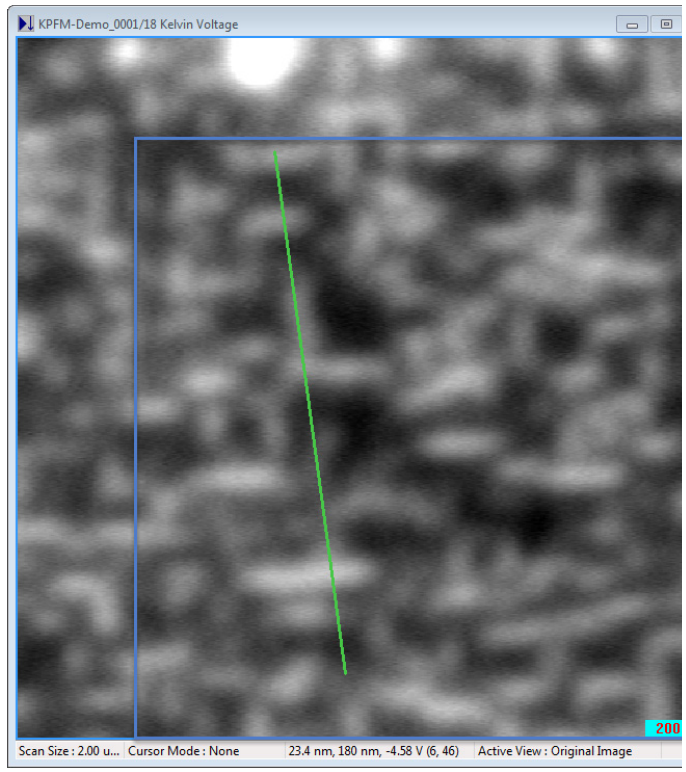

Figure 7 Sideband FM Kelvin result. The blue box marks the common scan area, the green line marks the position of the line scan in Fig. 9

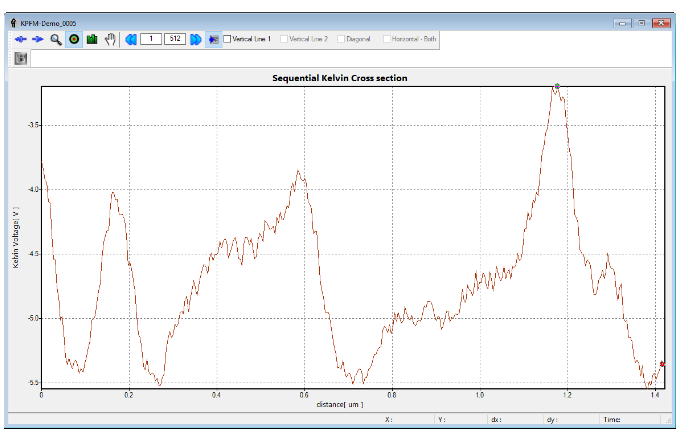

Figure 8 Cross section of our Sequential FM Kelvin measurement, taken along the green line in Fig. 6

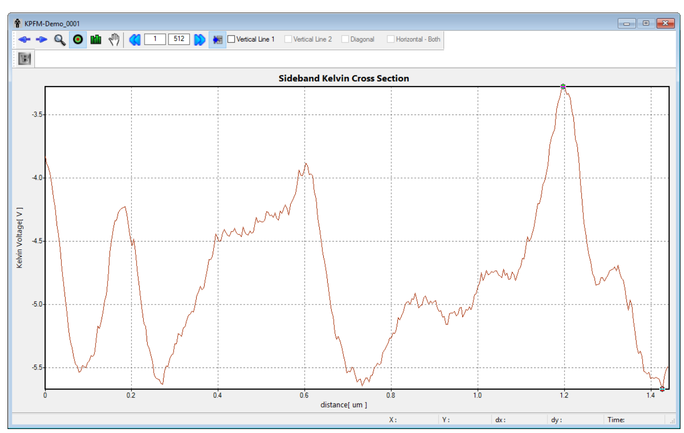

Figure 9 Cross section of our Sideband FM Kelvin measurement, taken along the green line in Fig 7

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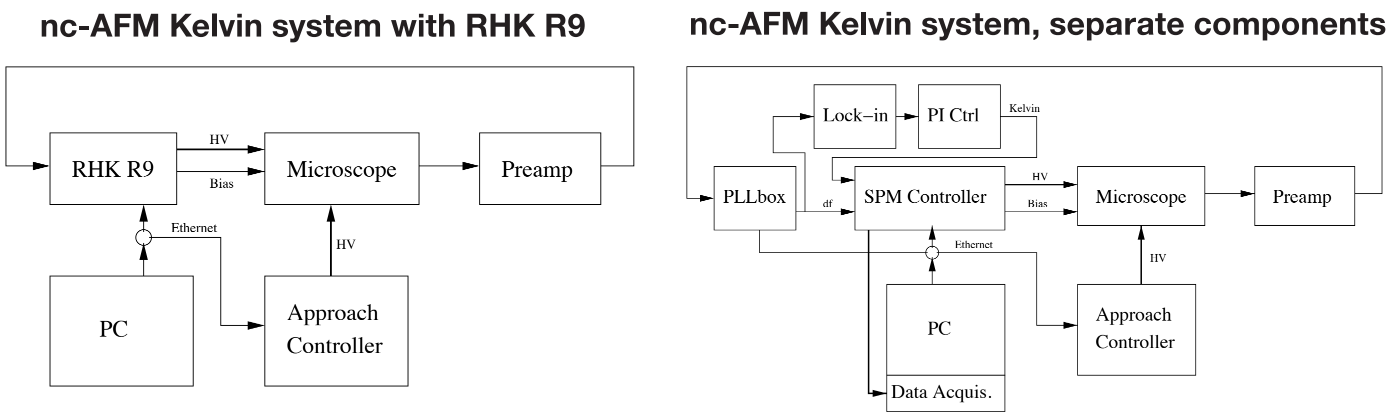

Figure 10 Using the RHK R9 (left) greatly simplified the system hardware, compared to our previous system (right)

References

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