

## Kelvin Probe Force Microscopy

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This tech note was written for software versions dating from Jan 2007. It is possible that changes have been made since that time. This technique is often called scanning surface potential microscopy (SSPM), surface potential AFM (SP-AFM) or surface potential imaging.

### Principles of Kelvin probe force microscopy (KPFM)

The goal of this technique is to determine the difference between the potential of the probe and that of the sample. The data collected by this sample will represent a combination of three contributing factors: the work function difference, trapped charge, and any permanent or applied voltage between the tip and the sample. For this reason the technique is generally considered a pseudo-quantitative technique, in that it gives an accurate measurement of the potential difference, but that number likely consists of more than one physical quantity.

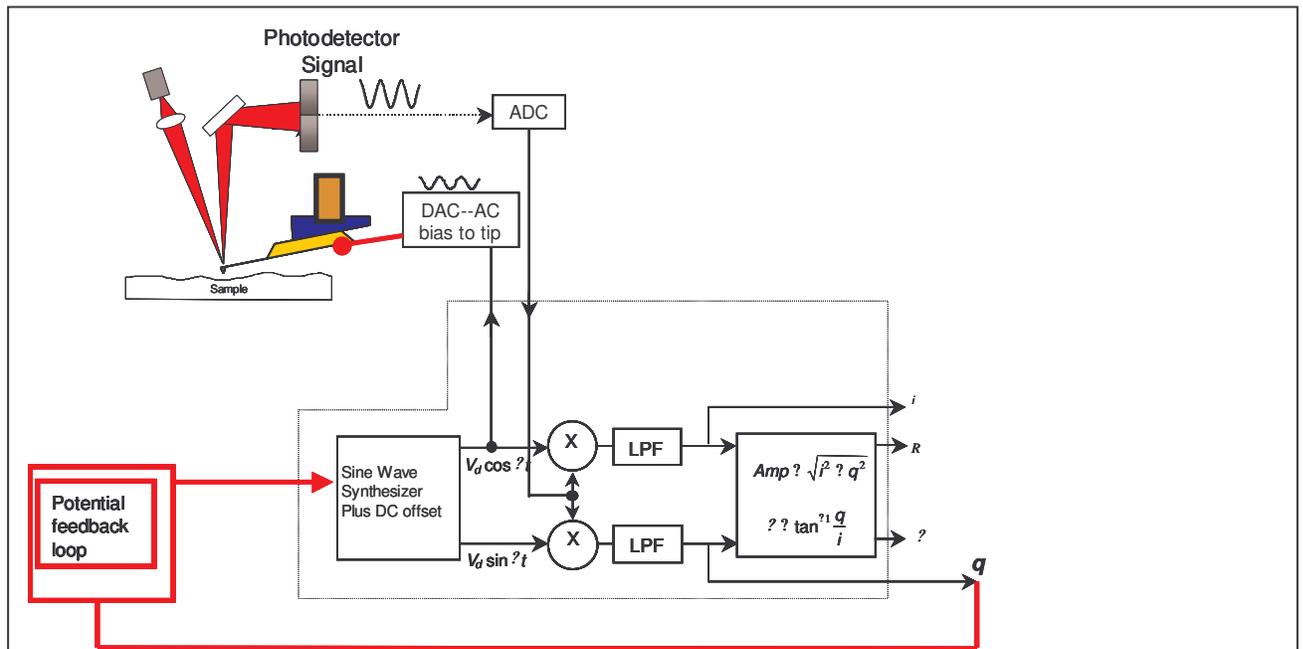


Figure 1. Schematic of the instrument setup for SKPM. Note that the cantilever is driven electrically, and a Voltage is added to the drive. This voltage is controlled as the output of a feedback loop that attempts to make the potential of the surface of the sample equal to the potential of the tip. The voltage needed is taken as the relevant data, and represents the potential difference between the tip and the sample.

The technique relies on an AC bias applied to the tip to produce an electric force on the cantilever that is proportional to the potential difference between the tip and the sample. No mechanically induced drive

(such as with a drive piezo in standard AC mode imaging or in Electric Force Microscopy). The only oscillations that the probe will have will be induced by an applied AC bias. Fig. 1 shows a schematic diagram of the setup for the technique.

An AC bias applied between the tip and the sample produces an electrostatic force between the two. If they are modeled as a parallel plate capacitor, then the force between the two plates is proportional to the square of the applied voltage:

$$(1) \quad F = \frac{1}{2} \frac{\partial C}{\partial z} V^2$$

The total potential difference between the probe and the sample is the sum of the applied AC bias ( $V_{ac}$ ), the potential difference we are trying to measure ( $V_{sp}$ ), and any DC voltage we wish to apply ( $V_{DC}$ ).

$$(2) \quad V = V_{sp} + V_{DC} + V_{ac} \sin \omega t$$

If we substitute this into (1) and do some rearranging, we get:

(3)

$$F = \frac{1}{2} \frac{\partial C}{\partial z} \left( \left[ (V_{DC} - V_{sp})^2 + \frac{1}{2} V_{ac}^2 \right] + 2[(V_{DC} - V_{sp}) V_{ac} \sin(\omega t)] - \left[ \frac{1}{2} V_{ac}^2 \cos(2\omega t) \right] \right)$$

Note that we have three force terms here summed together. The first force is static, meaning it has no frequency dependence. The second occurs at the AC voltage drive frequency,  $\omega$ . The third force oscillates at twice the drive frequency. The most important term here as far as surface potential is concerned is the second, since this depends not on the square of the voltage, but rather on the potential difference between the tip and the sample, multiplied by the magnitude of the applied AC voltage. This means that if there is a potential difference between the tip and the sample, then when an AC voltage is applied, there will be a oscillatory force at the frequency of the drive and proportional of the magnitude of the applied voltage, and also proportional to the potential difference.

Further, if we make  $V_{DC} = V_{sp}$ , with a potential feedback loop between the tip and the sample, then the oscillations at  $\omega$  will be nulled. This is exactly how the technique is done.

### Implementation on the MFP-3D

The probe used will generally be conductive or heavily doped semiconductors. Insulating probes will not work on the MFP-3D, as the bias is applied to the tip. To implement this technique on the MFP-3D, we use a two pass technique. The first pass is used to determine the topography of the surface, and is done exactly like a standard AC mode scan line. The second is used to find the surface potential, and is has the tip raised above the surface. To do this, a technique called nap mode is implemented. During a

nap scan, the tip is raised over the surface on a point by point basis while the potential feedback loop is implemented. Fig. 2 shows a cartoon of nap mode.

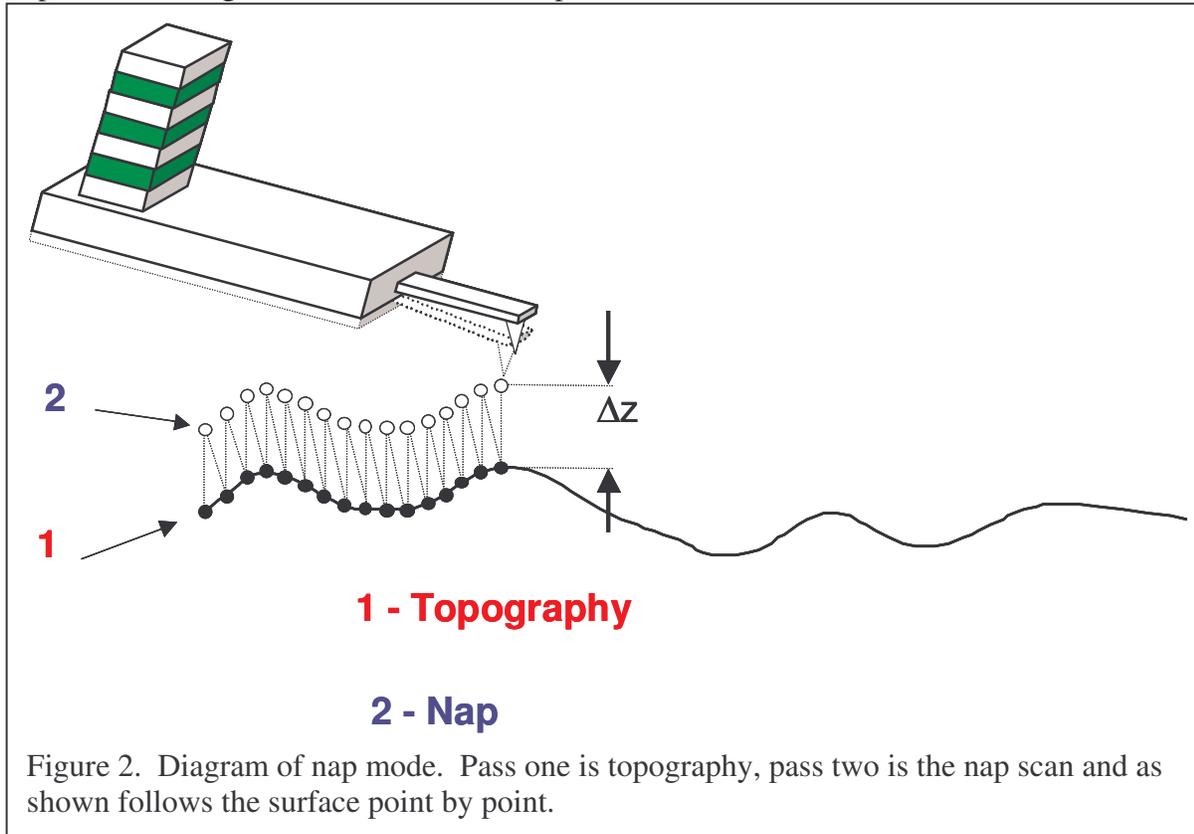
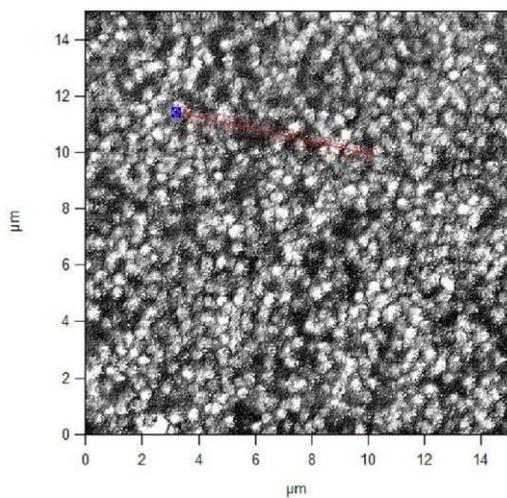
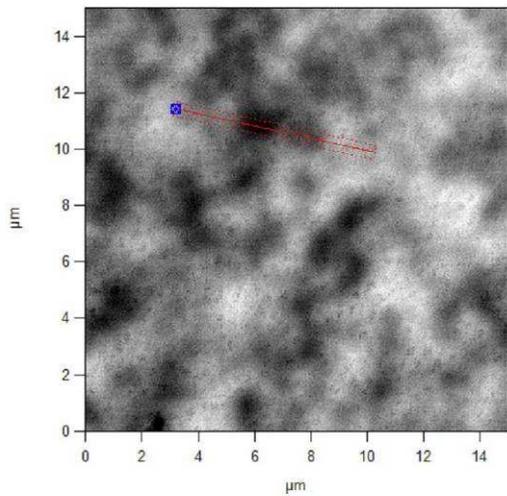


Figure 2. Diagram of nap mode. Pass one is topography, pass two is the nap scan and as shown follows the surface point by point.

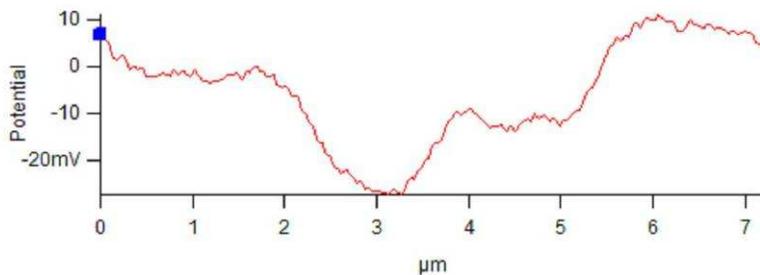
The resulting data from this technique will have a data channel for height and another for surface potential. An example of such data taken on microgels is shown in Fig 3. The three discrete layers of surface potential are likely due to incomplete coverage of the three different microgels. Note that in the section the actual potential differences are visible. In this case one is roughly at the same potential as the tip, one is about 10mV positive, and the other two levels are at -10mV and -30mV. It is important to be sure that data is collected with no processing in the save, since offsets in Surface potential data are relevant, unlike in some other techniques, where relative data is all that is needed.



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 ScanSize: 15.00  $\mu\text{m}$   
 ScanRate: 0.50 Hz  
 ScanPoints: 512  
 ScanLines: 512  
 ImagingMode: 1  
 Date: Wednesday, Jan 03, 2007  
 Time: 3:45:19 PM  
 ImageNote:



File: Courtney0002  
 DataType: NapPotentialRetraceModC  
 ScanSize: 15.00  $\mu\text{m}$   
 ScanRate: 0.50 Hz  
 ScanPoints: 512  
 ScanLines: 512  
 ImagingMode: 1  
 Date: Wednesday, Jan 03, 2007  
 Time: 3:45:19 PM  
 ImageNote:



SectionGraph

### Sample Courtesy Andrew Lyon, Ga Tech

Figure 3. Height (top) and Surface Potential (bottom) image of a three-layer microgel sample. The variation in surface potential is likely attributable to incomplete coverage of the three different microgel layers. The section shows three discrete potential levels on the sample. Sample and images courtesy of C. Sorrell and A. Lyon, Ga Tech.

## Step by step implementation of surface potential imaging on the MFP-3D

Note that this instruction set assumes the user is familiar with standard AC mode imaging, and that the user is opening a new MFP-3D template. This description matches software from Jan 2007. Earlier or later software may have differences.

1. Load a conductive AC mode probe into the AFM head, and set up the microscope for standard AC mode imaging.
2. Load the sample. Insulating and semiconducting samples need not be grounded, but metal samples should be grounded to the scanner plate to avoid arbitrary offsets in the potential that might mask the work function between the tip and the sample.
3. Do a standard tune on the cantilever to find the resonance frequency. Set the target amplitude to 1.0 V and set the target percent to -5%.
4. Open the electrical tune panel by clicking MFP Controls→Electric Tune Panel menu. This panel allows the user to find the resonance frequency of the probe when it is driven electrically, and more importantly, to set the phase properly for an electrically driven probe. This phase will be considerably different than that of the same probe driven mechanically with a piezo.
5. Engage on the surface in AC mode as if doing standard AC mode AFM.
6. At the top of the Electric Tune panel set the height above the surface to 300nm. Set the trigger to AmpVolts, and the trigger point to 800mV. Then click the button marked “single force”. When the force curve is done the tip will be hovering just above the surface.
7. Click on the button labeled “→” to copy the drive frequency over to the electric tune frequency.
8. Now click the button marked ‘electrical tune’. This will sweep an AC bias to the probe and show the response in the tune graph. The resonance frequency will be very close to that of the electric tune. Be sure that the tip voltage field under the Electric column reads 3.0V and that you have at least 500mV in the drive amplitude under the Electric column. Now click the ‘Center Phase’ button. The software will set the phase properly so that the feedback loop can function properly. These buttons are shown in Fig. 4.

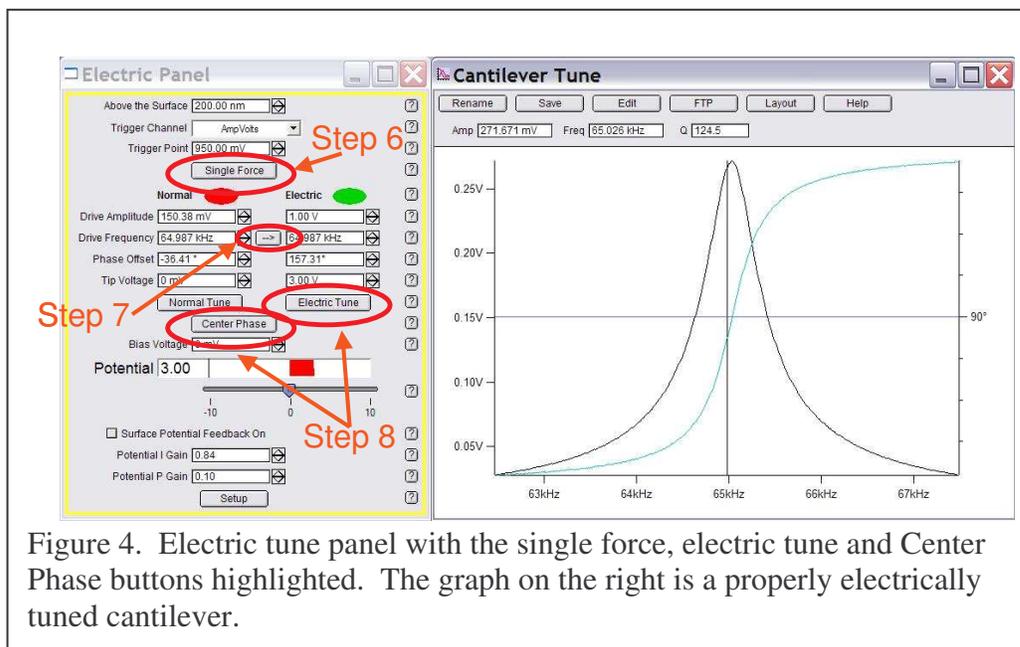


Figure 4. Electric tune panel with the single force, electric tune and Center Phase buttons highlighted. The graph on the right is a properly electrically tuned cantilever.

9. Open the nap channel panel from the MFP3D controls menu item (Ctrl+8). The nap channel panel controls how the nap panels are displayed. Channels to be viewed must be selected in the main scan in the master channel panel.
10. Select Potential as a data channel in the master channel panel. Selecting this automatically turns the potential feedback loop whenever the scanner is imaging. The specifics of the nap channels should be selected in the nap channel panel. This includes setting live flatten to none, and deciding if both the trace and retrace should be selected for viewing or saving.
11. Open the nap panel in the MFP3D controls menu item (Ctrl+9). The nap panel controls the real time operation of the system in nap mode. For nap mode, the system will make two passes back and forth (for a total of 4 individual scans across a single line) for every scan line. The first two passes gather topography, the next two, in the case of surface potential, raise the tip off of the surface on a point by point basis and collect the surface potential data. The 'surface' Column shows the settings for the main, or on the surface scan. The 'parm swap' Column shows the settings that will be used during the surface potential scan. Checking the box between the two columns (under the 'use' column) that correspond to a row will cause the system to override the 'surface' settings with whatever is entered into the 'Parm Swap' column during the nap pass.
12. Select Drive amplitude, Drive Frequency, and Phase Offset under the use column. See Fig. 5.

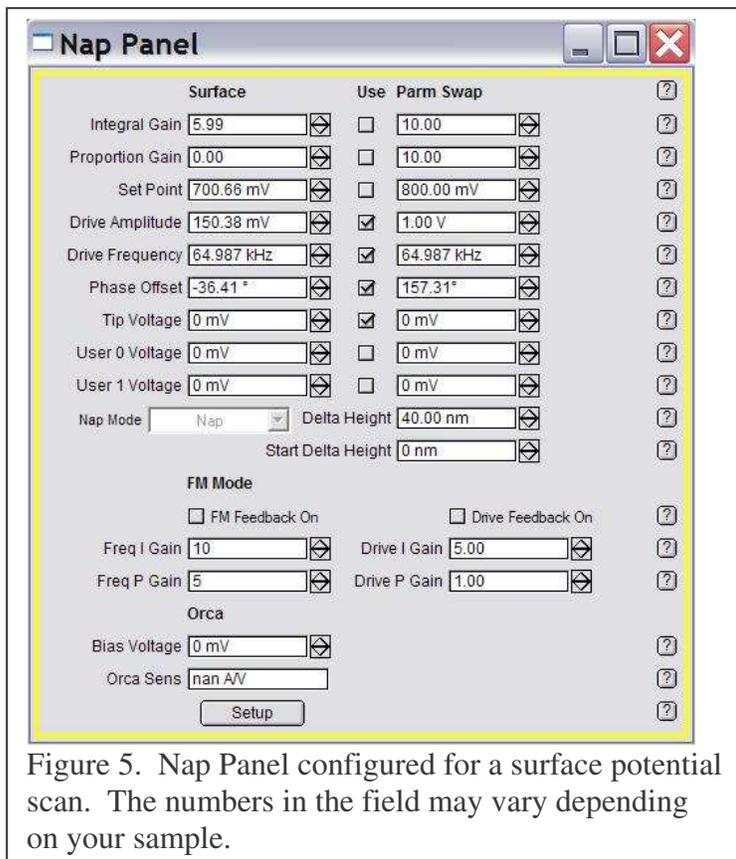


Figure 5. Nap Panel configured for a surface potential scan. The numbers in the field may vary depending on your sample.

13. Set the nap mode to 'nap'.
14. Set the delta height to 40nm. This will be lowered later if you want to try to get better lateral resolution. Because the reference height is based on the zero point of the cantilever oscillations,

and because the potential feedback loop will keep the amplitude at zero during the potential scan, it is actually possible to enter a value lower than 0 nm as a delta height. If too low a value is entered, the tip will strike the surface, and significant, obvious scan line errors will occur.

15. Set the Start Delta height to 40nm. If you are imaging a sticky sample, this might need to be raised to get the tip off of the surface for the nap scan.
16. Set your scan speed and scan size to reasonable values for the sample you are imaging.
17. Start the scan.
18. Look at the surface potential data. If the trace and retrace do not match, but seem to be tracking roughly, raise the potential I gain and potential P gain on the electrical tune panel. If the potential data is unstable or very chaotic, try lowering the gains quite a bit. A good starting value is around 1 for integral and 0.5 for proportional.
19. To improve lateral resolution slightly, try lowering the delta height. It might even be possible for this number to be negative, since the zero point is based on AC mode imaging, where the cantilever is oscillating.
20. Be sure to check the 'save images' so you save any good data you get.