

## Application

Electric Force Microscopy is relatively simple in concept and operation, but often requires some considerable thought in interpretation. The technique has the tip oscillating flexurally at its resonance frequency above the surface while a direct current (DC) bias is applied between the tip and the sample. The electric field between the tip and the sample creates a force gradient between the two that causes a shift in the resonance frequency of the cantilever. This shift in resonance in turn causes a shift in the phase lag between the drive and response of the cantilever. By monitoring the phase shift, the user can find areas under the tip that perturb the electric field between the tip and the sample.

The technique is most commonly used on samples that are a mix of conductive and non-conductive areas. EFM will tend to show contrast where areas of conductivity vary dramatically.

Conceptually, One can consider that the electric field lines between the tip and the sample take the shortest possible path to the ground plane:

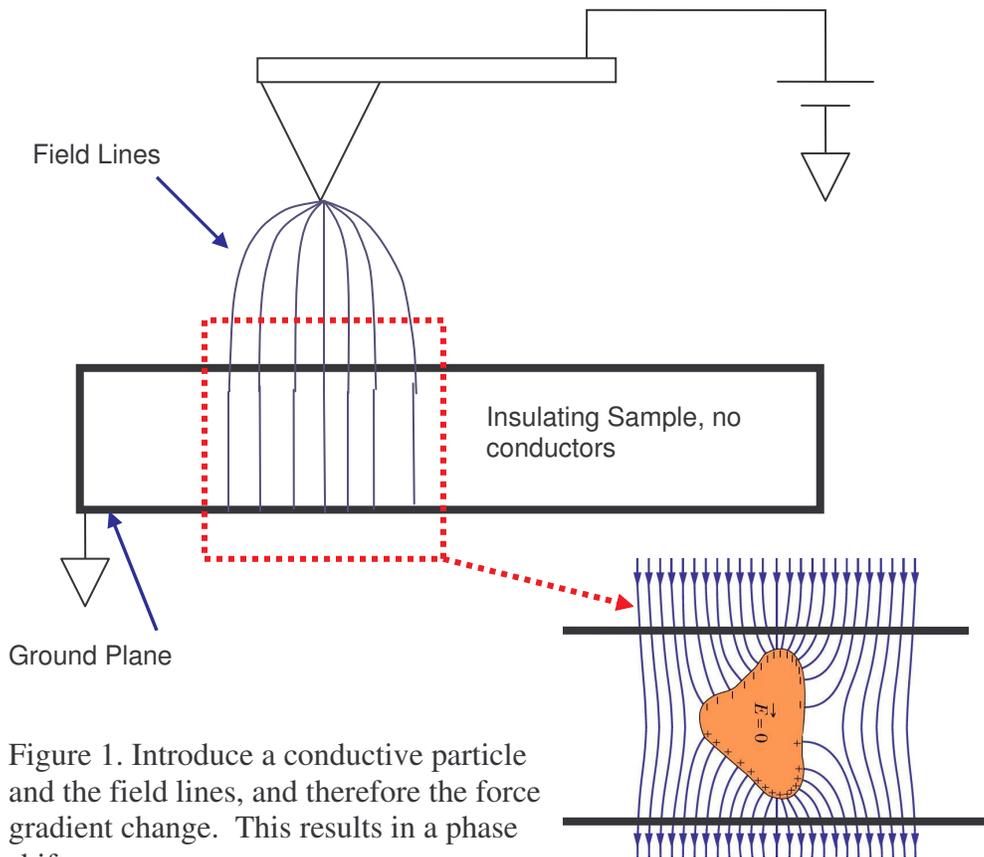


Figure 1. Introduce a conductive particle and the field lines, and therefore the force gradient change. This results in a phase shift.

When a conductor is introduced into the electric field, the lines move perpendicular to the surface of the conductor in order to make the electric field inside the Gaussian surface zero. Any change in the field lines causes a change in the field gradient. The phase lag of the response of the cantilever relative to the drive is sensitive to force gradient changes. Thus, conductors cause a phase shift that is detectable by the lock-in. This is particularly apparent when dealing with conductors in an insulating matrix. For insulators in a conducting matrix, the only contrast can be achieved when the conductors are on the surface, and that phase shift is minimal and often difficult to detect. If the insulating particles are below the surface, then they cannot be detected, as their presence is masked by the conducting surface.

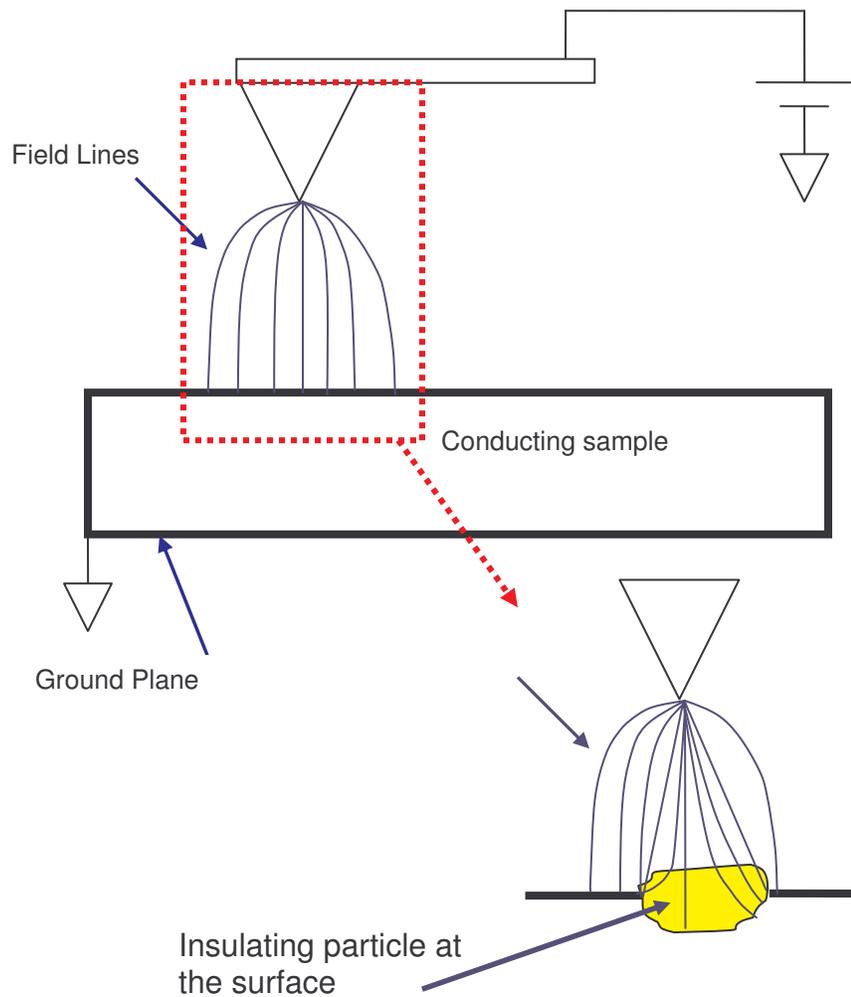


Figure 2. Cartoon of the field lines due to a conductor at the surface of an insulator.

## Theory

EFM relies on electrostatic forces between the tip and the sample to cause a shift in the phase lag of the cantilever that is driven mechanically. The tip-sample interaction is most often treated as a capacitor. To bring the charge element  $dq$  to a positive electrode in a capacitor with potential  $V$ , you need energy:

$$dU = Vdq$$

but for a parallel plate capacitor

$$V = \frac{q}{C}$$

where  $q$  is the charge and  $C$  is the capacitance. So

$$dU = \frac{q}{C} dq$$

Integrating gives the energy:

$$U = \int_0^Q \frac{q}{C} dq = \frac{1}{2} \frac{Q^2}{C} = \frac{1}{2} CV^2$$

Differentiating with respect to distance gives the force on the cantilever due to the electrostatic field (simplifying to the field only in  $z$ , or normal to the sample surface):

$$F = \frac{dU}{dz} = \frac{1}{2} \frac{dC}{dz} V^2$$

So the force depends on the capacitance and the applied voltage. The capacitance depends on the geometry of the capacitor(s), meaning the location, size, conductivity, dielectric constant, etc of the area between the tip and the ground plane. For example, in Fig. 1, before the conductor is introduced into the insulating sample, the capacitor can be considered to be the tip to the ground plane, with two dielectrics between. One the sample, the other the air between the tip and the sample (of course this is much more complicated if you consider the contamination layer). When the conductor is introduced, you now have essentially two capacitors, the tip – ground plane, and the conductor that has been introduced. Areas where there is no conductor will have a different  $dC/dV$  than areas where conductors exist.

It is also possible to get contrast due to the presence of charge, but this is normally very faint compared to the contrast due to the capacitor geometry under the tip. For samples that have varying charge, a much better technique to use is scanning Kelvin probe microscopy (SKPM).

EFM employs two techniques to deliver data. The first is phase detection. In phase detection, the cantilever is oscillated near resonance above the surface. Force gradients between the tip and the sample cause a shift in the phase between the drive and response of the cantilever. The principles of this interaction are derived from the simple harmonic oscillator (SHO) model, where the tip is a simple harmonic oscillator, and tip-sample force gradients act to damp the motion of the tip. The drive of the SHO system is the drive piezo that drives the cantilever substrate.

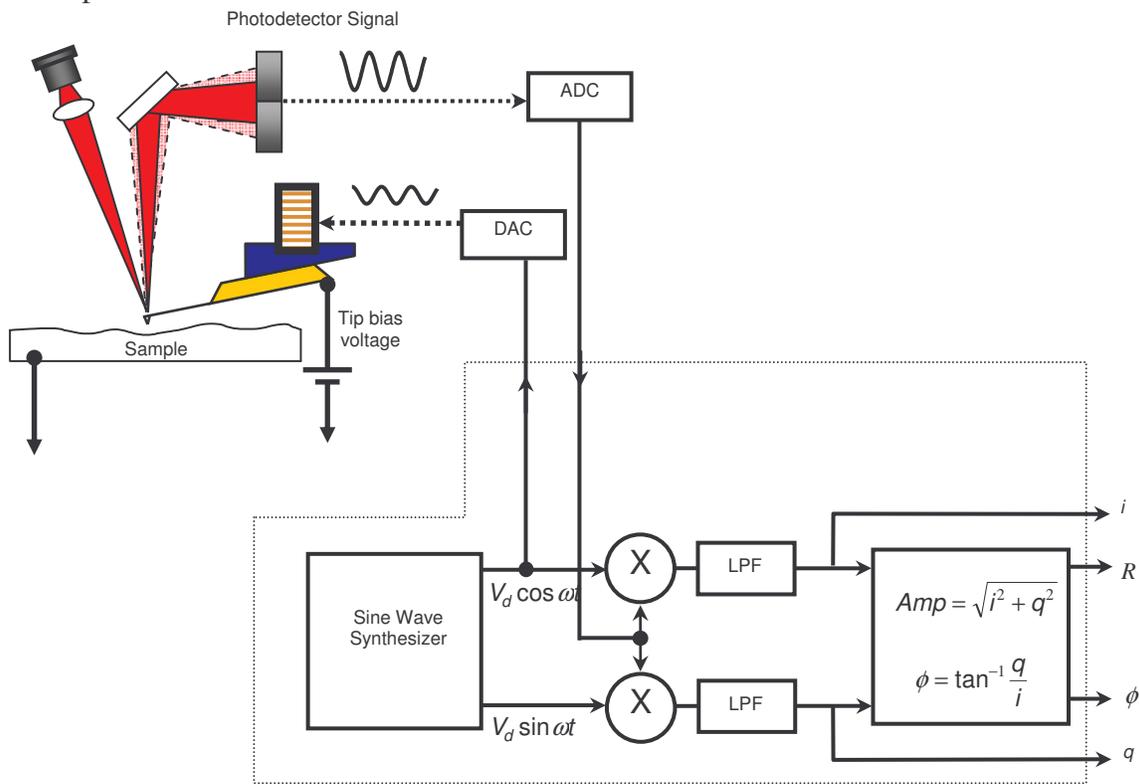


Figure 3. Schematic of the EFM set up. The cantilever is driven while a bias is applied between the tip and the sample. A lock-in amplifier is used to detect phase lag between the tip and sample as a result of an electric field gradient.

The electric field is decoupled from topography by performing two scans over each scan line. In the first, the normal topography is taken. On the second, on a point by point basis, the tip is raised above the surface and a phase measurement taken to see how the electric field lines vary as a function of lateral x-y position. This is done while a bias is applied between the tip and the sample. An illustration of this is shown in Fig 4.

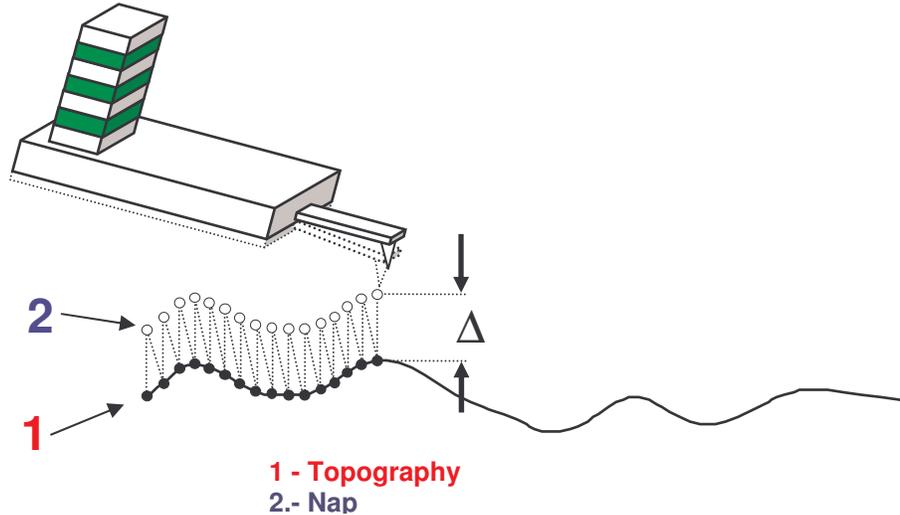


Figure 4. NAP mode on the MFP3D. On the first pass, topography is taken. On the second pass, on a point by point basis, the tip is raised above the surface and a phase measurement taken.

## Nap Mode

Nap mode on the MFP-3D uses two panels in addition to the normal controls for standard AC mode imaging. The first is the nap panel, which contains all the controls needed to run EFM. The second is the nap channel panel, which allows the user to choose channels that will be displayed while running EFM. The nap panel can be accessed by selecting MFP Controls->nap panel.

Nap mode on the MFP-3D is fairly simple in principle. Two passes each of a trace and retrace are made on the sample surface. The first is a normal topographic scan, and in general can be in either contact or AC mode (for EFM AC mode is used). The second trace and retrace set are definable by the user as one of four options in the nap panel. Nap mode is selectable on the nap panel under the drop down selector menu listed as 'nap mode'.

The first is normal, and simply does an ordinary scan. This setting is the equivalent of setting nap mode to disabled.

The second is nap, and causes the system to scan above the surface on a point by point basis. The resulting motion of the probe will be to trace out the topography a fixed distance from the surface on a point by point basis. Tip-sample force feedback is off during this type of nap scan. This technique is generally used for EFM, and normally does the best job of reducing topographic features affecting the EFM scans. This technique is shown in Fig 4.

The third setting is called ‘parm swap’, and will leave the tip and sample in feedback on whatever channel is being used as the primary scan. It will, however, use a different, user definable set of parameters. The parameters that may be changed for the main and nap scan are listed in the upper right corner of the nap panel and are:

Integral gain  
Proportional Gain  
Set Point  
Drive Amplitude  
Drive Frequency  
Tip Voltage

To select one of these, check the box in the column marked ‘use’ to the left of each of the items in the parm swap column.

As an example, consider that a user wants to have two scans taken over the same area of the sample with interleaved scan lines. One scan is to be at in repulsive mode, and the other is to be in attractive mode. The user may select a set point and drive amplitude as the two parm swap parameters. For the first scan the user may choose a frequency that is to the low side of the resonance peak, and an amplitude setpoint that is 50% lower than the free air amplitude. This will tend to drive the cantilever into repulsive mode. For the parm swap scan the user may choose a drive frequency that is slightly higher than the resonance peak, and an amplitude setpoint that is 85 or 90% of the free air amplitude. The user will then select the channels in the main scan that are to be displayed. The user will then open the nap channel panel, and select the channels that are to be displayed for the nap mode trace and retrace. Note that in order to bring up a channel in nap mode it must be selected in the main scan.

The fourth setting in nap mode is ‘snap’. This refers to linear, or ‘stupid’ nap mode. In this case, the tip is raised above the surface, and rastered back and forth during the nap trace and retrace, ignoring topography, except to compensate for the slope of the sample. Generally, either ‘nap’ or ‘snap’ mode will be used for EFM and surface potential, or scanning Kelvin probe (SKPM) imaging, and the more common technique is that using ‘nap’ mode.

The delta height parameter is used for snap and nap mode. During the nap and snap scans, the tip will be raised above the surface for each point by this amount.

The ‘FM feedback On’ box allows the user to have a phase lock loop enabled that will monitor the phase, and adjust the frequency so that the phase stays constant. This technique is used both in MFM and EFM.

## **EFM Operation**

To run EFM, open the nap panel, then open the nap channel panel. Load a cantilever that is conductive, and align the superluminescent diode as normal. The standard cantilever used for EFM is typically an Olympus Electrilever, however, any conductive probe will do provided that the cantilever can be driven at its resonance frequency. The key factor in choosing a probe is that you do not want the probe tip to be much larger or smaller than your surface features. This is because your phase shift due to the shift in the force gradient will be maximized when the tip and the sample features are on the same order. If your features are larger than 100nm, though this is not important. Any probe less than that amount should do fine, as the signal should be quite good. Load the sample as normal, but if possible ground the sample, or if it is an insulator, mount the sample to a good ground plane, such as a gold coated slide or a metal sample puck from ted pella. Though EFM can be done on an ungrounded sample, it is generally preferable to have the sample grounded. This is because the field lines emanating from the tip will have a higher density if the ground plane is flat and closer to the tip. It is the perturbation of the field lines that cause the phase shift in the EFM image. Once the tip and sample are loaded, Tune the cantilever as normal for an ac mode image. Once this is done, it may be wise to take a quick standard AC mode image to be sure the tip has landed on a good area of the sample. Once this is done the settings specific to EFM can be adjusted.

On the main and nap channel panels, choose height, amplitude, phase, i and q. More will be discussed later regarding i and q.

To set the system for EFM, first set the nap mode to 'nap' or 'snap'. For most applications, nap is preferred, as it reduces the effects of topography on the EFM image. Generally snap mode is used if the researcher has plans to model the data later to extract quantitative results. For the purposes of this description, nap mode will be used. Note that the two are identical in operation other than the initial selection of nap or snap. Once the system is set to nap mode, choose the parameters that will be used in EFM. These should be:

Drive Amplitude  
Tip voltage

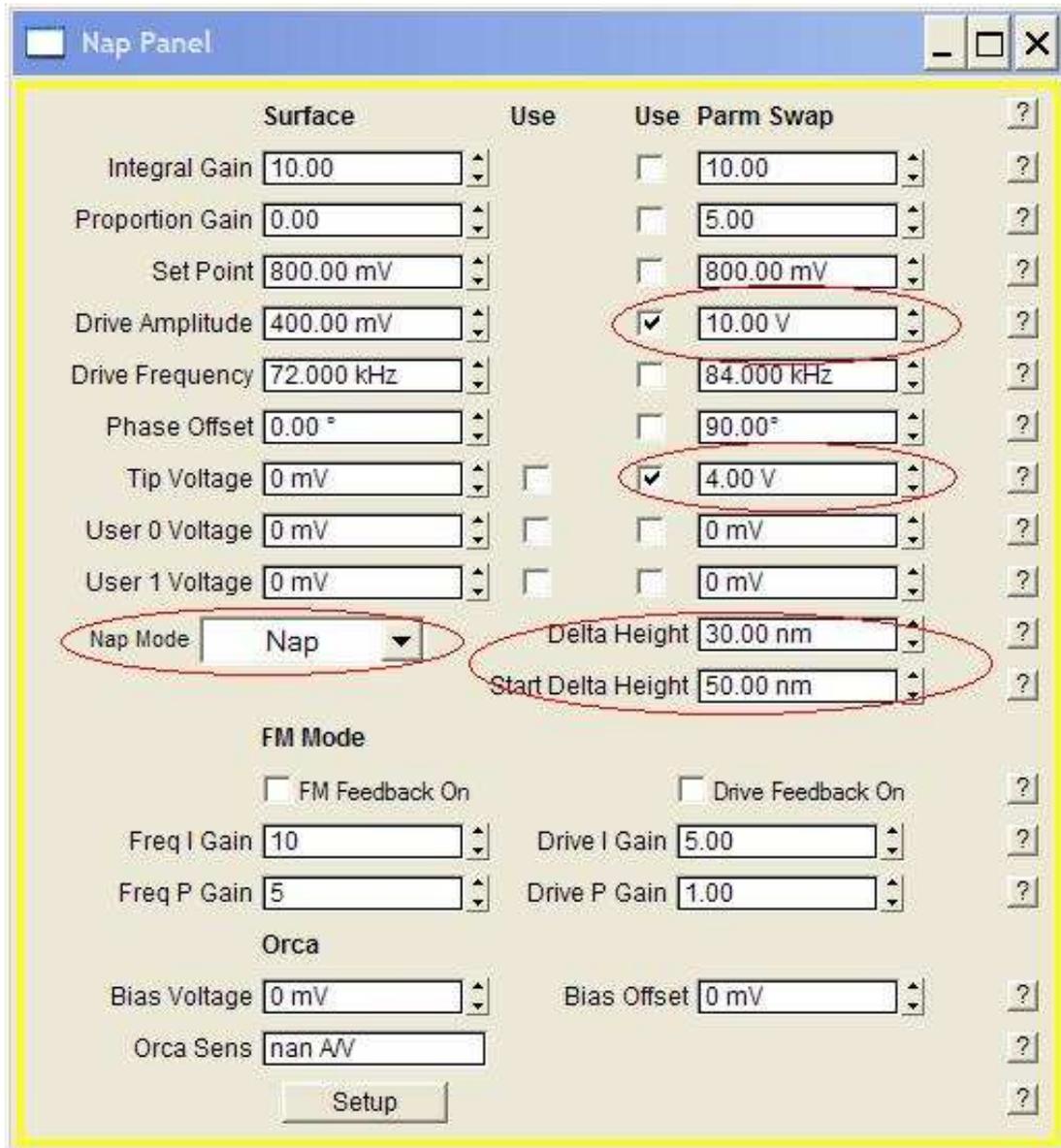


Figure 5. The nap panel for a typical EFM scan. Relevant fields have been circled.

To select these, check the boxes by the parameter boxes under the parm swap column on the nap panel. Next set the delta height to the desired value. A reasonable start point for this value is 40 or 50nm if the free air amplitude is 1V. This value should be linearly higher if the free amplitude is higher. Fig. 5 shows the nap panel with typical settings for EFM.

Generally to begin the scan, the drive amplitude is set to half the drive used for free air scans, and tip voltage can be set to zero. As the tip is scanned over the sample for the first frame, these values can be gradually raised until the desired EFM image is obtained. Once the values are set, begin a scan.

Note that 10 image windows will now appear once the scan starts. Of these, the main height, nap phase, i and q are probably the most important to bring to the front. The remaining channels can be dragged down to the bottom of the window until only the title bar remains visible on the screen. Minimizing the images will not remove them, as the software will eventually bring them back into view. Once the images are situated so that all of the desired images can be seen, adjustments can be made to the EFM parameters. Note that initially it is helpful to concentrate on the trace and retrace scopes rather than on the images, as it is typically easier to modify the parameters while monitoring real time changes there than in the image.

### **Adjust Scan Parameters**

The first priority is to be sure that the topographic scan is good, as the nap scans will rely on this to find each of the nap points. These adjustments are not different from normal imaging, so this description will not discuss this procedure at this time. Once the topography looks good, the drive amplitude and tip voltage can be adjusted under the parm swap parameter column on the nap panel. Slowly raise these two numbers until data begins to appear in the nap phase, nap i and nap q channels.

The amplitude during the main and nap scans can be viewed in real time on the sum and deflection meter. By watching the amplitude in real time, the user can ascertain fairly quickly if the tip is striking the surface during the nap scan. To do this, watch the amplitude during the nap scan. The drive amplitude during the nap scan should generally be lower than the drive amplitude during the main, or surface scan. The real time amplitude display for the nap and main channels can be distinguished by noting that the amplitude for the real time should match the setpoint under the surface column on the nap panel, and the setpoint parameter on the main scan panel.

At this point, the operation section of performing EFM is finished. From here, the user must adjust parameters to improve contrast to a point where the images are usable. One To be sure that images are related to the electrical properties of the sample and not the topography, the tip bias can be slowly changed during scanning. If the signal is related to electrical properties, then the phase contrast between the different features should increase with increasing voltages, rather than a general offset of the phase for the entire surface.

Note also that the EFM contrast is not just a result of the interaction between the tip and the sample. The cantilever, tip cone, and tip apex all contribute to the total contrast in EFM. If the tip apex is the dominant contributor, the highest resolution will result. It has been shown that this is best achieved if the tip is very close to the surface, so as minor adjustments are made, the tip should always be kept as close to the surface as possible. Further, a larger tip radius tends to increase the distance at which the major contributing feature is the tip apex, so it is recommended that the tip be no smaller than the smallest features on the surface that will be imaged.

Fig.5 shows an example image of EFM on Polypropylene with carbon black (CB) embedded into it. The clusters of CB particles show up in the EFM image as contrast in the amplitude and phase. Note that the phase and amplitude images from topography are quite different from the EFM scans.

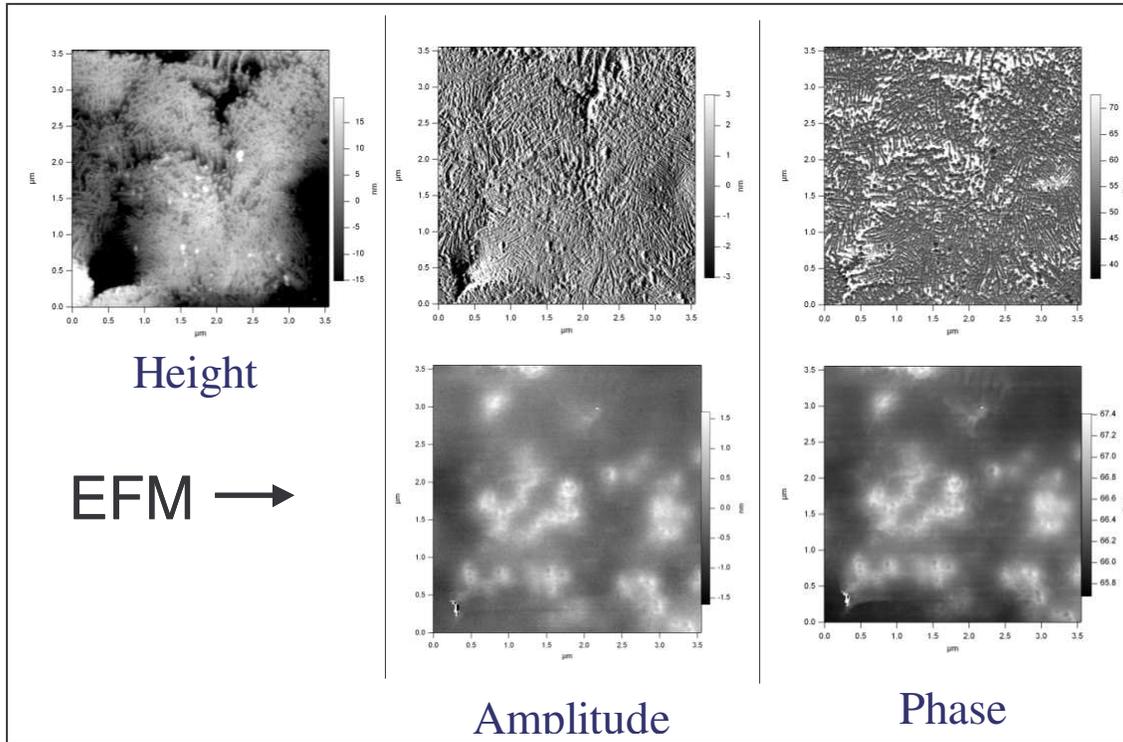


Figure 6. Example EFM image of polypropylene with carbon black particles (CB) imbedded into it. The areas of contrast in amplitude and phase are due to the presence of CB particles either at or below the surface of the polypropylene.