Conductive atomic force microscopy primer

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This primer details operational procedures for conductive atomic force microscopy, although much of the material is relevant to atomic force microscopy in general. The information is particularly relevant to Bruker/Veeco instruments.

Contents

1. Scope **3**

2. Module setup **3**

3. Probe setup **4**

3.1 Diamond-coated *4*

3.2 Metal-coated *4*

3.3 Metal wire *4*

3.4 Loading the probe holder *4*

4. Sample setup **5**

4.1 Cleaning the stage *5*

4.2 Cleaning the sample *5*

4.3 Affixing a wire with tape *5*

4.4 Affixing with a clamp *6*

4.5 Silver paint *6*

5. Scan management **6**

5.1 Scan parameters and making changes *6*

5.1.1 Scan size 6

5.1.2 Aspect ratio 7

5.1.3 Offset and stage reference 7

5.1.4 Scan angle 7

5.1.5 Scan rate/velocity 7

5.1.6 Scan lines 7

5.1.7 Setpoint 8

5.1.8 Gains 8

5.1.9 Z limit 8

5.1.10 Sample bias 8

5.1.11 CAFM feedback 9

5.2 Data types *9*

5.2.1 Height sensor 9

5.2.2 Height 9

5.2.3 Error 9
5.2.4 Friction 9
5.2.5 Current 10
5.2.6 Voltage 10
5.2.7 Trace and retrace data 10

5.3 Navigation 10

5.4 Data capture 10

5.5 Metadata 11

5.6 Probe wear 11

6. Experimental and troubleshooting methods 11

6.1 Constant voltage 11
6.1.1 2D imaging 11
6.1.2 0D time-domain measurements 11

6.2 Constant current 11
6.2.1 2D imaging 11
6.2.2 0D time-domain measurements 12

6.3 Current-voltage sweeps 12

6.4 Force-distance spectra 12

6.5 Thermal tuning 12

6.6 Resonant frequency sweeps 12

6.7 Estimating the applied force 12

6.8 Etching and tomography 13
6.8.1 Choosing parameters 13
6.8.2 Protocol for starting a process 13
6.8.3 Determining the etch depth 14
6.8.4 Drift 14
6.8.5 Sample phase change 14
6.8.6 Exercise (courtesy of Daniel J. Mannion) 14
6.8.7 Beast mode…. (not advised) 14

6.9 Probe cleaning 15
6.9.1 Sticky approach 15
6.9.2 Scraping approach 15
6.9.3 Thermal/melting approach 15

7. Data processing and analysis 15

7.1 Nanoscope analysis 15

7.2 Gwyddion 16

7.3 WSxM 16

7.4 Matlab 16

7.5 3D Slicer 16

8. Further reading 16

1. Scope

The aim of this primer is to provide sufficient operational information for a user to start working with advanced conductive atomic force microscopy (CAFM) procedures, such as tomography and constant bias time-domain measurements. It is most relevant to Bruker ICON/Veeco Dimension instruments, although many of the practices will be generally applicable to AFM/CAFM measurements. It is worth noting that the documentation/help files for Nanoscope and Nanoscope Analysis are very detailed and extremely useful. I would thoroughly recommend making use of them. I have not referenced material within the text, but I have included a selection of useful references at the end that are relevant to much of the technical discussion.

The details contained in this document combine the training I received when I started my EngD at UCL in 2013 with my experience using AFM and CAFM. Much of the information is based on approaches that I found worked during my experimental research, but they were not necessarily based on well-defined methodologies (although they were generally based on at least some modicum of science and/or scientific practice). As such, if you find alternative, more effective approaches, then I would recommend using them!

This primer describes measurement processes, from module and probe setup through software implementation and data analysis. It generally concerns contact-mode measurements (typical for CAFM) although some tapping-mode procedures will be mentioned. Notably, this is not an exhaustive training document for CAFM. Rather, it is written for users who already have some experience with AFM at least, in that they do not need to learn the theory and have a reasonable grasp of a typical scanning probe workflow. It should be used as a reference document rather than a training manual. Ideally, the user should take the information contained within and develop their own practical understanding and expertise, for which there is no substitute or alternative click-and-go approach. However, there should be sufficient detail here for a user to develop the workflow they need for their particular measurement.

Please email me with any questions or concerns, as I've certainly forgotten some details, described things poorly/incorrectly, or left some parts otherwise incomplete.

Enjoy!

2. Module setup

Always wear an anti-static wristband (plugged in…) when handling the CAFM module. If the wire of the wristband does not reach a nearby plug socket, then crocodile clips may be used to attach it to a ground point on the CAFM. Note that the stage is not at ground, it is either floating or used to apply bias. The metal guides below the stage are at ground, however, so the wristband may be attached here.

Attach the CAFM module to the scan head before loading the probe holder, while the scan head is fixed in place on the CAFM. At least partway tighten the bolts using the associated Allen key before plugging in the lead.

If the module calibration needs to be checked, it should be detached from the scan head, as the test resistor will not fit into the sockets if it is attached.

3. Probe setup

It is important to choose the right tool for the job. Each probe type has very particular strengths and definite weaknesses. There isn't really an overall "best" probe, but it is worth experimenting with different types in order to get a feel for which suits the demands of the work and of the user.

3.1 Diamond-coated

Diamond-coated probes are very stable and durable (e.g. Bruker DDESPV2). When boron-doped, they have a very high conductivity, and should demonstrate Ohmic behaviour on a metallic test sample (e.g. a gold film connected to the CAFM stage). The diamond grains that cover the probe will often produce a very sharp apex, giving the probe very high spatial resolution and, particularly, spatial resolution for current mapping. However, these apices will blunt quickly, particularly under high applied forces, so the probe may become blunter.

Another class are pyramidal, boron-doped full-diamond probes. Rather than coating a silicon probe, these are grown in a mould, so the entire probe tip is diamond. This makes them very tough, though it's debatable whether they are more durable than diamond-coated probes. My experience tells me that they are not, but their manufacturer (IMEC) would present the contrary position. These are much more expensive than diamond-coated probes, and the IMEC SSRM-DIA have 3 cantilevers per chip; this becomes an issue if you want to use the shorter cantilevers, as the longer ones must be bent out of shape, wasting them. These appear sharper, at least initially, than coated probes.

3.2 Metal-coated

Platinum/iridium coated silicon probes are the most generally used for CAFM measurements (e.g. NuNano Spark, which I would recommend, or Bruker SCM-PIC or OSCMPtR3). They are typically sharp and demonstrate good Ohmic behaviour, but the coating can wear off quite quickly, leading to a blunt and/or non-conductive probe. This is particularly true under high contact forces or during high-current measurements. Bear in mind, though the current may be small during a measurement, the current density will be huge due to the scale of the contact.

3.3 Metal wire

Full-platinum probes (I would recommend Rocky Mountain Nanotechnology RMN-Pt) are very useful for electrical measurements, but less effective for topographical imaging. They should always retain their conductivity (though the apex might become occluded by debris, but this can often be cleaned off), making them very useful for addressing small contact pads

3.4 Loading the probe holder

Always wear an anti-static wristband (plugged in, i.e. attached to ground) when handling any AFM probe, and do so on an anti-static mat using the loading mount. Static discharge can easily blow up the apex of a fresh probe during handling.

Sometimes there seem to be issues with placing the probe chip properly in the holder. The clip that holds the chip can extend past the apex of the probe, such that the probe cannot make contact with the sample during an approach. This only seems to be an issue with the CAFM probe holders. One solution is to move the chip forward in the holder, such that it overhangs the lip more. Also, the clip may be moved further down the chip, away from the cantilever end. Take care when tightening the clip screw, as overtightening can sometimes cause the end to pull up from the chip, so that it falls out when the holder is inverted.

It's worth checking that the CAFM probe holder wire is connected to the probe. Be gentle with the multimeter and try to avoid contacting the cantilever. If there is no continuity, wiggle the chip or clip around and retest. If it is not possible to attain continuity, check that the clip itself is connected to the wire. If it is, then a small blob of silver paint on the underside of the clip (properly dried…) might be sufficient to make a good contact with the probe.

Be careful connecting the wire of the probe holder to the socket of the CAFM module. The sockets are only soldered on so can be detached with force.

For contact mode measurements, align the vertical axis of the photodiode to -2 V. I'm not sure why this is, but I was trained this way and informed that this generally achieved optimum performance/sensitivity on Bruker ICON/Veeco Dimension microscopes.

4. Sample setup

Perhaps the most important part of setting up a CAFM measurement is ensuring that the sample is properly electrically connected to the stage. There are several steps and options to achieve this. If the sample substrate is to be used as an electrode (i.e. such that charge may be injected from the stage, to the substrate, to the sample, and collected by the CAFM probe) then it may just be placed on the vacuum chuck once the stage is clean.

4.1 Cleaning the stage

Ensure that the stage is reasonably clean. As (also, assuming) the microscope is typically used in ambient conditions and not in a cleanroom, we never know who's touched the stage or what sample(s) have been placed on the stage. This means there is often finger grease or small particulates on the stage, which can hinder adhesion between tape and the stage, as well as reducing the effectiveness of the vacuum chuck.

It's best to clean with IPA and some blue roll (or cleanroom/dust-free wipes), wiping the top of the stage fully and scrubbing it a few times with the roll. Drying can be improved with wafting the stage. Don't use acetone or any other solvents, unless you check that the other components of the instrument will not be damaged on contact. Bear in mind that there are numerous small holes for the solvent to get in to. Hence IPA is a good option, as it will not damage the microscope, but it will remove sufficient crud for an effective setup. Also be sure to clean any tweezers.

4.2 Cleaning the sample

It is also good practice to clean the sample before measurements, if unsure of how contaminated it might be, or if it is not behaving as expected. Ensure that cleaning will not adversely affect the sample materials, or, for example, remove contact pads or any other structures. Ultrasonication in deionised water (5 – 15 minutes, to remove larger particulates), then acetone (5 – 15 minutes, to remove grease and other organics), then IPA (5 – 15 minutes, to remove acetone and residual organics), repeated 1 or 2 times is usually sufficient, and deionised water is not always needed each time. Finish with IPA, as acetone leaves significant residual material. Adjust the duration of sonication depending on how dirty the sample is. Following, sonication, alternate squirting acetone and IPA on the sample several times, drying each time with a nitrogen gun. Finish with IPA.

4.3 Affixing a wire with tape

If the sample substrate is not conductive, or should not be used to inject charge, then an effective approach is to place a short piece of wire over a relevant contact point on the top of the sample. The wire may then be fixed in place with conductive aluminium tape and, as it's malleable, manoeuvred into good contact with the sample. Tape should always be handled using tweezers, to avoid

transferring finger grease to it, as this means it will stick poorly or not at all. Ensure that the wire is contacting the sample on the opposite side to where the probe holder will be. As the stage spins, this generally means that the sample should be set up rotated 180° from its intended alignment. Observe the components in the scan area to make a judgement on alignment.

With this approach, "good" contact should be around 30 Ω and stable, when checked with a standard multimeter (i.e. probe between the stage and somewhere one the sample that should be shorted to where the wire is touching, but don't contact the wire). If the resistance is more than 100 Ω or fluctuates a lot, then the connection is poor (unless the multimeter needs a battery change or the probes need cleaning).

4.4 Affixing with a clamp

The detachable Nanosurf stage with spring-locked arms makes a good alternative to tape preparation, and gives better control over the surface that the sample will be placed on (i.e. it may be cleaner) and makes electrical contact with a high applied force. Place one or both of the arms in contact with the optimum electrical contact point on the sample, but check that this does not flex the sample or cause it to not lie flat.

A drawback of this approach is that it can be much more difficult to address some parts of the sample, depending on its size. When using such a stage, take extra care to note the presence of components in the scan area, so as not to crash the scan head into the stage. Alternatively, it may be possible to get a bespoke sample holder made that is optimised for the CAFM instrument.

4.5 Silver paint

This approach is not recommended, although it can be very effective. Silver paint allows any location on the sample to be connected to a location of choice. For example, silver paint may be used to stick the sample onto a glass slide, and a track may be painted between the slide and a contact point on the top of the sample. Additionally, if the best contact point is a buried layer that is not accessible from the top or bottom of the sample, then painting the side of the sample should give good access.

If using this approach, make sure to use sufficient paint, but not so much that it leeches out into unwanted places. The paint should be thoroughly dried thermally, either on a hot plate or with a lamp (assuming this will not adversely affect the sample). The issues with this method are that silver is a very diffusive material, so it may end up where it is not wanted, and it also required thorough ultrasonication to remove the silver (during which process the silver will probably contact the entire sample surface). However, if done carefully, the electrical contact will be very strong and stable, and will last without the need for repeated setting up (though the sample may not readily be removed for measurements in any other instrumentation as it is now heavily contaminating).

5. Scan management

5.1 Scan parameters and making changes

5.1.1 Scan size

This is the length of the edge of the scan parallel to the fast scan axis (i.e. the direction the probe moves when tracing/retracing a scan line). The slow scan axis is the length of scan perpendicular to the fast scan axis (i.e. the direction the probe moves, incrementally, between lines). Turning the slow scan axis off can be a good way of optimising parameters over a fixed line of features.

5.1.2 Aspect ratio

The ratio of the length of the fast scan axis to the slow scan axis. This can only be positive. If the scan angle changes, the fast and slow axes will change, and the aspect ratio will rotate along with them.

5.1.3 Offset and stage reference

When navigating the sample, the stage will be moved relative to the probe. Using the stage reference is a good way to move by a controlled amount in a given direction, and is very accurate for precisely measuring feature locations on a sample for other analytical techniques (e.g. once a sample has been marked up or prepared for spectroscopy).

When changing the offset, the probe is moved relative to the sample, within the field of view. The axes are inverted, so positive x values will move the probe to a location to the left, and negative values to the right. Positive y values will move to a location below the probe's current location, and negative y values will move above.

5.1.4 Scan angle

The angle of the fast scan axis relative to the long axis of the cantilever. Scanning perpendicular to a linear feature is much more effective at measuring it that scanning parallel. The scan angle will also affect how the probe responds to the sample. Scanning at 90° will mean that the torsional stiffness of the cantilever plays a large part in maintaining the applied force. This can be a more effective means of applying a force than scanning at 0°, as the cantilever should bend less in the fast scan axis direction.

5.1.5 Scan rate/velocity

These two parameters are linked, though I would recommend making adjustments to the rate rather than the speed, as the rate will remain fixed if the scan size is changed, so the appropriate velocity may be set before changing scam size.

Scanning at less than 20 um/s is advisable, though I have found that not going above 15-16 um/s seems optimal. Faster scanning may cause the probe to wear more quickly, although the coefficient of friction between the probe and the sample is highest at very slow speeds. Be careful changing scan size, as the scan rate will stay fixed, so it is easy to accidentally set the probe to too high a velocity.

There is a distinct interplay between the probe velocity, gain and noise level. Higher scan rates will introduce periodic noise. I find that the gain may be reduced if the velocity or rate are high, as the probe movement induces a faster probe response.

For etching, slow scanning can be useful, to maximise the friction.

5.1.6 Scan lines

The chosen number of lines and samples per line determines the resolution of the image. Bear in mind that any probe will have a maximum topographical resolution, determined by the radius of curvature of the probe (usually around 10 mm for a good, uncoated silicon probe). Scanning with lines that are narrower than this will cause artefacts by repeated traversing of the same features/data.

A good means of finding an area of interest is to reduce the number of scan lines, to image more quickly. Journal-quality images should ideally have 512 samples per line (though 256 is usually acceptable). Images do not need to be square, i.e. the number of samples per line does not need to match the number of lines, but be aware that square images are required for 2D FFTs, at least in Nanoscope Analysis.

The number of lines can have a significant effect on the etching efficacy. Denser lines should remove more material in a single scan (in this context, the line separation is known as the "feed"). The compromise, as with all decisions on scan resolution, is that more lines will increase the scan time.

5.1.7 Setpoint

This is the value of the photodiode signal that the feedback loop aims to maintain. It should be set more positive that the initial photodiode alignment. In contact modes, this corresponds to the desired deflection of the cantilever to maintain a given force, so a more positive value corresponds to applying a greater force (in tapping mode, this is the desired amplitude of oscillation, so a lower value corresponds to bringing the probe closer to the sample). This will therefore be a distance, which can be determined by measuring the deflection sensitivity of the cantilever. Changing setpoint will induce a small spatial offset towards the probe chip, as the eucentric point of the cantilever is at its base, where it attached to the chip. Therefore, changing setpoint during etching, for example, can induce a misalignment of successive current maps with depth. The optimal setpoint is typically slightly closer to the sample than the value at which the probe loses contact.

5.1.8 Gains

These control the feedback loop, to maintain the setpoint. The I (integral) gain is best described as the sensitivity to topographical changes. If the I gain is off, then the microscope will not respond to changes in height, the deflection error will be large, and there will be no height data. If the I gain is very high, then the measurements will become noisy as the instrument responds to topographical features that aren't there. A "good" value for the I gain will depend on the probe, the sample, the day, the scan mode etc, there is no definitive method, other than the play around and see what gives a good and consistent line/image in both trace and retrace. Iterating between setpoint and gain is good advice. I have no idea what the P (proportional) gain does on Veeco Dimension/Bruker Icon instruments, I usually just set it slightly higher than the I gain, such that $P \approx I \times 1.125$, but it seems to make no difference what it's set to. This is based on advice from Bruker when they installed the ICON.

The gains can play an important role in point spectra (current-voltage, force-distance) as they will still try to maintain the setpoint. Switching them off can, to an extent, "ignore" the mechanical interaction with the surface, i.e. if the surface is distorted, the probe will not be moved to account for this. Gain adjustment in spectra, particularly current-voltage, can make a large difference to the behaviour. Note the deflection and the height sensor data during such ramps, as these can give a good indication of whether the probe height has changed. A jump in current might correspond not only to a change in conductivity, but also a change in the contact mechanics.

5.1.9 Z limit

The height measurement is binned, and the number of bins is fixed. Therefore, for very smooth samples (RMS roughness < 5 nm) it can be worth reducing the Z range so that the binning is finer. Be careful with making adjustments, as too quickly reducing the range can cause the probe to be out of absolute range of the surface, giving red or yellow feedback on the Z piezo. Always maximise the Z range when approaching the surface, offsetting the scan or withdrawing, to avoid the Z piezo going out of range. In very large scans, the piezo can go out of range between edges of the scan area.

5.1.10 Sample bias

Typically, electrical bias is applied from the stage, relative to the grounded probe. Thus, electrons are either injected from the stage (negative bias) or from the probe (positive bias). Inject from the stage to minimise anodic oxidation on the surface. Applying a bias can induce Coulombic effects (i.e. attraction/repulsion) so the topography may be convoluted. Often, streaks appear, or the surface can

become inverted. This also true of charging effects when no bias is applied, i.e. the probe is responding to Coulombic forces.

The sample bias should not be changed too quickly, as the current density at the probe apex can be very high. Bear in mind that the contact is nanoscale, so even a few nanoamps can produce a density in the range of a few A/cm^2. Furthermore, a quick change in bias can also cause a quick and large change in the attractive or repulsive forces between the probe and sample. A good rule of thumb is to change the bias no faster than 1 V/s.

Of course, some measurements/processes will require a more rapid change in bias (e.g. time-dependent measurements, or voltage steps). In order to avoid crashing the probe during a quick change, the probe may be pressed into the sample at a higher contact force than is used for imaging (i.e. make the setpoint more negative). If this effect is negated, then the main risk is blowing up/melting the apex during the voltage ramp. Assuming this is not an issue, i.e. if using a platinum wire probe that's already blunted or the probe is in contact with a larger metal contact, then quick voltage changes may be applied.

The sample bias should be turned off when entering ramp mode, as it will continue to be applied, unless this is desired. The bias should also be turned off if the scan is paused, as it will not automatically turn off. Thus, spots on the sample may be heavily stressed as the probe will hold position.

5.1.11 CAFM feedback

This are the parameters for constant-current mode. With the feedback on, the instrument will seek to maintain the current setpoint by varying the voltage within the specified range. The feedback gain controls the sensitivity, with high gain introducing significant noise in the voltage. Optimum feedback is achieved at very slow scan rates. I would suggest the minimum rate of 0.1 Hz, regardless of scan size, as the probe velocity seems less important. When engaging feedback mode, the probe speed will slow down significantly, but this may be addressed by subsequently setting the desired rate. When disengaging feedback, the probe speed will jump up, so take care that the velocity does not become too high. I would also recommend 512-1024 samples per line, for optimum monitoring of the voltage. The CAFM sensor samples at 56 Hz, so it is good practice not to use imaging settings at a higher sample rate than this.

5.2 Data types

5.2.1 Height sensor

The measured height inferred from the deflection error data and height sensitivity calibration.

5.2.2 Height

Distinct from height sensor. Sometimes, noisy interference in height sensor data will not appear in height data, so it's worth using height instead of height sensor.

5.2.3 Error

The raw signal of the probe deflecting as it scans over the sample. Not really necessary to capture if you need the channels for other data types.

5.2.4 Friction

A measure of the side-side (torsional) deflection of the cantilever (i.e. orthogonal pairs of photodiode quadrants to those used to measure vertical/Z deflection). Often useful in determining that the probe has picked up debris, which will cause a torsional deflection without any topographical change. This will show up as a complex diagonal streak in friction imaging.

5.2.5 Current

Raw current, calculated from an output voltage (driven across a resistor, probably) according to the selected gain/sensitivity of 1 nA/V (high) or 100 nA/V (low). High sensitivity gives a noise floor of around 750 fA, with a saturation of around 12.3 nA. Low sensitivity gives a noise floor of around 75 pA, with a saturation of around 1 uA.

5.2.6 Voltage

The best way to record the sample bias is to set one of outputs to DC Sample Bias and set one of the channels to an Input (1, 2 or 3). Then, connect the input and output via BNC on the CAFM controller. Otherwise, each data file will just record a single value for the applied voltage. Most generally, bias is applied from the stage, with the probe held at ground.

5.2.7 Trace and retrace data

Trace data (blue in Nanoscope) corresponds to the first extension of the probe along a scan line. Retrace data (red in Nanoscope) corresponds to the probe tracking back along the same line before moving to the next line. An analogous configuration applies to the extend/retrace/ramps of point spectra.

5.3 Navigation

The optical focus of the probe and sample are used to determine a starting point for the probe to engage and start measuring. They are not, and should not be, at the same height. For most probes it is fine to focus on the back of the cantilever. For some probes, such as platinum wire (for CAFM, or even STM pulled-wires) in which the apex extends from the cantilever, focusing on the apex can aid in engaging with the sample.

Always approach the sample at minimum zoom. Typically, the sample is approached after focusing on the tip at high zoom, so this is something to be aware of. Otherwise, it is easier to overshoot the focal point of the optical microscope and crash the probe into the sample (this is always easy at fast focus speed).

If applying a high loading force (e.g. greater than that used for imaging), then the force should be reduced before changing the offset. Otherwise, the probe will drag on the sample with the setpoint force, which can scratch the sample and blunt the probe. The same is true when in ramp mode; whatever setpoint is entered in the scan parameters menu will be applied constantly unless changed.

5.4 Data capture

Make sure to save your data with appropriate, future-proof, fool-proof labels. This will save a huge amount of time in the long run, particularly if you're accumulating a large number of files. The probe type and probe number are useful but overlooked pieces of information (e.g. in case particular probes are used only on particular samples, or their history needs to be recorded). It's good practice to make a new capture directory in your own folder for each measurement session and save data directly here, rather than later moving the data. Make sure that your chosen folder is selected before you start capturing data, so that you know where you're saving to and don't lose anything.

The capture controls are not entirely intuitive. Turning 'Capture' on will save the next frame if 1) all scan lines in one or other direction have been completed and 2) no parameters have been changed (although samples bias may be changed without affecting the capture). 'Capture continuous' will capture every frame that satisfies the above capture conditions, until it is switched off. 'Capture now' will capture however many lines of the image have been accumulated from the top or bottom since

the frame has started. Capture last will save the previous frame, regardless of whether it was complete, or parameters were changed.

For imaging, it is recommended to scan with a probe velocity no greater than 20 um/s. Slower is often better, to reduce both probe and sample wear. I recommend around 15 um/s for best imaging results and probe lifetime. Be careful with setting the scan rate, as this will determine the probe velocity (although the velocity itself may be set to determine the rate). In particular, if the scan size is changed, then the scan rate will stay the same, so the velocity will change. Make sure to change the rate before the scan size, to avoid implementing a rate much greater than 20 um/s.

5.5 Metadata

Each scan file, at least in Veeco/Bruker Nanoscope data, will contain thorough metadata, including all sensitivities, scan direction, angle, time etc. Exported as ASCII data with a header, all of this information will be accessible when reading the file, and consistently locatable using, for example, Matlab.

5.6 Probe wear

The probe will become worn over the course of measurements. Unfortunately, this is unavoidable, though it may be minimised and accounted for by using appropriate measurement practices and careful analysis. Lower applied forces (via choice of setpoint) will reduce probe wear, so the optimum imaging setpoint for minimal wear will be “just” in contact with the sample. Reduce the setpoint until the probe loses contact (the Z piezo goes very negative, turns yellow, and then red) and then increase the setpoint slightly to regain soft contact. If Z goes too red, it can be challenging to regain the contact, so once it starts turning yellow then the setpoint should be increased. One drawback of such a low contact force is that the probe can lose contact over the course of a scan, particularly for large areas.

6. Experimental and troubleshooting methods

6.1 Constant voltage

6.1.1 2D imaging

Imaging with a sample bias applied applies this voltage across the entire scan area in order to map the resulting current. The topographical mapping may be affected by Coulombic interactions, so artefacts are common and something to be aware of. It may be advisable to take separate topographical and current maps, i.e. map topography before current, and then again afterwards, to account for topographical artefacts. However, charge accumulation can also cause persistent topographical artefacts, so monitoring topography over several scans, or a longer duration, may be necessary.

Other than for deflection, no feedback is engaged in this mode, i.e. the current is not limited, though the detector may become saturated. Another approach to current limitation is to place a resistor between the stage and sample.

6.1.2 0D time-domain measurements

If the scan size is reduced to 0 nm, then the image will retain the same number of lines and samples per line, but will be only be sampling in time, and not spatially. Thus, a user-controlled voltage may be applied to a spot on the sample and the resulting evolution of current measured in time.

6.2 Constant current

6.2.1 2D imaging

With the CAFM feedback on, the instrument will try to apply the current setpoint across the whole scan area, by varying the voltage between the selected upper and lower limits. In this way, the current

and the voltage may be limited by the additional feedback loop. As with constant-voltage scanning, topographical artefacts should be expected due to charge injection.

6.2.2 0D time-domain measurements

Setting 0 mm scan size allows the user to apply a constant current, or limited current/voltage measurement to a spot on the sample, and track the change in voltage over time.

6.3 Current-voltage sweeps

There is an issue with changing ramp parameters. It seems to occur when switching between monitoring current and monitoring deflection, as well as when changing data types. If possible, set up the ramp parameters before engaging, to avoid software crashes while the probe is in contact with the sample. This is not always possible, of course, for example when wanting to perform mechanical and electrical spectra at the same location without breaking contact or losing position. Good luck...

Current-voltage sweeps allow a voltage ramp to be applied at a fixed location, with a fixed rate, while monitoring current, as well as other parameters. Mechanical parameter, such as deflection sensor and height, can give useful feedback on probe movement during a ramp. A sample bias may be applied while ramping sample bias. If entering ramp mode, any applied sample bias will be left on.

6.4 Force-distance spectra

Force-distance spectra allow mechanical properties of the sample to be probed. In particular, performing these measurements on a nominally hard sample (e.g. sapphire) allows the instrumental deflection sensitivity to be determined. This corresponds to the measured change in photodiode voltage as the probe height is changed (pressed into the sample) in nm/V. This is the linear part of the force-distance spectrum. There might be some slight difference between the extend and retract values; the retract value is more commonly used. The value may vary by a few percent between measurements that are nominally the same.

6.5 Thermal tuning

Thermal tuning may be performed in contact modes (e.g. CAFM mode) to determine the spring constant of the cantilever. The instrument allows the cantilever to oscillate under thermal fluctuations while reading the deflection signal. This gives a resonant peak with a power determined by the cantilever temperature. Typically, it is assumed that the cantilever is at 21 C, but this is hard to be sure of. This technique is also less effective with stiffer cantilevers (such as might be required for etching), as the oscillation is not very pronounced, leading to a very noisy peak. Multiple peaks may be exported as ASCII data and summed to improve the signal/noise ratio. Don’t cancel or abort tuning as can crash the software.

6.6 Resonant frequency sweeps

Resonant frequency tuning may be performed in non-contact (tapping) to determine the resonant frequency of the cantilever. The cantilever is driven through a range of frequencies while reading the photodiode signal, and the strongest peak is selected. In tapping mode, when imaging, the cantilever is driven just below this frequency. The resonant frequency changes very little over short time periods and the signal/noise ratio is very good, so a single measurement should generally be sufficient to characterise a probe. Don’t cancel or abort tuning as this can crash the software.

6.7 Estimating the applied force

The normal force, F, applied by the probe to the sample follows Hooke’s law, $F = k.x$

The extension of the probe, x, is the product of the setpoint voltage, V, and the deflection sensitivity, S. So, $F = k.V.S$

The spring constant of the cantilever, k, may be estimated using thermal or resonant frequency tuning. If using the resonant frequency, consider the probe as a simple harmonic oscillator (mass on a spring), wherein the resonant frequency, $f = [\sqrt{(k/m)}]/2\pi$

Making the assumption that the oscillating mass, m, is the same for any probe, we may then use the manufacturer's quoted values for nominal spring constant and resonant frequency to determine the real spring constant from the measured resonant frequency via $k_{real} = k_{nominal} \cdot (f_{mreasured}/f_{niminal})^2$

A further factor can be incorporated by measuring the cantilever length, as k is proportional to the length cubed. Using the minimum and maximum values of the cantilever parameters (if available from the manufacturer), a more accurate estimate of k may be made within the expected range of values that may apply for a given f. We may consider this additional level of complexity as letting the mass vary. However, the gain in accuracy may not merit this addition, as this is still, ultimately, just an estimate, as the mass and temperature of the cantilever are hard to determine. Highly accurate determination of the spring constant is a big challenge and not necessarily necessary for most procedures. A reasonably accurate estimate, stated as such, should be sufficient.

An additional factor of 1.44 is applied to account for the angle of the cantilever relative to the nominally flat sample surface.

6.8 Etching and tomography

6.8.1 Choosing parameters

6.8.1.1 Applied force

This will depend on the sample material, the desired scan duration, the feature(s) under investigation. Higher forces may remove more material, but will also cause more compression and may have a greater effect on the phase of the sample. Bear in mind that the pressure at the contact point will be very high because the contact area is so small. The probe will also blunt faster at higher forces.

The force may also change the current mapping, as a higher force will make better contact, so the sample bias may need changing to account for this and avoid detector saturation or high currents.

6.8.1.2 Number of lines

A denser feed should remove more material and produce a smoother trench, but will also cause more probe wear and a slower measurement.

6.8.1.3 Probe velocity

Slower scans will give a higher coefficient of friction, so should remove material more effectively, but measurements will take longer.

6.8.1.4 Integral gain

Scanning with the gain on will cause the probe to track the surface. In some cases, particularly on softer materials, it is useful to prevent this from occurring as the material deforms around the probe. So, we want to avoid tracking this deformation and instead focus on applying a desired force to the etch region, without feedback on height. Note that turning the gain off entirely can cause the probe to detach from the surface gradually or suddenly, so it can be useful to just set the gain very low.

6.8.2 Protocol for starting a process

I have found that an effective procedure is to begin scanning at a force appropriate for imaging. Then, gradually increase the applied force to the desired level over several scans. This prevents edge

ruptures, wherein starting the measurement at the desired force causes a weakness in the sample at the start edge, eventually resulting in a tear and an uneven trench.

Patience can be a virtue. It can be tempting to increase the force or velocity or change some other parameter if etching does not appear to be happening. It is often worth waiting a little while to see whether this is really the case, as measurements can be very slow. I have found the most success with extremely slow processes (e.g. 8-12 hours overnight) at relatively low force (up to a few uN).

6.8.3 Determining the etch depth

It is difficult to track the true etch depth during a measurement, because the scanner can drift gradually or suddenly, and the applied force affects the measured height. However, there are some indicative observables.

As material is removed is removed, it will pile up at the edges of the etch trench. Thus, the edges will gradually show an increase in height and friction as material accumulates.

Surface features will gradually become smoothed, or pockmarks will be produced in some locations of the mechanical or contact properties of the etch region vary spatially.

For a precise measure of etch depth, reduce the applied force to a suitable value for imaging and zoom out to scan a larger area. Scanning with the fast scan axis perpendicular to that used for etching can reduce the likelihood of knocking debris back into the trench.

6.8.4 Drift

Over the course of long measurements, and as the probe height changes, the scan may drift. Thus, it may be necessary to account for this.

Changing force will cause drift.

6.8.5 Sample phase change

Note that an amorphous material mat become crystalline under pressure from the probe. Conversely, a crystalline sample may be amorphised. Such a change in phase can cause the sample to harden. It is also possible that the material may become soften, although if we also consider that the probe is compressing the sample, then it may be most likely that we would only observe hardening..

Line density.

6.8.6 Exercise (courtesy of Daniel J. Mannion)

Try writing your name, or EE, or UCL or some other acronym or shape in a material that you're studying! This is good practise for getting to grips with the scan offset (navigation), scan angle and aspect ratio (scan shape), changing applied force, depth measurements, changing scan size. Depending on the size of the features written, different etch depths ma given different colours optically. Alternatively, by curating the colour scale of the data, it would be possible to draw something with multiple colours or shades.

Tweet your results, put in plenty of @s and #s!

6.8.7 Beast mode…. (not advised)

When etching for the purpose of producing a coarse structure with a blunt probe, for example a sample marker, sometimes it is very difficult to remove any material at all, even at maximum setpoint, either because the probe is too blunt, the sensitivity is too low or because the sample material is too compressed. One workaround is to adjust the alignment of the laser on the cantilever, or the

photodiode (though I have not tried this second option). This reduces the deflection signal, causing the probe to be pressed harder into the sample in order to reach the setpoint.

6.9 Probe cleaning

It is possible to clean a dirty probe, and even re-sharpen a blunted metal probe, although the process might also cause blunting. A combination of approaches may be necessary to condition the probe sufficiently for use. Ultimately, mechanical or thermal energy is used to agitate dirt from the probe tip. These techniques should be used if the measured topography is very peculiar, much flatter than expected, has a significant, very large probe artefact, or the probe is not demonstrating stable conductivity.

6.9.1 Sticky approach

Try scanning on a surface that is stickier than the probe, such as gold, at a number of angles (e.g. 0, 90, 135). The aim is to transfer any debris from the apex to the scan area. Scan back and forth repeatedly until the image becomes clearer. If debris is deposited in the scan area, it is worth moving periodically to a new area to see whether the scan quality has improved. Unfortunately, this is not a definitive approach, as it is possible for adhered debris to behave as an apex. Thus, the image quality and resolution might improve, but it may be sharp piece of debris that is doing the imaging.

This approach may be performed at any force of scan speed. At low contact forces, it may be slower but gentler, gradually cleaning the probe. At higher forces, the process may be quicker and better at removing more stubborn debris. This approach is best used on blunt or diamond-coated probes, i.e. where it is not necessary to retain a very sharp apex, as blunting may occur during the cleaning process.

6.9.2 Scraping approach

Another approach is to try and remove debris that is adhered to the tip by scraping it across a tall, high aspect ratio feature. This is a similar approach to 6.9.1, but the added tall feature can improve the effectiveness. Suitable features are step edges, lithographic patterns, and even rough/mucky sample areas (e.g. tweezer scratches), assuming that these will not make the probe dirtier in the process. Or, this is a good approach for removing large pieces of debris, before utilising another technique for finer cleaning.

6.9.3 Thermal/melting approach

This is particularly effective on full-metal probes. It can be attempted on metal-coated probes, although the coating may be lost. The probe

7. Data processing and analysis

7.1 Nanoscope analysis

To download v1.4 http://nanoscaleworld.bruker-axs.com/nanoscaleworld/forums/t/812.aspx

v1.4 is a little bit frustrating to use, if you're used to v1.5. Some of the functions are broken (e.g. selecting an area to measure roughness, rather than the whole image). v.1.5 is available somewhere. I have a copy, if needed.

The step analysis tool is invaluable for assessing height differences, such as for an etch trench. It averages over a wide cross section, so it's far better than a cross section. The data may also be levelled without needing to perform a plane fit or flatten, which can sometimes be challenging.

The FFT function is excellent for removing noise from images without losing information. Only works on square images, i.e. where the number of lines is equal to the number of pixels per line.

Batch processing may only be done on a version that is linked with Nanoscope (i.e. on an acquisition machine, such as in the Nanolab, 904 Roberts Building), although I think v1.4 might allow batch processing on other machines. This is very useful for e.g. cropping a region of interest from multiple scans in a sequence, or flattening/FFTing a set of images, or removing baseline/background from multiple FD spectra etc. Make sure to select 'Save modified files' to save the processed data as new files.

7.2 Gwyddion

Gwyddion is a reasonably good piece of software, given that it's free. It's not as polished as Nanoscope, but much of the functionality is there. I found Gwyddion more useful to begin with, but once I started to learn how to use Nanoscope analysis in combination with Matlab more effectively, I stopped using it. I would recommend using Nanoscope analysis if possible, but Gwyddion is a good alternative if necessary.

7.3 WSxM

Maybe I'm missing something, but I don't know why anyone uses this software, it's awful. Not recommended.

7.4 Matlab

My Matlab scripts for data processing and analysis are available via MathWorks and GitHub.

https://uk.mathworks.com/matlabcentral/fileexchange/73789-surface-analysis

https://github.com/MarkBuckwell/surface-analysis

Scripts are available for batch processing thermal tuning, resonant frequency tuning, FD spectra, current-voltage measurements, 2D images/cross sections, constant bias tracing/spiking statistics etc. ASCII files exported from Nanoscope Analysis are generally usable, with or without headers included.

There is an array of other user-created Matlab (and otherwise) scripts available online.

7.5 3D Slicer

3D Slicer may be used to assemble 2D image slices into 3D tomograms. The software is designed for bio-imaging. There might be better freeware/open source software available elsewhere, although Slicer itself is quite open-source and has bolt-on modules, including the capacity for module design with Python.

https://www.slicer.org/

You can import stacks of scans and render a composite 3D model. The colour blending, transparency, orientation, clipping/cropping can all be adjusted. The software is clunky, but it works. Additional modules can be installed to export meshes for Blender, for example.

8. Further reading

The following is a non-exhaustive list of useful/informative/relevant publications for CAFM, particularly tomography.

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Ohler, B. (2007). Practical Advice on the Determination of Cantilever Spring Constants. http://nanoscaleworld.bruker-axs.com/ AN94, 1–12. Available at:

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p.s. please cite me and these other excellent authors!