Laboratory for Energy and NanoScience

COMPENDIUM

CHAPTER 1:
Setting-up an amplitude-modulation (AM) AFM for tapping mode

LENS COMPENDIUM
Published July 2020

Authors:
Sergio Santos[1,3]
Chia Yun Lai[1]
Tuza Olukan[1]
Carlo Alberto Amadei[4]
Matteo Chiesa[1,2]

Corresponding author: Sergio Santos - ssantos78h@gmail.com

Affiliations:
1. Department of Physics and Technology, UiT The Arctic University of Norway, Tromso, Norway
2. Laboratory for Energy and NanoScience (LENS), Khalifa University of Science and Technology, Masdar Campus, Abu Dhabi, UAE
3. Future Synthesis, Skien, Norway
4. The World Bank, 1818 H Street, NW Washington, DC 20433 USA

Design and layout: Maritsa Kissamitaki[1]

CONTENTS

Setting up an AFM for Amplitude Modulation operation

In this chapter, we are demonstrate how to set-up an amplitude-modulation (AM) AFM for tapping mode operation. We use a Cypher scanning probe microscope from Asylum Research. We employ our techniques for tip characterization and discuss the attractive and repulsive regimes.

A detailed video for this demonstration can be found here.

Brief introduction of an AFM system

An AFM system includes a cantilever with a sharp tip to scan and sense a sample's surface. In the typical (2020) setup, a laser beam needs to be placed on the front end (Figure 1) of the cantilever to detect its position and to be able to take a reference to move the cantilever towards or away from the sample's surface. There are different types of cantilevers where several properties can vary from the dimensions of the cantilever, the coatings on the tip, and the materials it is made of. Criteria on how to choose the cantilever depends on purposes defined by the experiments and the sample's properties.

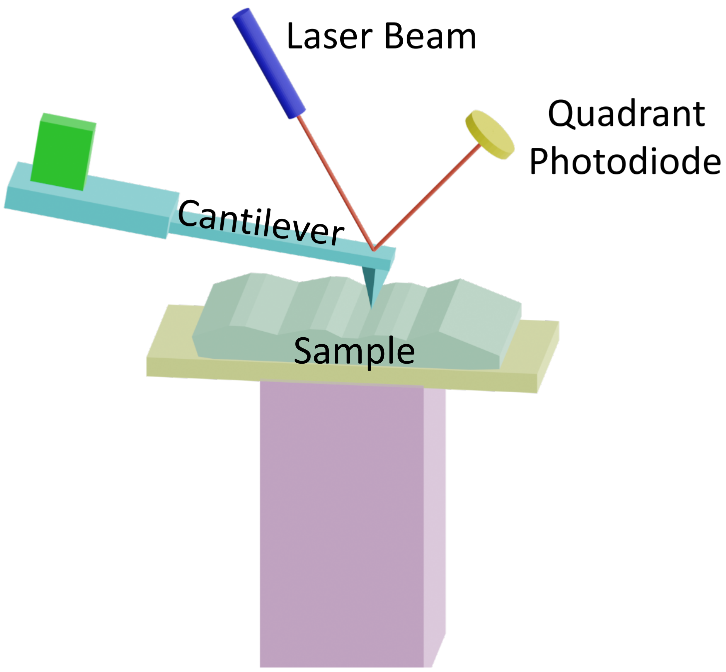


Figure 1: Laser Beam place on the front end of the cantilever.

Calibrating the cantilever

In tapping mode, the cantilever is oscillating at its resonance frequency with a certain amplitude. The resonance frequency can be found using thermal analysis (Figure 2). As for the cantilever's parameters (such as spring constant, quality factor etc.), these can be tuned by fitting a Lorentzian to the thermal curve (Figure 3). The thermal data simply makes the cantilever oscillate due to random motion of molecules in the air. The energy of these molecules is dependent on the equipartition theorem and therefore on temperature. Simple models are employed to fit the curve. If your AFM lacks thermal fitting one can always select the peak by inspection and apply the equipartition theorem: $1/2k_BT$=average energy of each degree of freedom of the cantilever.

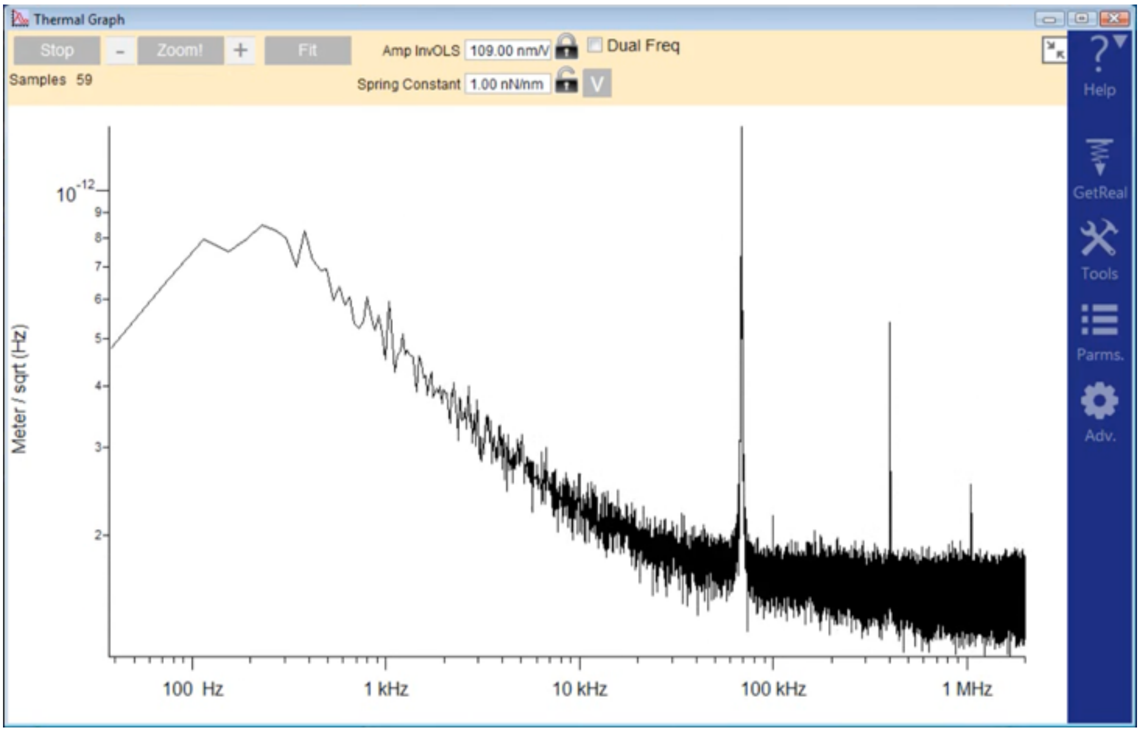


Figure 2: Thermal analysis panel.

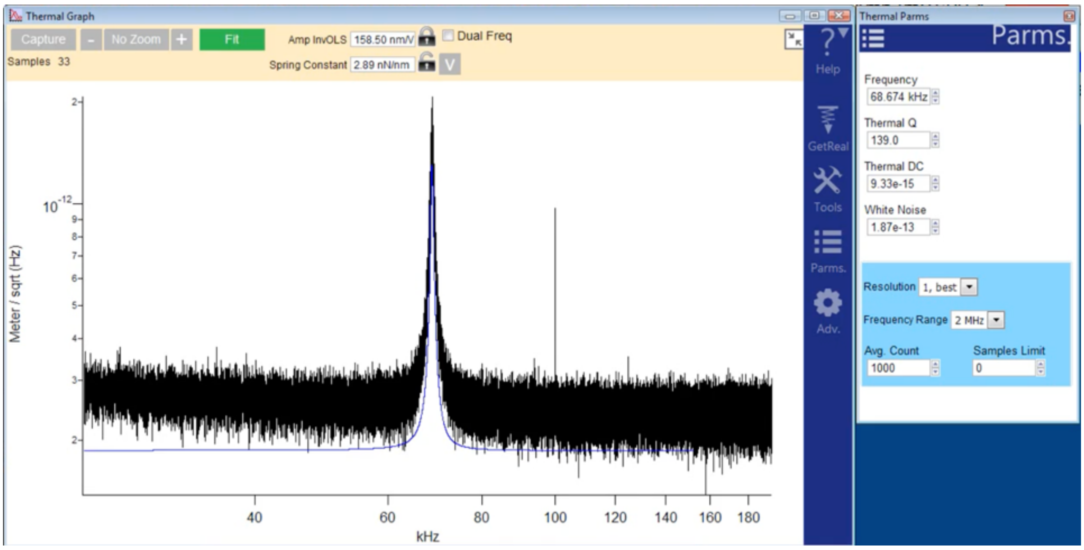


Figure 3: Fitting the thermal curve to extract the quality factor and spring constant.

Steps to approach the tip to the surface

1. Insert the cantilever into its holder (Figure 4).

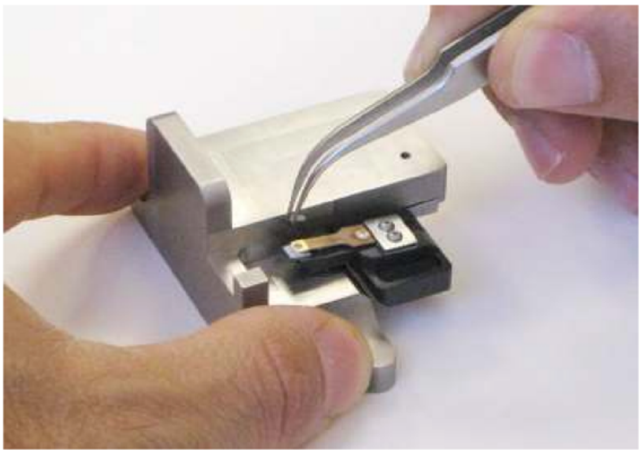


Figure 4

2. Fix the cantilever holder on the Cypher head (Figure 5).

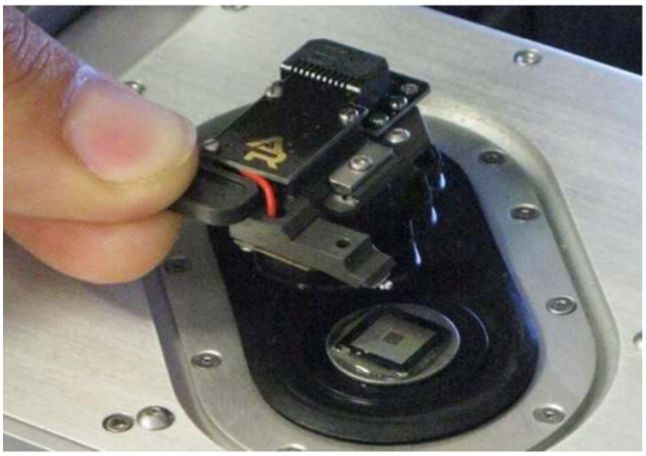


Figure 5

3. Place sample area of interest towards the center of scan plate. You can use magnets to hold the sample holder on the Cypher scan plate (Figure 6).

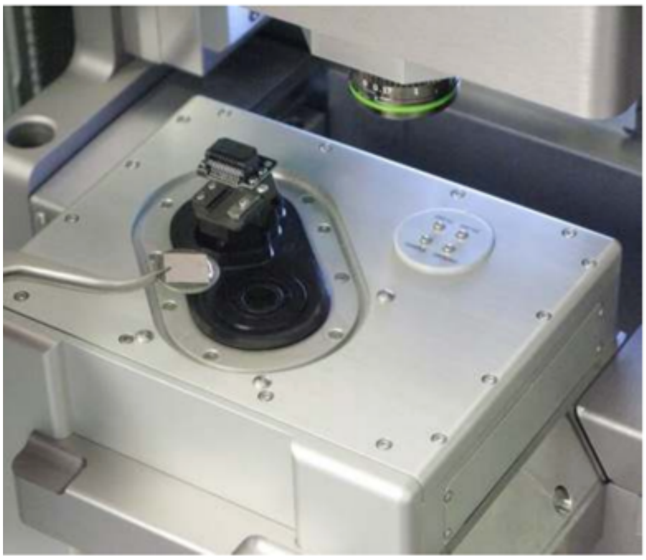


Figure 6

4. Click on the "standard" tab then "topography" and "AC Air Topography". This indicates that we will operate the tapping mode in air. Choosing the appropriate mode will set up some basic parameters for you, so you don't need to adjust it afterward (Figure 7).

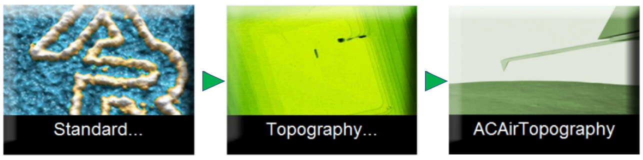


Figure 7

5. Use the **Igor software** to put the laser spot (Figure 8), find the focus of the tip (Figure 9) and the focus of the sample (Figure 10).

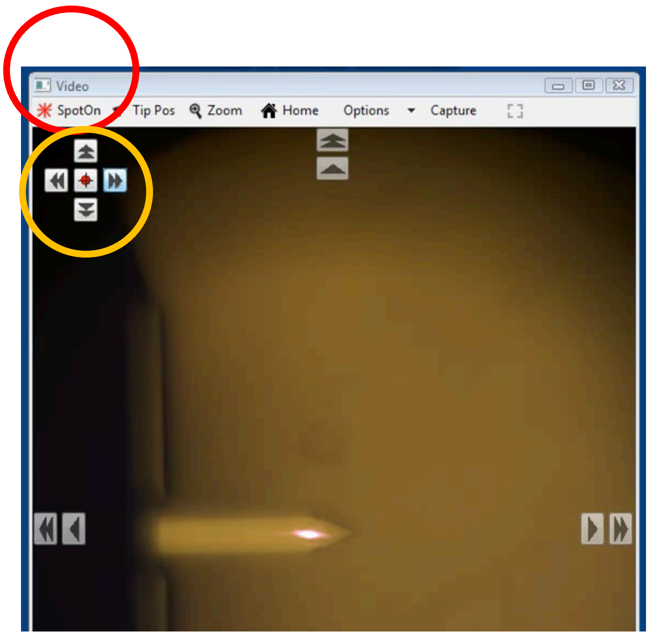


Figure 8: Use the "spot on"(red circle) to place the laser spot. The directional arrows in the orange circle can be used to adjust the position.

Figure 9: Ensure that the "Move Focus" is activated. Arrows in yellow rectangle can be used to adjust the focus on the tip. Use the "SET" button to register the focus position.

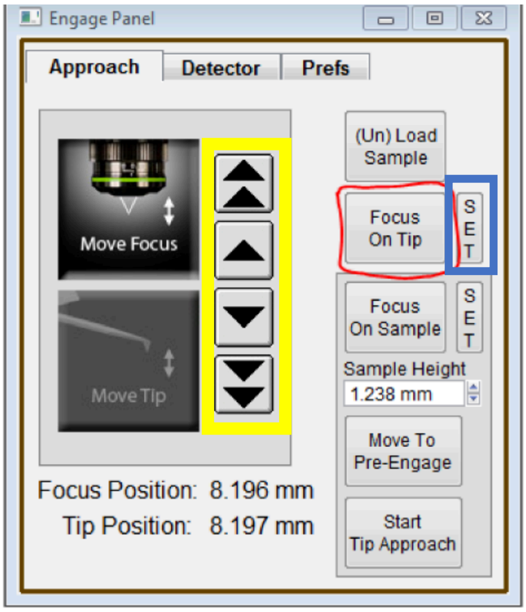
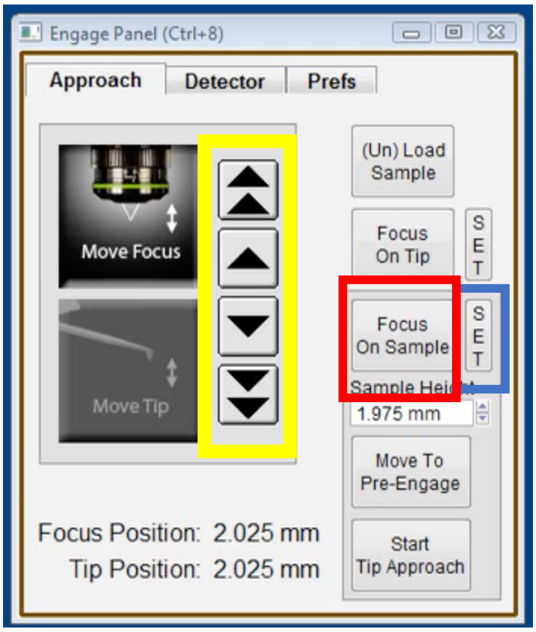


Figure 10: Use arrow keys to focus on the sample. Then accept the focus position by clicking on the "SET" tab.



6. Click Move to pre-engage (Figure 11).

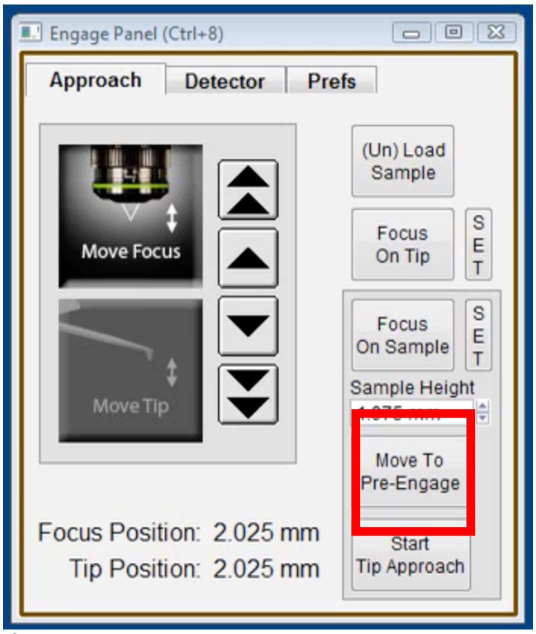


Figure 11

7. The cantilever should be driven at the resonance frequency. To find the natural frequency, you need to perform the Thermal test (Figure 12). After the thermal test, copy the frequency on the thermal tab and paste it in the main tab of the master panel (Figure 13).

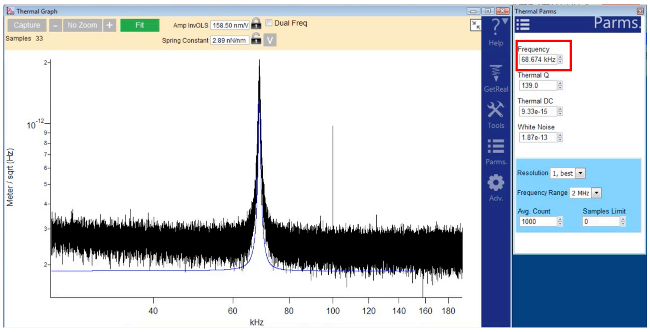


Figure 4

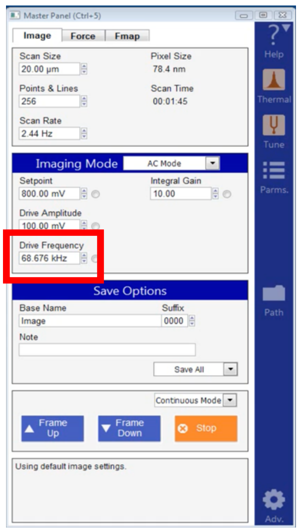


Figure 13

8. Now the free amplitude can now be adjusted. Usually for this system, I set the amplitude to about 100 millivolts and the set point to 80% of the free amplitude (80 millivolts) (Figure 14). This corresponds to approximate 5 nm of free amplitude A0 and 4 nm of Set point. This would be a soft engage since the energy of the free cantilever is approximately $1/2kA_o^2$ (k is the spring constant not Boltzmann's constant).

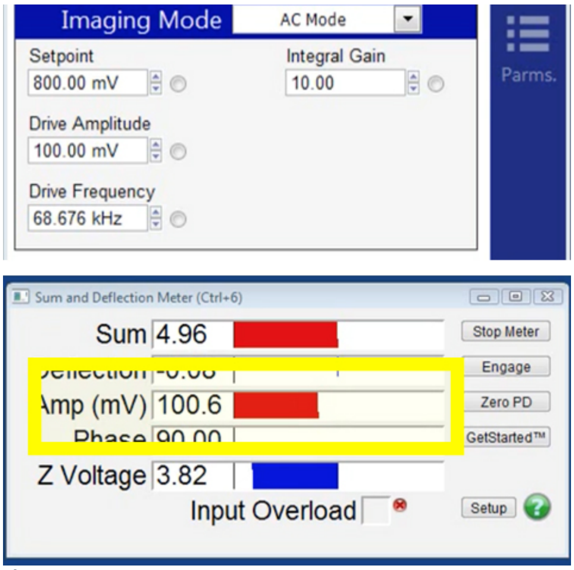


Figure 14

9. The phase should be 90 degrees so adjust the phase to 90 degrees and zero the PD (Figure 15).

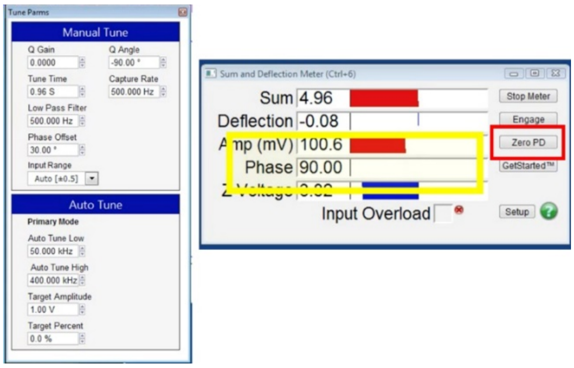


Figure 15

10. Then approach the cantilever by clicking on the "Tip Approaching" button on the engage panel (Figure 16).

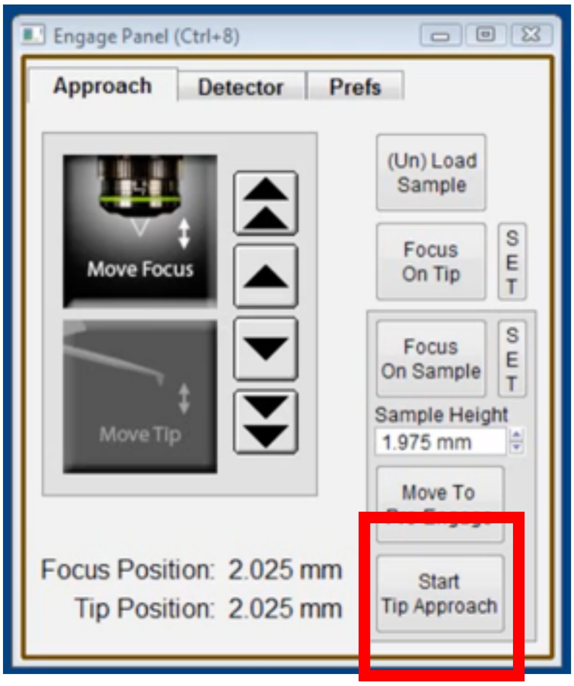


Figure 16

Checking tip status - Critical amplitude method

As a key trait of dynamic AFM, there is a minimum (or critical) free amplitude A_c for the cantilever to transition abruptly from the attractive to the repulsive force regimes for a given set-up[1-5]. An example has been shown in Figure 17. The cantilever is in the repulsive regime when intermittent mechanical contact between the tip and sample prevails. While it is in the attractive regime, when the tip–sample interaction mainly involves non-contact. In Amplitude Phase Displacement (APD) curves (Figure 18), the amplitude, phase lag, and deflection are recorded. When the phase of a free oscillating cantilever lies above 90°, the average force is attractive (attractive regime) and when it lies below 90°, the average force is repulsive (repulsive regime). This is a rule of thumb method to distinguish between intermittent and non-intermittent mechanical contact that was established at the end of the 90s and beginning of the first decade of the century by several groups. For details see the work by Garcia *et al*.[2] For small free amplitudes, the attractive regime prevails for all perturbed amplitudes. As the (free) amplitude is increased, there is a range of values of free amplitudes for which bi-stability is observed.

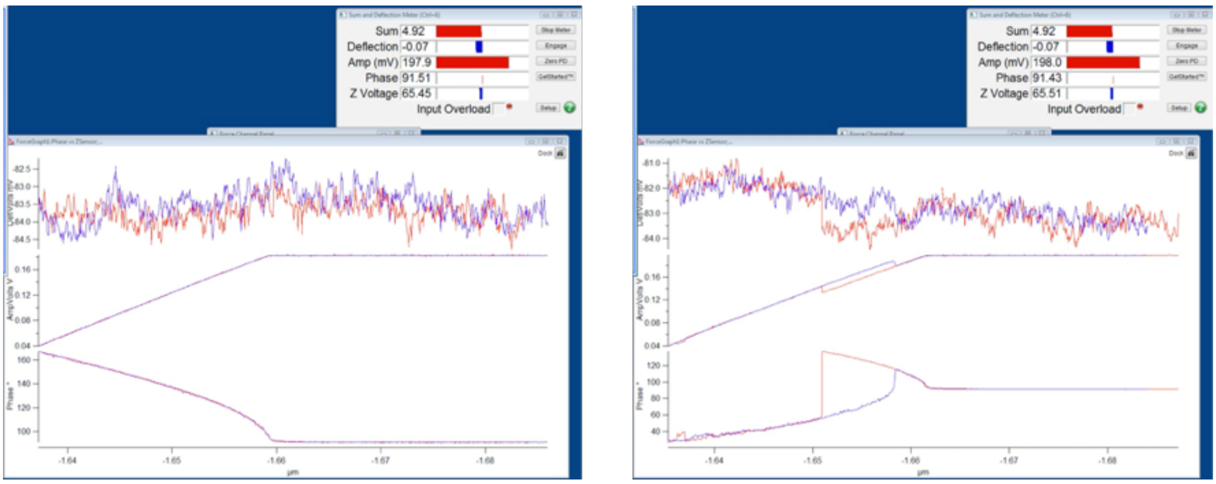


Figure 5: The Critical Amplitude of the tip.

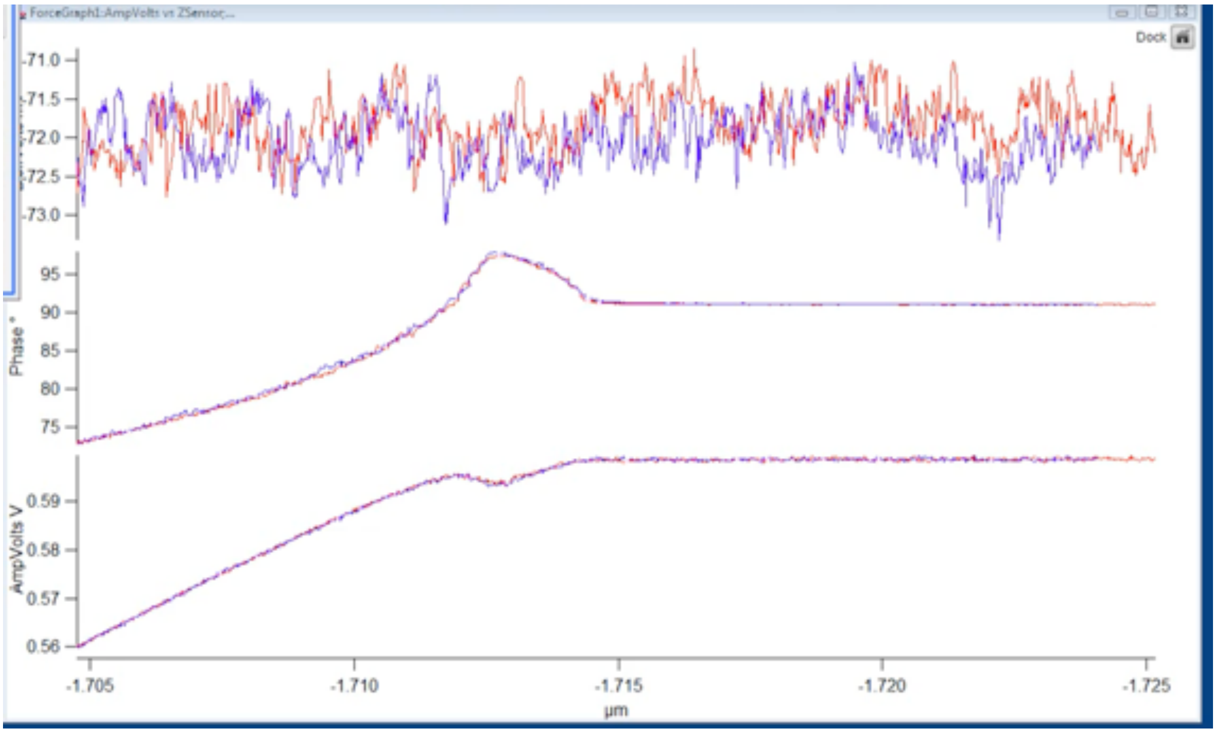


Figure 6: The APD curve.

Bi-stability is a sharp stochastic transition from attractive to repulsive regimes (Figure 19). The range of free amplitudes where this abrupt jump occur is defined as the range of critical amplitude. The critical amplitude is the minimum value of free amplitude (A) required to transit from the attractive to the repulsive regime at resonance[6-16]. The critical amplitude depends on cantilever–sample properties and can only be defined as a range. The magnitude of the chosen free amplitude determines the regime. So, to image in the attractive regime set the free below the critical amplitude usually

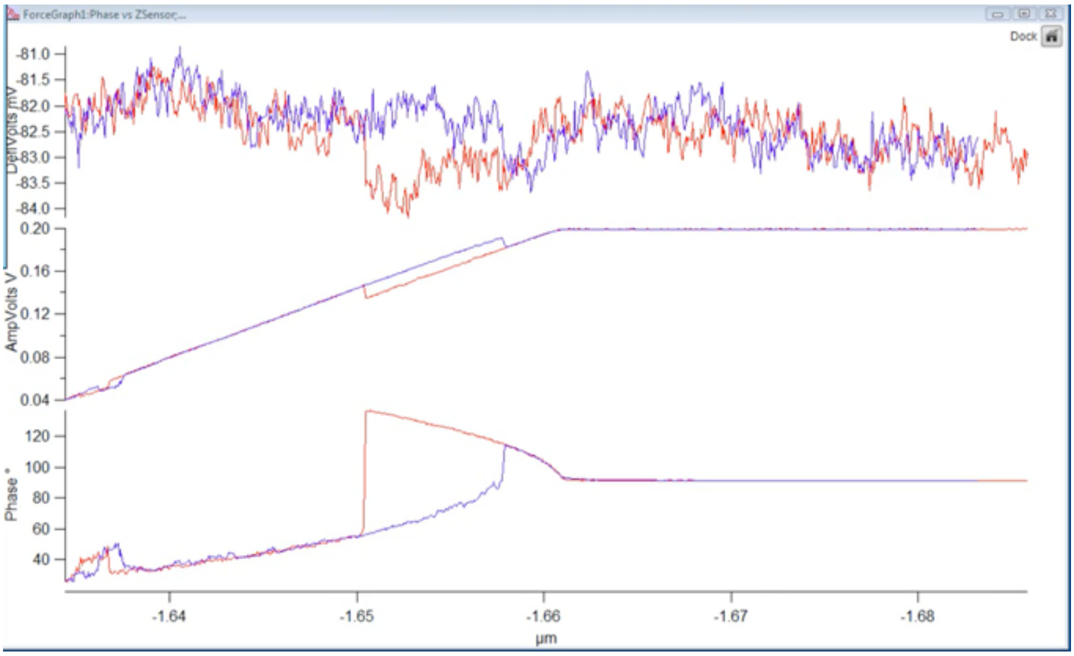


Figure 7: Bi-stability curve.

about half of the critical amplitude[8,10]. To image in the repulsive regime, increase the free amplitude above the critical amplitude[7]. In this case, you can double the value of the critical amplitude. To find the critical amplitude, a force – distance curve rather than imaging needs to be plotted. Force plot is used to find the binding forces between the tip and the sample while imaging is use to determine the topography of the sample. Note that as the tip wears away the critical amplitude will increase and it might happen that you are imaging in the repulsive regime and eventually a transition to the attractive regime occurs during imaging. This is because the tip radius has increased. You will have to increase A_0 in order to image in the repulsive regime. See for example Ref. 4.

Steps to find the critical amplitude

1. Click on the master panel and click on the force tab panel (Figure 20).

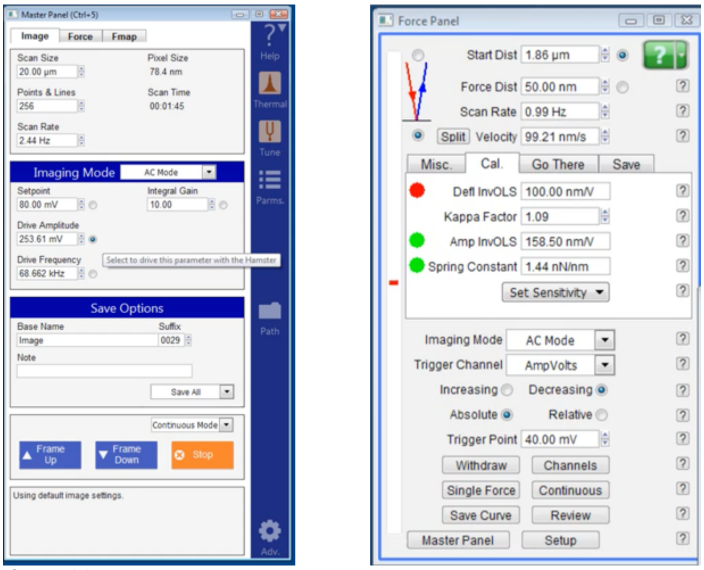


Figure 20

2. Set the cantilever amplitude to about 100 millivolts and set point 80 milli-volts.

3. Click on the engage button. And now set the force distance to about 30nm (Figure 21).

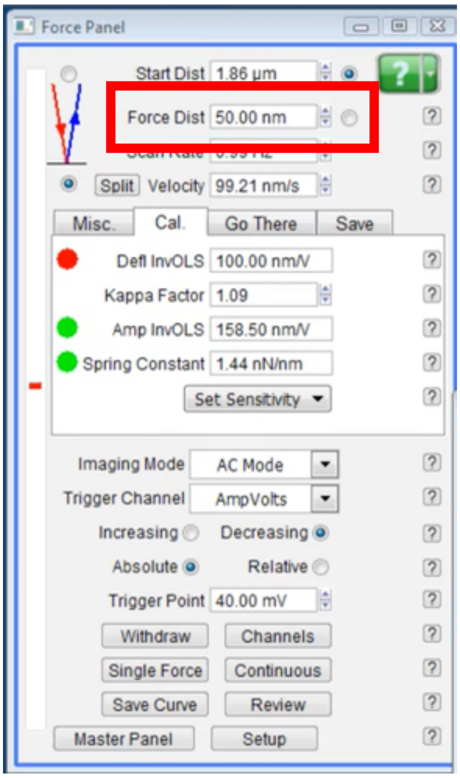


Figure 21

4. The force-distance is how long the force- distance cycle will be, provided a trigger point isn't arrived at before that. The trigger point is defined as the

minimum value of amplitude that is reached before the tip is retracted while recording APD curves. Set the trigger point to 30 millivolts (Figure 22).

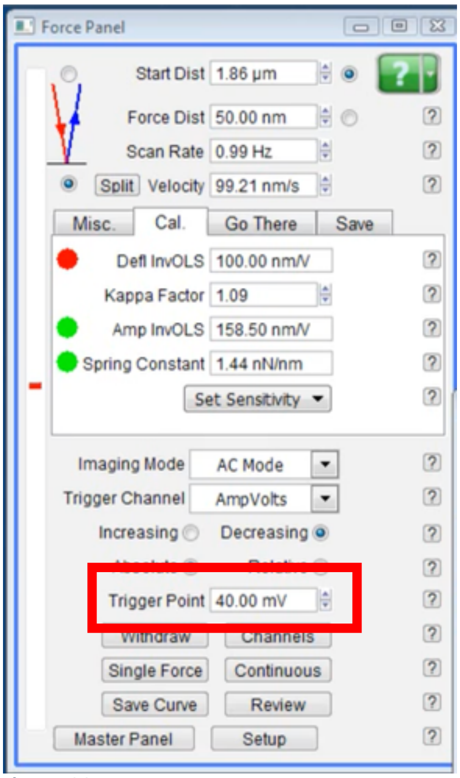


Figure 22

5. Move the mouse to the white bar on the left of the master panel and Shift right click. This help sets the start distance to be the force distance above the current Z position (Figure 23).

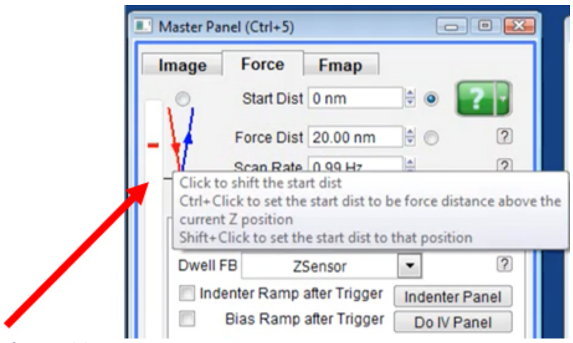


Figure 23

6. Now click on the single force tab (Figure 24).

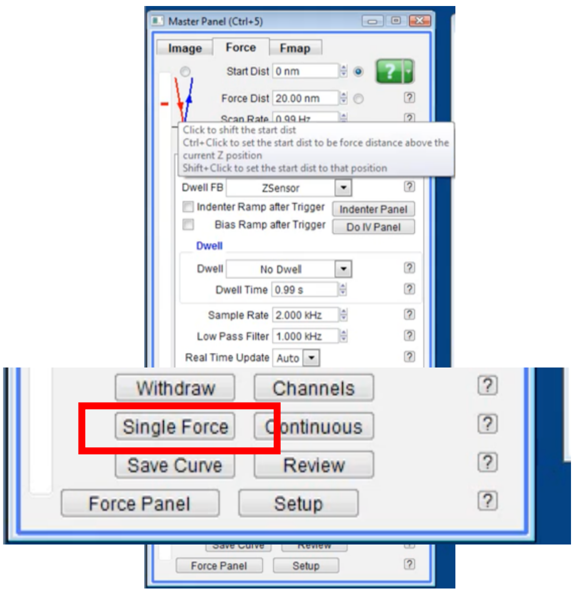


Figure 24

7. Keep increasing the free amplitude gradually and repeating the force plot until the abrupt jump is observed. As explained earlier, bi-stability exist in a range of free amplitude, and the minimum amplitude it occurs is the critical amplitude.

Examples of force curves and their interpretation in the literature

The attractive and the repulsive regimes and bi-stability

In the paper "A method to provide rapid in situ determination of tip radius in dynamic atomic force microscopy"[4], we showed (Figure 8) examples of phase (top) and amplitude (bottom) curves taken on a mica sample with the Cypher AFM.

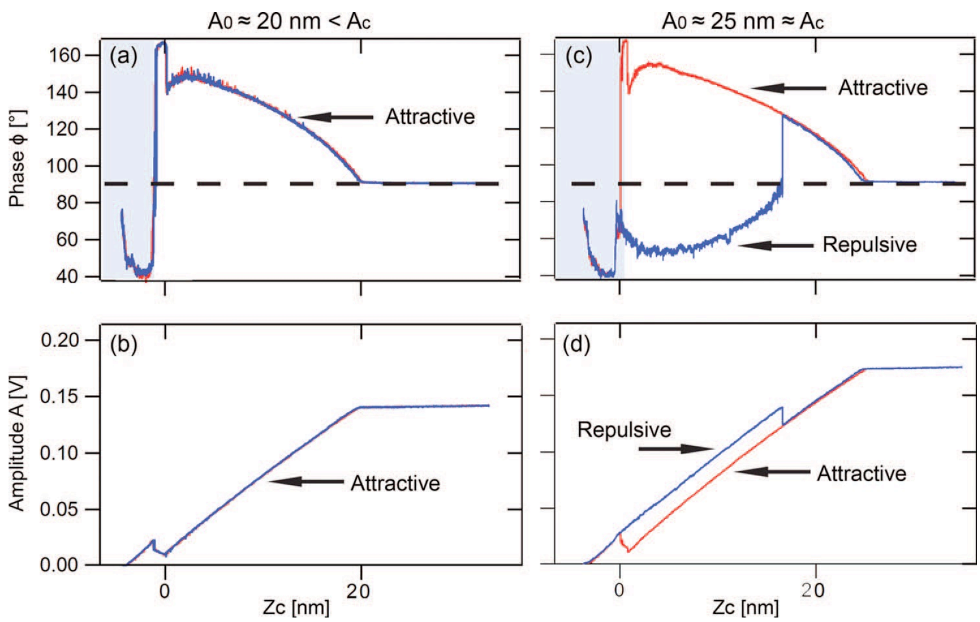


Figure 8: Experimental APD curves (a) and (b) below Ac and (c) and (d) at Ac where bi-stability is present. (a) and (b) Below Ac the attractive regime prevails throughout both on approach (red) and retraction (blue). This can be deduced by observing that at other than A/A0_1 (light blue colored region) the phase always lies above 90◦. (c) and (d) As the free amplitude reaches Ac, a discrete transition between force regimes is observed. Note that the phase lies below 90◦ in some regions of the curve (and at other than A/A0_1). At this point two (c) phase and (d) amplitude branches co-exist for a given separation z_c. The co-existence of these two branches implies that the system displays bi-stability. Experimental parameters: cantilever model AC240TS, f0 = f ≈ 70 kHz, Q ≈ 150, and k ≈ 2 N/m. For (a) and (b) A0 ≈ 20 nm while for (c) and (d) A0 ≈ 25 nm. The sample is aluminum (Al).

The relationship between the tip radius and the attractive and repulsive regimes

As the tip radius increases, for example by scanning the surface, the critical amplitude A_c increases with it. The user should be thus aware that this parameter A_c might change during experiments. In fact, A_c can be used to monitor the stability of the tip radius. If it does not change, typically it means the tip has not changed. Again, this is discussed in Ref. 4. An example of tip variation is given in Figure 9. In particular, we see in the figure how the critical amplitude for the same cantilever-surface system varies with time. The tip-surface system is therefore varying with time. This means any attempt to scan the sample should take into consideration that the tip is not a constant parameter. The fact that the A_c observable changes with it can be exploited to monitor the changes.

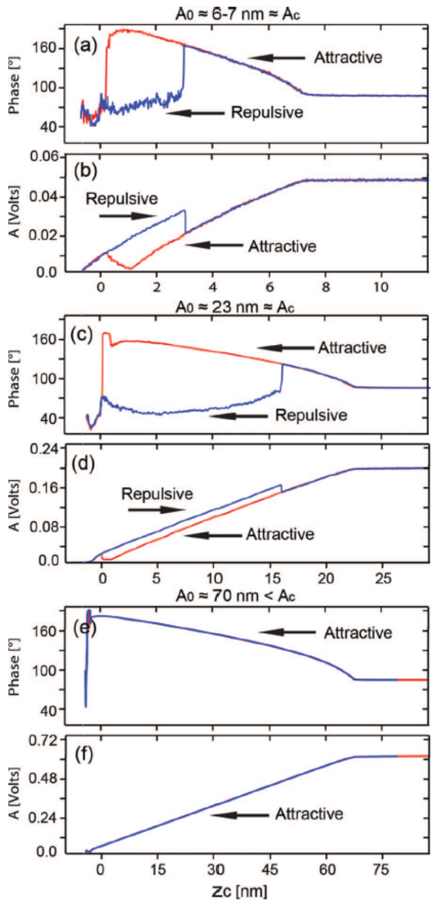


Figure 9: Sequence of APD curves obtained on a mica sample with an AC160TS cantilever where the corresponding amplitudes ((a), (c), (e)) and phases ((b), (d), (f)) are shown. The approach and retractions curves are shown in red and blue, respectively. The attractive and the repulsive regimes, and force transitions in the curves can be readily distinguished in both the amplitude and phase curves. These occur at different tip-sample separations during approach and retraction. Furthermore, step-like jumps in both amplitude and phase are observed. These are also a characteristic of bi-stability that can be used to establish if force transitions have occurred. The experimental parameters are: cantilever model AC160TS, f0 = f ≈ 300 kHz, Q ≈ 500, and k ≈ 40 N/m. The sample is mica.

Bibliography

1 Garcia, R. & San Paulo, A. Amplitude curves and operating regimes in dynamic atomic force microscopy. *Ultramicroscopy* **82**, 79-83 (2000).

2 Garcia, R. & San Paulo, A. Attractive and repulsive tip-sample interaction regimes in tapping-mode atomic force microscopy. *Physical Review B* **60**, 4961 (1999).

3 Ramos, J. Tip radius preservation for high resolution imaging in amplitude modulation atomic force microscopy. *Appl. Phys. Lett.* **105**, 043111-043114 (2014).

4 Santos, S. *et al.* A method to provide rapid in situ determination of tip radius in dynamic atomic force microscopy. *Review of Scientific Instruments* **83**, 043707, doi:10.1063/1.4704376 (2012).

5 Maragliano, C., Glia, A., Stefancich, M. & Chiesa, M. Effective AFM cantilever tip size: methods for in-situ determination. *Meas. Sci. Technol.* **26**, 015002 (2014).

6 Santos, S. *et al.* The Mendeleev–Meyer force project. *Nanoscale* **8**, 17400-17406, doi:10.1039/C6NR06094C (2016).

7 Lai, C.-Y., Santos, S. & Chiesa, M. Systematic Multidimensional Quantification of Nanoscale Systems From Bimodal Atomic Force Microscopy Data. *ACS nano* **10**, 6265-6272, doi:10.1021/acsnano.6b02455 (2016).

8 Lai, C.-Y., Perri, S., Santos, S., Garcia, R. & Chiesa, M. Rapid quantitative chemical mapping of surfaces with sub-2nm resolution. *Nanoscale*, doi:10.1039/C6NR00496B (2016).

9 Garlisi, C., Scandura, G., Palmisano, G., Chiesa, M. & Lai, C.-Y. Integrated Nano- and Macroscale Investigation of Photoinduced Hydrophilicity in TiO2 Thin Films. *Langmuir : the ACS journal of surfaces and colloids* **32**, 11813-11818, doi:10.1021/acs.langmuir.6b03756 (2016).

10 Chia-Yun, L., Sergio, S. & Matteo, C. Reconstruction of height of sub-nanometer steps with bimodal atomic force microscopy. *Nanotechnology* **27**, 075701 (2016).

11 Lai, C.-Y., Olukan, T., Santos, S., Al Ghaferi, A. & Chiesa, M. The power laws of nanoscale forces under ambient conditions. *Chem. Commun.* **51**, 17619-17622, doi:10.1039/C5CC05755H (2015).

12 Lai, C.-Y., Cozzolino, M., Diamanti, M. V., Al Hassan, S. & Chiesa, M. Underlying Mechanism of Time Dependent Surface Properties of Calcite (CaCO3): A Baseline for Investigations of Reservoirs Wettability. *The Journal of Physical Chemistry C* **119**, 29038-29043, doi:10.1021/acs.jpcc.5b10595 (2015).

13 Chang, Y.-H. *et al.* Establishing Nanoscale Heterogeneity with Nanoscale Force Measurements. *The Journal of Physical Chemistry C* **119**, 18267-18277, doi:10.1021/acs.jpcc.5b04456 (2015).

14 Plummer, A., Tang, T.-C., Lai, C.-Y. & Chiesa, M. Nanoscale Hydrophilicity Studies of Gulf Parrotfish (Scarus persicus) Scales. *ACS Applied Materials & Interfaces* **6**, 16320-16326, doi:10.1021/am504568w (2014).

15 Lai, C.-Y. *et al.* A nanoscopic approach to studying evolution in graphene wettability. *Carbon* **80**, 784-792, doi:http://dx.doi.org/10.1016/j.carbon.2014.09.034 (2014).

16 Amadei, C. A., Lai, C.-Y., Heskes, D. & Chiesa, M. Time dependent wettability of graphite upon ambient exposure: The role of water adsorption. *The Journal of*

chemical physics **141**, 084709, doi:doi:http://dx.doi.org/10.1063/1.4893711 (2014).