# Atomic Force Microscopy

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# Abstract

In this experiment, you will understand how to use an AFM and how to use it to do research on different types of data storage and their information density.

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#### 1 Introduction

An atomic force microscope (AFM) is a type of scanning probe microscope (SPM). SPMs use a physical probe that scans the surface of a sample to create an image of it. They differ in the physical quantity that is measured to generate the image of the sample, e.g. the atomic force, the tunneling current between probe and sample, the capacitance, the thermal conductivity etc.

An AFM measures the attractive and repulsive forces from the atoms of the sample, usually described by the Lennard-Jones potential. Because the forces change with the distance between sample and probe, we can use them to generate a height map of the sample by scanning the sample line by line with the probe.

To sense the forces, a probe consisting of a tip at the end of a thin needle (called the cantilever) is used. The cantilever itself is connected to a carrier chip. The cantilever bends depending on the forces that act on the atoms in the tip. A typical probe has a length of only  $\sim 4 \,\mathrm{mm}$ , with a cantilever length of  $\sim 100 \,\mu\text{m}$  and a tip length of  $\sim 15 \,\mu\text{m}$ .



Figure 1: Probe for AFM: It consists of a tip on a cantilever which is connected to the carrier chip. [1]

To measure the amount of bend of the cantilever, different techniques can be ap-**12** plied:

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- **Piezoresistive detection:** the strain changes the resistance of the cantilever; compact but noisy due to heat.
- **Piezoelectric detection:** the strain induces an electrical polarization; high resolution but limited when leak currents occur.
- Interferometric detection: the cantilever and the exit of a glass fiber close to it form an interferometer that is used by the laser light from the fiber; high resolution but difficult to align.
- Optical beam deflection detection: the bend changes the reflection direction of a laser beam; easy but interference between sample and cantilever reflections can occur.



Figure 2: Optical beam deflection detection: Laser light reflects in different angles depending on the amount of bend of the cantilever.

However, instead of *calculating* the forces and thereby the distance to the sample from the bend of the cantilever, it is much easier to *keep* the bend constant during the scan by adjusting the cantilever's vertical position and monitoring this value. This means the cantilever *follows* the topography of the sample by keeping the distance between tip and sample constant.

In order to keep the distance between sample and tip constant, a PID controller is used. A PID controller is a feedback mechanism to drive a system towards a desired setpoint (here the constant bend of the cantilever) depending on the current position of the system and its past evolution in time. An AFM can operate in two different modes:

- **Contact Mode:** The tip is so close to the sample that it touches it. The cantilever bends from the repulsive forces of the sample.
- Dynamic/Tapping Mode: The tip does not touch the sample (or only in the low point of its oscillation) and the experienced forces are too weak to be measured immediately. Instead, a piezo drives the cantilever to oscillate at its resonance frequency. Even weak atomic forces can dampen the amplitude of the oscillation and induce a phase shift which both can be measured using a lock-in amplifier.

# 2 Setup

### 2.1 Overview

In this experiment, we use an AFM manufactured by Anfatec. The microscope consists of a body that hangs vibration isolated above the base plate, and a head mounted on top of the body (see Fig. 3 and 4).



Figure 3: The Anfatec AFM. The heavy body with the sample stage hangs vibration isolated above a stone plate. The head with the probe sits on top of the body. [2]

They contain the following parts:

- Base plate:
  - wiring

- Body:
  - weights against vibration
  - magnetic sample stage
  - 2x manual positioners for coarse x & y of sample stage before the scan
  - 3x stepper motors with screw heads at the top for coarse z & angle of the cantilever to approach the sample
  - 2x piezo motors for x & y of sample stage during scan
  - 1x PID-controlled piezo motor for z of sample stage during scan
- Head
  - probe
  - laser
  - -4x photodetectors
  - driving piezo for dynamic mode that drives the cantilever's oscillation through sound waves (also called 'actuator')
  - camera

Note that the head does not contain any motors and that the cantilever cannot move within the head! The only exception is the driving piezo that drives the oscillation of the cantilever in dynamic mode.



Figure 4: Simplified side view of the Anfatec AFM. The x & y adjusters and piezos are not depicted.

### 2.2 Optical Beam Deflection Detection

Fig. 4 also shows the optical path of the detection setup: A laser beam hits the upper side of the cantilever and the light is reflected onto an array of four photodetectors. The mirrors are used to align the incidencent laser beam onto the cantilever and

the reflected beam onto the center of the array. The photodetectors measure the position of the deflected optical beam. Because the laser beam is similarly large compared to the detection areas of the diodes, the beam can be partially detected by all photodetectors at the same time. The intensity relation between the detectors is a measure of deflection ( $I_{top} - I_{bottom}$ , in the software T-B) and torsion ( $I_{left} - I_{right}$ , in the software L-R) of the cantilever. The total intensity ( $I_{top} + I_{bottom} + I_{left} + I_{right}$ ) is a measure of how well the system is aligned. It relates to how well the laser beam hits the cantilever and its reflection the center of the detector.



Figure 5: Optical beam deflection detector. Four photodetectors in a square-arrangement measure the intensity of incoming light. The laser spot diameter has a similar size as the photodetectors so the differences between the detector signals can be used as a measure of the laser spots position. [2]

### 2.3 PID Controller

In the AFM, a PID controller is necessary to keep the distance between probe and sample constant. A proportional-integral-derivative (PID) controller is a control loop mechanism. It reads a sensor, calculates the difference compared to a desired target value or "setpoint" and computes an output that can be used to bring the system closer to the setpoint the next time. The computation depends on three parameters that depend on the absolute value of the error (proportional parameter), the changing rate of the error (derivative parameter) and the accumulation of the error over time (integral parameter). After the PID has computed the output, it repeats the process.

In our case, the PID controller first reads the signal I from the detector (either T-B or L-R) and calculates the error

$$\Delta_{\text{new}} = I - S, \qquad (1)$$

where S is the given setpoint of how much deflection should actually occur. From there, it computes the output

$$z_{\rm new} = z_{\rm old} + K_{\rm i}\Delta_{\rm new} + K_{\rm p}(\Delta_{\rm new} - \Delta_{\rm old}),$$
(2)

where  $z_{\text{old}}$  is the old height of the sample stage, where  $K_i$  and  $K_p$  are parameters that need to be optimised manually for a good performance of the PID controller (Note that the manufacturer has messed up the nomenclature for the parameters:  $K_i$  actually is the proportional parameter,  $K_p$  is the derivative, and the integral parameter is missing). Usually,  $K_p$  has only very little influence and therefore can be left at 0, although Anfatec suggests  $K_p = 3K_i$ .

The output  $z_{\text{new}}$  is then forwarded to the z-piezo motor below the sample stage. By changing the distance between the cantilever and the sample, the bend and therefore also the amount of deflection will be different next time, ideally closer to the setpoint. Also,  $\Delta_{\text{new}}$  will become  $\Delta_{\text{old}}$  for the next iteration.

With the right parameters, the z data of the PID controller corresponds to the topography of the sample. If the parameters are to low, the PID controller does not react fast enough to height changes in the sample and the image will look blurry. If the parameters are to high the PID controller will overshoot the target values several times before approaching them and the image will show ripples at sharp edges on the sample

In the software, the setpoint (there called 'Ref.'),  $K_i$  and  $K_p$  can be changed in the window 'Parameters ncAFM R'.

### 2.4 Approach & Setpoint

Before we can take an image of the sample, the tip needs to approach the sample safely. Anfatec offers the option of 'auto-approach', where the AFM automatically approaches and stops the tip in front of the sample. However, it needs a given condition on when exactly to stop. Because this position is the same as the setpoint of our PID controller later during the scan, we must choose our setpoint already before the auto-approach.

In **contact mode**, the setpoint directly relates to the T-B value from the photodetectors, thus the desired amount of bend of the cantilever. It should be slightly larger than the T-B value shown in the window 'Crosshairs' when the tip is still far away from the sample (so not bended yet). A good start can be 100 mV above the start value (slight bend, the tip is very close to the sample), but up to 600 mV (strong bend, the tip is pushed into the sample) can be possible. Start low to not break the cantilever.

In **dynamic mode**, the setpoint is a percentage. If the tip is close to the sample, the atomic forces will dampen the oscillation at the cantilever and the amplitude at the original resonance frequency will drop (because the resonance frequency is shifted somewhere else). The setpoint defines the lower limit of how low the amplitude is allowed to be compared to the reference amplitude measured far away from the sample. For new cantilevers, the setpoint should be about 80% to keep a large distance to the sample, for old cantilevers it can go down to 40% because the cantilever does not react that strongly anymore. A low value will mean that the cantilever is closer to the sample and thus make better images but also it gets damaged quicker.

To find the cantilever's resonance frequency at which the setpoint is chosen, we need the resonance spectrum of the cantilever. It can be measured in the window 'Dynamic Non-Contact' where you can also specify the amplitude voltage of the driving piezo inside the AFM head. The higher the voltage, the higher the oscillation amplitude of the piezo and thereby also the reaction of the cantilever will be. During the acquisition of the spectrum, the piezo will sweep through all frequencies. For several frequencies, the cantilever will show some weak resonance but there should be one peak that is exceptionally narrow and high. This is the resonance frequency the cantilever was designed for. Very often, it is quite different from the resonance frequency specified by the manufacturer (0% to 20%). After this procedure, you can choose the setpoint and approach the probe.

#### 2.5 Lock-In Amplifier

In the window 'Dynamic Non-Contact', you can also set an input gain. It corresponds to the pre-amplification of the deflection signal before the lock-in amplification in the AFM.

A lock-in amplifier can measure the amplitude of an oscillation with a known frequency that is buried in noise and oscillations with other frequencies. In our case, after having found the resonance frequency of the cantilever, we would like to monitor the change of the cantilever's amplitude and phase at exactly this frequency both during the approach and image acquisition while the cantilever might switch to other frequencies due to atomic forces and the photodetectors measure other noise sources too.

The main principle of a lock-in amplifier is the following: If we multiply two sine waves oscillating around zero with different frequencies (see Fig. 6c), their product will be a more complicated wave that also always oscillates around zero (see Fig. 6d, blue curve). Taking the average of the product values over a longer time (green curve) gives us a value of 0. However, if we multiply two sine waves oscillating around zero at the same frequency (see Fig. 6a), their product will be another sine wave oscillating around a value depending on the phase shift  $\Delta \varphi$  between the two waves! Taking the average of the new sine curve will result in a non-zero value (if we choose the phase shift accordingly).

Hence, a lock-in amplifier needs the following parts:

- 1. Phase shifter
- 2. Multiplier
- 3. Low-pass filter/Integrator

The lock-in amplifier accepts two inputs: the measurement signal with the oscillation of interest  $x(t) = A\cos(\omega_{\rm m}t)$  but also additional noise, and a reference wave m(t) =



Figure 6: Basic lock-in principle. The product of two sine waves with the same frequency (a) is another sine wave with an average y-value depending on their phase relation (b). The product of two sine waves with different frequencies (c) is always a waveform with an average value of 0 (d). [3]

 $B\cos(\omega_{\rm m}t)$  with the same frequency  $\omega_{\rm m}$  (see Fig. 7). We multiply both signals with an analog multiplier device so that each frequency component of the noisy signal is multiplied with the reference wave. After that, we take the average by sending the product through a low-pass filter that blocks the high frequency components. Thus, the output of the lock-in amplifier will be a constant signal  $y_s \propto A \cdot B \cdot \Delta \varphi$  where  $\Delta \varphi$  is the phase difference between the two input oscillations at  $\omega_{\rm m}$ . The output shows only the amplitude of our measured oscillation x(t) and its evolution over time. To maximise the output signal, a phase shifter is inserted between the reference signal input and the multiplier to optimise the phase shift between the two input signals.



Figure 7: Basic lock-in amplifier. An analog multiplier multiplies a noisy input signal with a reference signal of the desired frequency  $\omega_{\rm m}$ . After a low pass filter, only the amplitude of the oscillation  $\omega_{\rm m}$  in the noisy signal will remain as a quasi-DC signal. [4]

For the AFM, the deflection signal from the photodetectors is used as the noisy input signal that contains the oscillation amplitude at the specified resonance frequency. The lock-in amplifier filters this amplitude out and forwards it to the PID controller as signal I.

Because the multiplier of the lock-in amplifier works best when its whole dynamic voltage range is used, it can make sense to amplify the noisy input signal before the lock-in amplifier, even if this amplifies the input noise too! This pre-amplification corresponds to the chosen input gain in the window 'Dynamic Non-Contact'. Ideally, the signal itself already has a high dynamic range and thus does not need to be amplified (gain = 1). In this case, the sensitivity of the lock-in amplifier is very high (due to the comparatively low noise). If the signal has a low dynamic range, it should be preamplified (gain = 10, 100) which will also amplify the noise and thus lower the sensitivity, but still be an improvement compared to leaving the gain at 1. Nevertheless, the input signal also should not be pre-amplified too much to prevent saturation of the multiplier.

Note that the input gain is proportional to the output signal and thereby also to the PID input. When increasing the gain, you might want to decrease  $K_i$  and  $K_p$ .

## 2.6 Topo, Amplitude & Phase Window

During a scan, always three images will be captured the same time, displayed in different windows. The 'Topo' image depicts the z-piezo's height and thus the surface topography of the sample. The 'Amplitude' image shows the oscillation amplitude of the cantilever at its resonance frequency. Finally, the 'Phase' image shows how the phase shift of the oscillating cantilever at its resonance frequency with respect to the driving piezo's phase. In theory, the amplitude and phase images should look much better because they are very sensitive to changes in the surface structure.

You can always change all parameters at any point in time, even during a scan. Hence, to optimise the scan parameters, just start the acquisition and play around with the settings until the image improves. After that, you can acquire the actual image with constant settings.

# 2.7 Oscilloscope

You can use the window 'Oscilloscope' to plot several AFM parameters over time. This can improve the understanding of what is actually happening in the AFM. A good choice is 'Topo' (showing the current topography height which corresponds to the current height of the PID-controlled z-piezo), 'Amplitude' (showing the current amplitude of the oscillation), and 'T-B' (showing the current amount of bend of the cantilever).

#### 2.8 Motions & Length Scales

Using the AFM becomes much easier when you know which motors and components move in which situations, and when you have a intuition for the length scales involved in the experiment.

Sample Alignment: The interesting area of the sample usually has a size of about  $1 \text{ mm}^2$ . Only when the cantilever is retracted(!), use the manual positioners at the side to move the sample stage in the x-y plane until the target area of the sample is directly below the cantilever. You can also see the movement in the camera window. The total range of the manual positioners is 6.3 mm, a  $90^\circ$ -turn corresponds to 75 µm.

Approach/Coarse Move: The stepper motors will rotate and thereby lower the three screws in the body on which the head is positioned. The head will slowly descend. The total range of the stepper motors is several mm, the smallest step is 0.6 µm.

**Setpoint:** To keep the distance between sample and tip constant during the scan, one of the piezo motors below the sample stage can lift and lower the sample accordingly. The total range is  $7.5 \,\mu\text{m}$  (which is larger than the smallest step of the coarse move!), the smallest step is  $0.06 \,\text{nm}$  (= z resolution of the 'Topo' image). The position of the piezo motor is visualized by the position of the tip in the window 'Parameters ncAFM R' (although actually the sample stage is moving!). If the tip is colored red or yellow, the piezo motor is at its limit and cannot reach the setpoint anymore. Hence, you either need to do a manual coarse step to bring the piezo motor back in range (click on one of the arrows in the window 'Coarse Move'), or you change the setpoint to bring the setpoint back in reach. Ideally, the tip is always colored green.

Scan: During the scan, two piezo motors will move the sample in the x-y plane to do the line-by-line scan of the sample. Their to-tal range is  $50 \,\mu\text{m}$ , the smallest step 0.38 nm (= x-y resolution of the image).

## 2.9 Magnetic Force Microscopy

An AFM can also be used to perform magnetic force microscopy (MFM) where an image of the vertical component  $B_z$  of the sample's magnetic field can be captured. By magnetizing the probe, its deflection will now depend on atomic and magnetic forces. To distinguish between the two types of forces, each location on the sample is scanned twice (called 'dynamic 2nd trace mode' or 'fly mode'): First, the tip scans a single line as usual and saves the contour it just followed. Now it switches into 'fly mode': The PID loop is deactivated, the probe retracts to a long distance of 10 nm to 50 nm above the sample and now follows the contours of the line backwards. Because magnetic forces are stronger than atomic, they dominate in the second measurement. Furthermore, since the distance to the sample is constant, the amount of bend from the atomic forces is constant too. Only magnetic forces will now shift the resonance oscillation to different frequencies which can be seen the best when looking at the phase image.

# **3** Procedures

Here, several important operation procedures will be explained step by step. You do not need to read it before starting with the experiment.

## 3.1 Warnings

- Although an AFM can measure many different things, it cannot measure everything! If the height range of the sample is larger than the z-piezo's range of 7.5 µm, the tip will sooner or later crash into the surface because it cannot retract enough. Always do some quick research about your sample before measuring it.
- The sample stage is very fragile because it is only glued to the piezo motors below it. When placing a sample on or removing it from the stage, be very gentle and do not exert vertical forces to its edge to prevent leverage effects.
- One AFM probe costs roughly 20€. Hence, we do not want to damage probes unnecessarily:
  - Always touch the probe with a tweezer but never at its end with the cantilever. The cantilever is damaged once it is touched. Even if it looks okay, damages can have occured to its internal structure.
  - Always be careful when removing or placing the head when the cantilever is mounted. After a scan, retract the probe by at least 300 µm when you want to remove the head. Before placing the head, always make sure that the tip will not touch the sample, especially when you have a new sample with a different thickness! Placing the head on a flat table surface does not damage the cantilever.
  - Stop the manual approach early enough before crashing the cantilever into the sample at full speed. Rather wait a little longer during the auto-approach afterwards.

• To be able to reuse probes, always place the box of the current probe (and no others) next to the AFM so you do not forget where it came from.

# 3.2 Probe Change & Optical Alignment

- 1. Turn of the laser by clicking on the red dot in the window 'Crosshairs'.
- 2. Retract the tip from the sample by right-clicking on the green arrow in the window Parameters and choosing  $100 \,\mu\text{m}$  at least  $3 \times$ . This will move the probe 0.3 mm away from the sample so that it will not be damaged when lifting the head from the body.
- 3. Carefully take the head of the body, flip it over and hold in your weak hand so that the cantilever points towards your strong hand.
- 4. Take a tweezer with your strong hand, grab the carrier chip at its sides and then release the chip by pushing with the thumb of your weak hand onto the spring contraption.
- 5. Check if the old probe is still complete and put it into the according box.
- 6. Grab a new probe at the long sides of the carrier chip in a flat 30° angle so that its pointing towards your hand. If its sticking to the silicon bed, first push the tweezer past the chip into the bed to loosen it and then grab the chip on the way back. Push it in the released spring contraption. The visible area of the carrier chip should be square-shaped.
- 7. Carefully put the head on the light microscope facing downwards. You might need to move the microscope very close to the AFM. Open the window Camera and slightly turn on the transmitted beam of the microscope so that you can barely see the cantilever.
- 8. Turn on the laser and use the screws 'Laser X' and 'Laser Y' of the head to first search for the laser beam on the camera image. Be gentle, they should already be almost correctly aligned.

Then, align the beam to the *center* of the outer third of the cantilever. The intensity shown in the window 'Crosshairs' should be above 5300 for NSC15 and above 2000 for CSC17. If impossible, go to step 6 and push in or pull out the cantilever a little bit. Usually you need to pull it out, especially for very short cantilevers. In that case, the laser doesn't hit the mirror fully anymore and intensity is lost.

- 9. In the window 'Crosshairs', click on the gear and change the gain (the zoom level of the crosshair image, it does not have any effects on any hardware) to ×1. Use the screws 'Detector X' and 'Detector Y' to move the laser spot into the center of the four photodetectors. Repeat this with gain ×10 and ×100. The intensity should always stay above the limit. If not, go back to step 8 and choose a slightly different spot on the cantilever. The reflection becomes worse at the edges of the cantilever.
- 10. Carefully put the head back on the body and check if the intensity and spot location is still correct. If the intensity is too low, go back to step 8. If the spot location is wrong, use the detectors screws for correction while the head remains on the body.

# 3.3 Contact Mode

- 1. Mount a CSC17 cantilever. Align it to T-B = -400 mV. Otherwise, the laser spot will not hit the photodetectors anymore when bending too much.
- 2. In the toolbar, choose 'Options'  $\rightarrow$  'Feedback' and select 'Contact Mode'. Enter the spring constant of the cantilever that is specified on its box.
- 3. Choose a setpoint, preferably start with -300 mV but you can increase it later to possibly up to 200 mV.
- 4. If the tip is more than 0.5 mm away from the sample, you can first do a manual approach which is faster than auto-approach. In the window 'Coarse Move', click on the downwards arrow

to move down the tip by one step. To move across a longer distance, hold Shift and click on the downwards arrow. Stop *early* to not crash into the sample.

- 5. In the window 'Parameters', right-click on the red arrow and select 'Auto-Approach' (later, you can just use left-click to repeat the last operation). As soon as the auto-approach starts, the  $K_i$  value will change to a second value. This value should be 500, otherwise change it. The approach should stop automatically once the setpoint is reached for the first time. If the laser intensity suddenly drops to  $\leq 300$ , you have crashed into the sample and broken the cantilever.
- 6. We will now take the first image. In the window 'Parameters', enter a  $K_i$  of 1000, a range of 20 µm (the x-y scan range and thus the size of the image), a speed of 1 line/sec (how fast the image will be captured), a resolution of 256 pixels, and set x-center, y-center and angle to 0 (this refers to the central location of the scan on the sample). In the main window, click on the green triangle to capture an image
- 7. Improve the quality of the following images by changing the setpoint in steps of 50 mV to 100 mV,  $K_i$  in steps of 500-1000,  $K_p$  in steps of 1000, and the scan speed in steps of 0.5 lines/sec. A good region can be setpoint = 0 mV,  $K_i = 6000, K_p = 1000, 1 \text{ line/sec.}$
- 8. You can move around the sample by retracting the tip and moving the sample with the manual positioners or by entering other start values at 'x-Center' and 'y-Center'. You can also zoom in by reducing the scan range and/or changing the resolution.

## 3.4 Dynamic Mode

1. Mount an NSC15 cantilever. Align it to the center of the crosshair. Do not approach yet.

- 2. In the toolbar, choose 'Options'  $\rightarrow$  'Feedback' and select 'AFM Amplitude R'. Enter the spring constant of the cantilever.
- 3. In the window 'Dynamic Non-Contact', choose a gain of 100 and a driving voltage of 0.1 V.
- 4. Right-click on the spectrum and choose the first option to scan the whole spectral range. Wait for the plot.
- 5. Look at the plot. It features a black line depicting the measured amplitude of the cantilever for different excitation frequencies as well as a grey line depicting the phase shift between the cantilever's and the piezo's oscillation from  $-180^{\circ}$  to  $180^{\circ}$ . If the cantilever is undamaged, the amplitude curve should show several low but only one exceptionally narrow and high peak. This is the resonance oscillation the cantilever was designed for.
- 6. Measure a smaller and better resolved part of the spectrum around the suspected resonance peak by dragging across the x-range of interest with the left mouse button. Repeat this until you can see the peak very clearly.
- 7. Hover above the peak of the amplitude curve. At the bottom right, you can read off the resonance frequency in Hz and the amplitude in mV. The resonance frequency can be very different from the resonance frequency specified by the cantilever's manufacturer (up to 20%). Because the cantilever works best with an oscillation amplitude of 20 nm to 30 nm, where 1 nm corresponds to  $\approx 1 \,\mathrm{mV}$  of deflection, the height of the resonance peak should be roughly 20 mV to 30 mV. If this is not the case, adjust the driving voltage and remeasure the spectrum by clicking on the green triangle. Start with steps of 0.1 V.
- 8. Set a setpoint by left-clicking somewhere into the spectrum. The xcoordinate of your click will set the resonance frequency (shown in the window 'Dynamic Non-Contact' on the left)

that is used in the lock-in amplifier, the y-coordinate is the setpoint as an percentage of the full amplitude at this frequency (shown in the window 'Parameters ncAFM R'). Start with a setpoint of 70 %.

- 9. If the tip is more than 0.5 mm away from the sample, you can first do a manual approach which is faster than auto-approach. In the window 'Coarse Move', click on the downwards arrow to move down the tip by one step. To move across a longer distance, hold Shift and click on the downwards arrow. Stop *early* to not crash into the sample.
- 10. In the window 'Parameters', right-click on the red arrow and select 'Auto-Approach' (later, you can just use left-click to repeat the last operation). As soon as the auto-approach starts, the  $K_i$  value will change to a second value. This value should be 1000, otherwise change it. The approach should stop automatically once the setpoint is reached for the first time. If the laser intensity suddenly drops to  $\leq 300$ , you have crashed into the sample and broken the cantilever. If the auto-approach stops after one z-piezo "cycle" (in the window 'Parameters ncAFM R', the tip moves only once from top to bottom), you have chosen the wrong amplitude peak. Go back to step 4.
- 11. We will now take the first image. In the window Parameters, enter a  $K_i$  of 6000, a range of 20 µm (the x-y scan range and thus the size of the image), a speed of 1 line/sec (how fast the image will be captured), a resolution of 256 pixels, and set x-center, y-center and angle to 0 (this refers to the central location of the scan on the sample). In the main window, click on the green triangle to capture an image
- 12. Observe the range of deflection in the window 'Crosshairs' or 'Oscilloscope'. You might need to change the input gain to optimize the sensitivity of the lock-in amplifier or prevent its saturation. If the deflection is larger than  $\pm 60 \text{ mV}$ , switch to 10; if it is larger than  $\pm 600 \text{ mV}$ , switch to 1. Keep in mind,

that this also influences the PID controller by  $\times 10$  each, so you might want to change  $K_i$  and  $K_p$  too.

- 13. Improve the quality of the following images by changing the setpoint in steps of 10%,  $K_{\rm i}$  in steps of 1000,  $K_{\rm p}$  in steps of 1000, and the scan speed in steps of 0.5 lines/sec. A good region can be setpoint = 50%,  $K_{\rm i}$  = 8000,  $K_{\rm p}$  = 1000, 1 line/sec.
- 14. You can move around the sample by retracting the tip and moving the sample with the manual positioners or by entering other start values at x-Center and y-Center. You can also zoom in by reducing the scan range and/or changing the resolution.

## 3.5 Magnetic Force Microscopy

### COMING SOON.

## **3.6** Measure an F(z)-Curve

With the software, we can generate a plot of the atomic forces in dependence of the distance between sample and probe. For that, we want to start the measurement with the tip as close to the sample as possible, retract it (while the PID loop is disabled) and approach the setpoint again and measure the amount of bend at each location. Because the tip can "stick" to the sample when retracted, the curve will look different for retraction and approach, so both directions should be measured.

- 1. Approach the probe to the setpoint.
- 2. Open the window 'Spectroscopy'.
- 3. At the top left, choose 'X(z)'. This will measure a quantity X in dependence of the distance z to the sample.
- 4. Our quantity of interest is the deflection 'T-B'. Select and display it below 'Aquire'.
- 5. Set coordinates 'X' and 'Y' of data acquisition to 0, Delay1 =  $1000 \,\mu\text{s}$  and AquT =  $20\,000 \,\mu\text{s}$ .

- 6. We now want to plan the measurement route of our probe. Click on the gear symbol and select the tab 'Cycle'. 'Lead In and Out' should be disabled and the cycle be set to 'full'. Below, you can see the planned route. The probe will start at its current location ('now'), then go to position 1, start the measurement, then 2 (red line!), then 1 again (blue line!), stop the measurement and finally back to the start. Close the settings.
- 7. 'dz1' and 'dz2' correspond to positions 1 and 2 *relative* to the current location (= 0). Positive numbers are higher than the current location, negative numbers lower and thus closer (!) to the sample. Set the following route:
  - In contact mode: The probe is already at the sample so we do not want to go any closer. Hence, we start the measurement at the current location (dz1 = 0 nm) and then go far away (dz2 = 200 nm).
  - In dynamic mode: Currently, the probe is sensing the atomic forces from a distance, so we need to get closer to measure a better curve. Hence, we start the measurement from a closer position (dz1 = -200 nm) and then go far away (dz2 = 200 nm).
- 8. Click on 'Pre' to check the planned route. The horizontal grey line is the current position, the sample is located towards the bottom.
- 9. Click on the green triangle to measure. In the window 'Oscilloscope', 'Topo' should follow exactly the planned route (since the z-piezo is moving). After a few seconds, plotted data shows up. The red line is the deflection measured on the way from position 1 to position 2, the blue line is the deflection measured on the way back.
- 10. Play around with the setpoint (which marks the start point), 'dz1' and 'dz2'. You can use the 'Topo' value from the oscilloscope and the deflection shown in the window 'Crosshairs' to get a sense

of how close your probe is to the sample and over what distances you can move it. Be careful to not crash the probe!

# 4 Task & Report

You have free choice in designing the experiments for your report. Make sure to save all important measurements. The report should answer or at least give insight on each of the following questions (you can choose the order by yourself):

- How does an AFM work? How do its two modes differ?
- What 'atomic forces' exactly does an AFM measure? Measure and analyze an F(z) curve with respect to the expected forces. Why can the tip "stick" to the sample?
- How does an AFM image typically look like?
- Which important parameters influence an AFM image in what way? How do the images usually look like in each case? Focus on setpoint,  $K_i$ ,  $K_p$ , scan speed & driving voltage.
- How does MFM work?
- How do a vinyl, a cassette tape, a CD and a hard drive store data?
- What are the four types of data storage made out of and how does it translate to their visible surface structure?
- How many minutes of audio can you roughly store on the five types of data storage based on your images and some estimation? You can and will need to make a few additional assumptions. Ignore compression. Compare the result with a speech printed on paper.
- What bit string can be seen on your image of the CD? Choose one line of your image and translate it to bits.
- Why can a bit string not directly be imprinted on a CD but needs some kind of encoding? Present three ideas on how data could be encoded on a CD and calculate their efficiency.

- The length should be 10 to 12 pages • (without title page, references, appendix etc.). Each question should be answered in less than 1.5 pages. The language should be English or German.
- Only introduce/explain what you need to answer the questions. For most questions, no theoretical depth is needed.
- The target audience of your report should be other students of your semester, who know nothing about AFMs (but also don't want to know everything). They are not interested in the specific operation of the Anfatec AFM and its software but still would like to know how to acquire good im-They also do not ages with AFMs. know much about data storage and are interested in how the different types work, look like and how their information densities compare.

#### $\mathbf{5}$ Troubleshooting

I changed a value but nothing hap**pens!** If that input window is grey the value has not been accepted yet. Click again into the window and press 'Enter' to apply the new settings.

I am far away from getting a laser intensity of 5000 with my alignment. If the cantilever is at the wrong position, you will align your beam over the edge of the second mirror so that the amount of light is limited by the mirror's edge. Reposition the cantilever (very likely further away from the holder).

My topography image has lots of black and/or yellow regions. This means that the z-piezo below the sample stage would like to move the sample further/closer but is at the end of its range. In the window 'Coarse Move', move the tip further/closer until it is colored green and located in the center again. This will move the z-piezo back into its central range.

For the report, keep the following in mind: I am confused, does the PID controller move the tip or the sample?? The zpiezo controlled by the PID controller is located *below* the sample stage and thus moves the sample. The tip cannot move, only the whole head during the approach using the stepper motors. However, the visualization in the window 'Parameter ncAFM R' shows the tip moving. It is supposed to go well with the intuition of how you would expect the AFM to work but it is also misleading.

> My image looks extremely flat, like a section of just one huge cylinder or sphere. The PID controller might be too slow to follow the topography of the sample and therefore stays more or less at the same position. Increase  $K_i$  to 8000. If you are in dynamic mode, decrease the setpoint to 40% to check if the tip is just too far away from the sample to feel details. Slow down the scan speed to give the tip more time to follow the surface contours. If you extend the scan range to see if you can zoom out and nothing changes, you are not measuring the sample's surface properly.

> My image shows regular waves along the x-axis. The detectors might measure interference between light coming from the cantilever and from the sample (this happens especially when not all light is reflected at the cantilever but goes partially past by). Try to play around with all parameters or realign the laser spot on the cantilever.

> What are the initial settings of all parameters? In 'Parameters':  $K_i = 1000$ ,  $K_{\rm p} = 0$ , Bias = 0, Active Level = auto, Range =  $20 \,\mu\text{m}$ , Pixel = 256, x-Center =  $0 \,\mu\text{m}$ , y-Center =  $0 \,\mu\text{m}$ , Angle = 0. In 'Dynamic Non-Contact': Input A Gain = 1. Output A = 0.1 V.

> My surface seems to be tilted. Take the image again. Almost every sample plane will have some kind of tilt but the software will correct the discrepancy betwen sample plane and scanning plane in the next measurement according to the tilt of the previ

ous (complete) image. To check, go to the 'Level' tab in the window 'Parameters' and set it to 'auto'.

The tip oscillates a lot/in an uncontrolled way already before approach. Reduce  $K_i$  to stabilize the feedback. Otherwise increase the driving amplitude to stabilize the cantilever oscillation.

My F(z) measurement shows only noise and/or interference patterns. Make sure that the laser intensity in the detector is high enough. Otherwise, you are probably measuring to far away from the sample where the photo-detection is dominated by noise and interference effects between sample and cantilever reflections. Choose a setpoint that moves the sample closer the probe.

# References

- James Vicary. A pedant's (Christmas) guide to AFM probe terminology. URL: https://www.nunano.com/blog/ 2016/12/12/a-pedants-christmasguide-to-afm-probe-terminology. (accessed: 24.06.2024).
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