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# Atomic Force Microscopy

Instructions to the Advanced Lab course

# WS 2018/2019

Location of practical course:
KIP, INF 227, room 00.222

Web:
`http://www.kip.uni-heidelberg.de/ag_pucci/teaching/afm`

Revised on September 4, 2018, by C. Huck, KIP.

# Preliminary Note

The aim of this practical course experiment is to give the students an insight into the technique of scanning probe microscopy (SPM) with an atomic force microscope (AFM) taken as example. Since this kind of microscopy is widely-used in research areas which deal with structures on the nanometer scale, it might serve as a prototypic example for an up-to-date method in surface science. At the same time the used device is sufficiently simple to be used by students in the frame-set of a lab course without the permanent presence of the supervisor.

The students should be familiar with chapter 1 to 3 of this tutorial *prior* to the beginning of the experiment. They should focus on the underlying principles of the physics of the AFM. In addition to this instructions, a large variety of information about the subject and related methods is available for instance in the world wide web. The list of questions and tasks in the preparation section 2.3 should be answered in advance to the lab course.

Chapter 4 deals with the instrumental details for the lab course and some additional information about problem solving. This chapter **is not** necessary to read in advance.

This instructions are work in progress, so every hint on errors or on possible improvements is appreciated.

# Contents

# 1 Introduction

## 1.1 Scanning Probe Microscopy

The method of scanning probe microscopy (SPM) was developed in 1981 by Binnig and Rohrer with the invention of the scanning tunnelling microscope (STM) [1, 2]. This technique exploits the fact that a tunnelling current through a potential barrier of the width $d$ is proportional to $e^{-d}$ (provided a constant and final height of the potential barrier). With a sharp tip close to a surface, one can measure by applying a voltage between tip and surface the tunnelling current between the tip and the surface atoms in the vicinity of the tip. The strong dependence of the current on the distance $d$ allows one to detect changes in distance on a sub-atomic scale. By moving the tip parallel to the surface, the height of the surface can be scanned. To control such distances, electromechanical devices of high precision are required. This task is commonly solved by piezo-ceramics. For an understanding of the images generated this way one has to take into account that the tunnelling current is also a function of the chemical properties of the surface. For a more detailed description and discussion of STM see e.g. [3, 4].

The scanning tunnelling microscope is (in general) restricted to conducting materials. The surfaces of insulators, structures in liquids and biological samples can be imaged non destructive with high resolution by the atomic force microscope (AFM) (also often scanning force microscope (SFM)), which was developed in 1986 by Binnig, Quate and Gerber [5].

## 1.2 The Scanning Force Microscope

*This subsection is mainly taken from [6] and slightly adapted.*

The design and development of the atomic force microscope are very closely connected to those of the scanning tunnelling microscope. The central component of these microscopes is basically the same. It is a fine tip positioned at a characteristic small distance from the sample. The height of the tip above the sample is adjusted by piezoelectric elements. In STM the tunneling current gives the information about the surface properties, whereas in AFM the forces between the tip and the surface are used to gain this information. The images are taken by scanning the sample relative to the probing tip and measuring the deflection of the cantilever as a function of lateral position. The height deflection is measured by optical techniques, which will be described in more detail later.

A rich variety of forces can be sensed by scanning force microscopy. In the non-contact mode (of distances greater than 1 nm between the tip and the sample surface), van der Waals, electrostatic, magnetic or capillary forces produce images, whereas in the contact mode, repulsion forces take the leading role. Because its operation does not require a current between the sample surface and the tip, the AFM can move into potential regions inaccessible to the STM, for example samples which would be damaged irreparably by the STM tunnelling current. Insulators, organic materials, biological macromolecules, polymers, ceramics and glasses are some of many materials which can be imaged in different environments, such as in liquids, under vacuum, and at low temperatures.

In the non-contact mode one can obtain a surface analysis with a true atomic resolution. However, in this case the sample has to be prepared under ultra-high vacuum (UHV) conditions. Recently, it has been shown that in the tapping mode (a modified non-contact mode) under ambient conditions it is possible to achieve a sufficient resolution to observe, similar as in STM investigations, single vacancies or their agglomeration [7]. Besides this, a non-contact mode has the further advantage over the contact mode that the surface of very soft and rough materials is not influenced by frictional and adhesive forces as during scanning in contact mode, i.e. the surface is not "scratched". Additionally to height profiles, state-of-the-art atomic force microscopy allows for the imaging of nanomechanical properties, such as adhesion or Young's modulus.

# 2 Basics

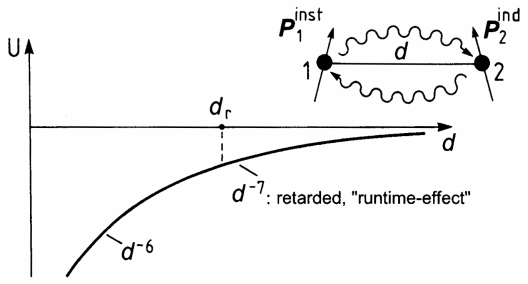



**Figure 2.1:** Van der Waals potential $U$ between two atoms. $d_r$ is the critical distance above which the transit time effects weaken the interaction. (Taken from [6].)

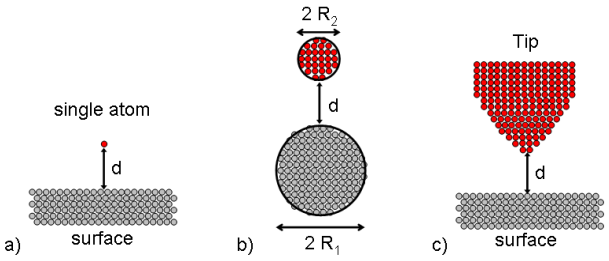



**Figure 2.2:** Schematic illustration of the three described interaction cases in the text: a) sphere with half-space, b) two spheres and c) sphere (Tip) with half-space.

## 2.1 Theoretical Principles

*This section and subsections are mainly taken from [6] and slightly adapted.*

### 2.1.1 Van der Waals interaction

As already mentioned above, van der Waals forces lead to an attractive interaction between the tip on the spring and the sample surface. Figure 2.1 shows schematically the van der Waals potential between two atoms. The potential can be described in a simpler classical picture as the interaction potential between the time dependent dipole moments of the two atoms. Although the centres of gravity of the electronic charge density and the charge of the nucleus are exactly overlapping on a time average, the separation of the centres of gravity is spatially fluctuating in every moment. These statistical fluctuations give rise to an transient dipole moment of the particle while it might be in average unpolarised. The dipole moment of an atom can again induce a dipole moment in the neighbouring atom and the induced dipole moment acts back on the first atom. This creates a dipole-dipole interaction on basis of the fluctuating dipole moments. This interaction decreases with $d^{-6}$ in the case of small distances $d$ (Lenard Jones potential). At larger distances, the interaction potential decreases more rapidly ($d^{-7}$). This arises from the fact that the interaction between dipole moments occurs through the exchange of virtual photons, as indicated in figure 2.1. If the transit time of the virtual photon between atom 1 and 2 is longer than the typical fluctuation time of the instantaneous dipole moment, the virtual photon weakens the interaction. This range of the van der Waals interaction is therefore called retarded, whereas that at short distances is unretarded.

The scanning force microscope is not based on the interaction of individual atoms only. Both the sample and the tip are large in comparison to the distance. In order to obtain their interaction, all forces between the atoms of both bodies need to be integrated. The result of this is known for simple bodies and geometries. In all cases, the summation leads to a weaker decrease of the interaction. Some examples:

- **Single atom over half space**
  A single atom at a distance $d$ relative to the half-space (see figure 2.2 a)) leads to an interaction potential of

$$U = -\frac{C\pi\rho}{6}\frac{1}{d^3} \tag{2.1}$$

  where $C$ is the interaction constant of the van der Waals potential and $\rho$ the density of the solid. $C$ is basically determined by the electronic polarisability of the atoms in the half-space and of the single atom.

- **Two spheres**
  If one has two spheres with radii $R_1$ and $R_2$ at distance $d$ (distance between sphere surfaces, as shown in figure 2.2 b)) one obtains an interaction potential of

$$U = -\frac{HR_1R_2}{6(R_1+R_2)}\frac{1}{d} \tag{2.2}$$

  where $H$ is the so called Hamaker constant. It can be defined for a van der Waals body-body interaction as $H = \pi^2 \times C \times \rho_1 \times \rho_2$ where $\rho_1$ and $\rho_2$ are the number of atoms per unit volume in two interacting bodies and $C$ is the coefficient in the particle-particle pair interaction [8]. It

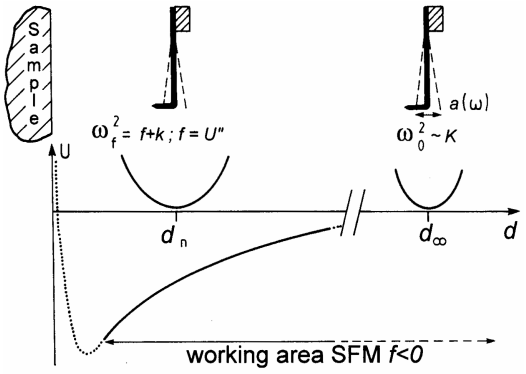



**Figure 2.3:** Schematic representation of the effect of the van der Waals interaction potential on the vibration frequency of the spring with tip. As the tip approaches the surface, the resonance frequency of the leaf spring is shifted. (Taken from [6].)

is material specific and essentially contains the densities of the two bodies and the interaction constant $C$ of the van der Waals potential.

- **Sphere over half-space**
  If a sphere with the radius $R$ has a distance $d$ from a half-space (see figure 2.2 c)), an interaction potential of

$$U = -\frac{HR}{6}\frac{1}{d} \tag{2.3}$$

is obtained from Eq. (2.2). This case describes the geometry in a scanning force microscope best and is most widely used. The distance dependence of the van der Waals potential thus obtained is used analogously to the distance dependence of the tunnel current in a scanning tunnelling microscope to achieve a high resolution of the scanning force microscope. However, since the distance dependence is much weaker, the sensitivity of the scanning force microscope is lower.

### 2.1.2 Approaching the surface (Force distance behavior)

For large distances between the tip and the sample the bending of the cantilever by attractive forces is negligible. After the cantilever is brought closer to the surface of the sample (point "a" Figure 2.4) the van der Waals forces induce a strong deflection of the cantilever which, simultaneously, is moving towards the surface. This increases the forces on the cantilever, which is a kind of positive feedback and brings the cantilever to a direct contact with the sample surface (point "b"). However, when the cantilever is brought even closer in contact to the sample, it actually begins to bend in the opposite direction as a result of a repulsing interaction ("b-c"). In the range ("b-c") the position of the laser beam on two quadrants of the photo diode (see 2.2.1), which is proportional to the force, is a linear function of distance. On reversal this eristic shows a hysteresis. This means that the cantilever loses contact with the surface at a distance (point "d") which is much larger than the distance on approaching the surface (point "a").

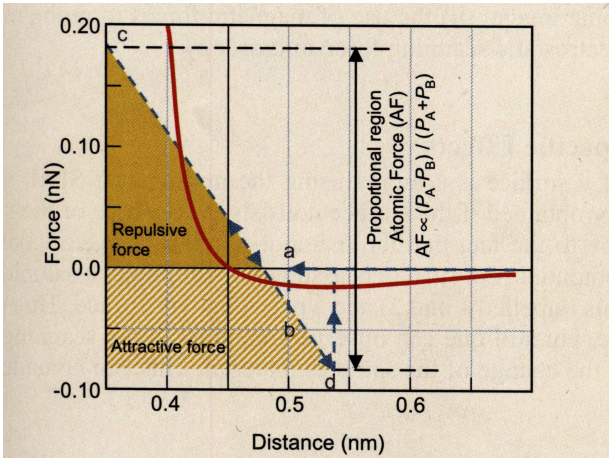



**Figure 2.4:** Cantilever force (dashed line) as a function of the tip–sample distance. (Taken from [6].)

### 2.1.3 Mechanical Surface Properties

In recording the force-distance behavior, one gets information on the force acting on the tip. In order

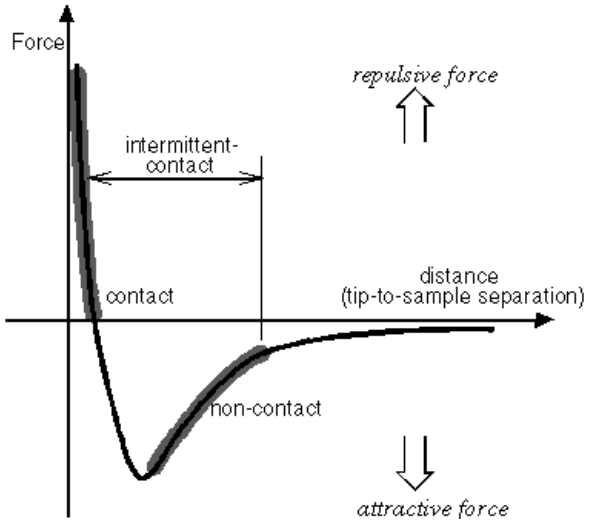



**Figure 2.5:** Force-distance curve. Classification of the AFM operation modes within the working regime of the van der Waals potential.

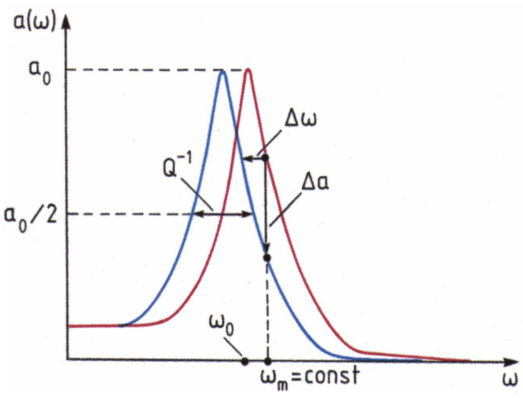



**Figure 2.6:** Resonance curves of the tip without and with a van der Waals potential. The interaction leads to a shift $\Delta\omega$ of the resonance frequency with the consequence that the tip excited with the frequency $\omega_m$ has a vibration amplitude $a(\omega)$ attenuated by $\Delta a$. (Taken from [6].)

to obtain the mechanical properties of the surface, e.g. Young's modulus or adhesion force, the so called DMT (Derjaguin-Muller-Toporov) [9] model, describing the adhesive contact mechanics of touching solids, is used. For the force on the tip it yields:

$$F_{\text{tip}} = F_{\text{adh}} + \frac{4}{3} Y \sqrt{r d^3}. \tag{2.4}$$

With the tip radius $r$ and the indentation $d$ one can calculate (fit) the adhesion force $F_{\text{adh}}$, as well as the reduced Young's modulus $Y$.

### 2.1.4 Operation Modes

Many AFM operation modes have appeared for special purposes during the further development of AFM. These can be classified into static modes and dynamic modes. Another classification could be contact, non-contact and intermittent-contact which is reclined to the working regimes (see figure 2.5).

Here the three commonly used techniques, namely static mode (contact mode) and dynamic mode (non-contact mode, tapping mode and peak force tapping mode) will be shortly described. Further information can be found in textbooks or review articles.

#### Contact mode/static mode

In contact mode the tip scans the sample in close contact with the surface and the force acting on the tip is repulsive of the order of $10^{-9}$N. This scanning mode is very fast, but has the disadvantage that additionally to the normal force frictional forces appear which can be destructive and damage the sample or/and the tip.

**Constant height mode:** This mode is particular suited for very flat samples. The height of the tip is set constant and by scanning the sample only the deflection of the cantilever is detected by the optical system of the AFM (described in 2.2.1) and gives the topographic information.

**Constant force mode:** In this mode the force acting on the cantilever, i.e. the deflection, is set to a certain value and changes in deflection can be used as input for the feedback circuit that moves the scanner up and down. The system is therefore responding to the changes in height by keeping the cantilever deflection constant. The motion of the scanner gives the direct information about the topography of the sample. The scanning time is limited by the response of the feedback circuit and therefore not as fast as the constant height mode.

#### Dynamic Modes

The dynamic operation method of a scanning force microscope has proved to be particularly useful. In this method the normal force constant of the van der Waals potential, i.e. the second derivative of the potential, is exploited. This can be measured by using a vibrating tip (Figure 2.3). If a tip vibrates at a distance $d$, which is outside the interaction range of the van der Waals potential, then the vibration frequency and the amplitude are only determined by the spring constant $k$ of the cantilever. This corresponds to a harmonic potential. When the tip comes into the interaction range of the van der Waals potential, the harmonic potential and the interaction potential are superimposed thus changing the vibration frequency and the amplitude of the spring.

This is described by modifying the spring constant $k$ of the spring by an additional contribution $f$ of the van der Waals potential. As a consequence, the vibration frequency is shifted to lower frequencies as shown in Figure 2.6. $\omega_0$ is the resonance frequency without interaction and $\Delta\omega$ is the frequency shift to lower values. If an excitation frequency of the tip of $\omega_m > \omega_0$ is selected and kept constant, the amplitude of the vibration decreases as the tip approaches the sample, since the interaction becomes increasingly stronger. Thus, the vibration amplitude also becomes a measure for the distance of the tip from the sample surface. If a spring with a low damping $Q^{-1}$ is selected, the resonance curve is steep and the ratio of the amplitude change for a given frequency shift becomes large. In practice, small amplitudes (approx. 1 nm) in comparison to distance $d$ are used to ensure the linearity of the amplitude signal. With a given measurement accuracy of 1 %, however, this means the assembly must measure deflection changes

of 0.001 nm, which is achieved most simply by a laser interferometer or optical lever method.

**Non-contact mode:** Since in the non-contact regime the force between the tip and the surface is of the order of $10^{-12}$ N and therefore much weaker than in the contact regime the tip has to be driven in the dynamic mode, i.e. the tip is vibrated near the surface of the sample and changes in resonance frequency or amplitude are detected and used as input for the feedback circuit. In this case the resonance frequency or the amplitude are held constant by moving the sample up and down and recording directly the topography of the sample.

**Tapping mode:** Tapping mode is an intermittent-contact mode. In general it is similar to the non-contact mode described before, except that the tip is brought closer to the surface and taps the surface at the end of his oscillation. This mode is much more sensitive than the non-contact mode and in contrary to contact mode no frictional forces appear which can alter the tip or the surface. Therefore this is the mode of choice for the most experiments and during the lab course.

**Peak Force Tapping mode:** Like the Tapping mode, the Peak Force Tapping mode is operating in intermittent contact with the sample. In contrast to the tapping mode, the cantilever is oscillated far below its resonance frequency. Force distance curves (described in 2.1.2) are executed at each pixel of the recorded image. The maximum force acting between tip and sample determined from each force curve is used as input for the feedback system. Therefore this maximum force is a directly controllable parameter in this mode. Due to the pixelwise recording of the force curves, this mode enables the imaging of mechanical properties (e.g. adhesion) in addition to the topography of the sample surface.

## 2.2 Operation Principles

### 2.2.1 Optics

The main electronic components of the AFM are the same as for the STM, only the topography of the scanned surface is reconstructed by analysing the deflection of the tip at the end of a spring. Today, the interferometrical and the optical lever method dominate commercial AFM apparatus. The most common method for detecting the deflection of the cantilever is by measuring the position of a reflected laser-beam on a photosensitive detector. The principle of this optical lever method is presented in Figure 2.7. Without cantilever displacement, both quadrants of the photo diode (A and B) have the same irradiation $P_A = P_B = P/2$ ($P$ represents the total light intensity). The change of the irradiated area in the quadrants A and B is a linear function of the displacement.

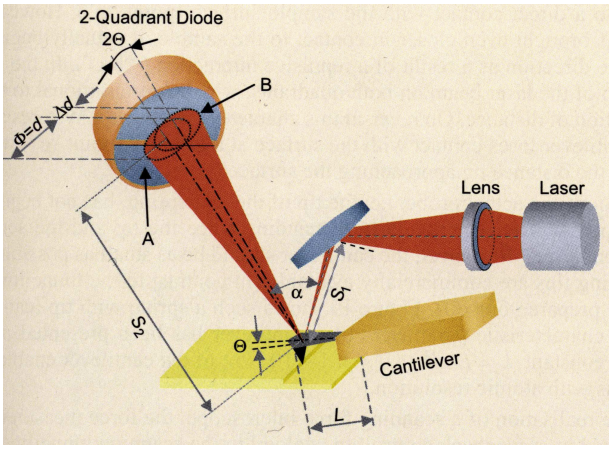



**Figure 2.7:** The amplification of the cantilever motion through the optical lever arm method. Optical laser path in the standard AFM set-up. (Taken from [6].)

Using the simple difference between $P_A$ and $P_B$ gives therefore the necessary information on the deflection but in this case one cannot distinguish between the displacement $\delta$ of the cantilever and the variation in the laser power $P$. Hence the normalised difference is used, which is only dependent of $\delta$:

$$\frac{P_A - P_B}{P_A + P_B} = \delta \frac{3S_2}{Ld}. \tag{2.5}$$

The "lever amplification" $\Delta d/\delta = 3S_2/L$ is about a factor of one thousand. On the basis of this kind of technique one is able to detect changes in the position of a cantilever of the order of 0.001 nm. These changes are used as input signal for the feedback system described below.

### 2.2.2 Feedback control

For the realisation of a scanning force microscope, the force measurement must be supplemented by a feedback control (see figure 2.8), in analogy to the scanning tunnelling microscope. The controller keeps the controlled quantity — which can be deflection, vibrational amplitude or resonance of the cantilever, or peak force, depending on the operation mode — and thus also the distance to the sample, constant. During scanning the feedback controller retracts the sample with the scanner of a piezoelectric ceramic or shifts towards the cantilever until the controlled quantity has reached the setpoint value again. *The scanning*

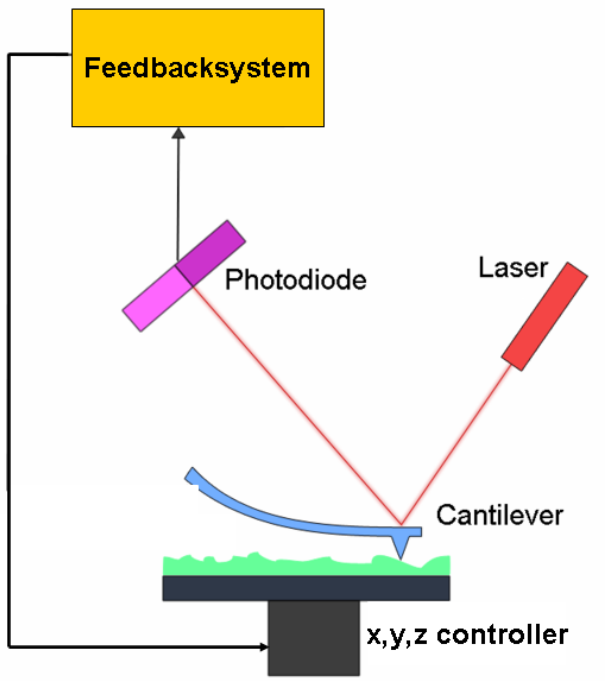



**Figure 2.8:** Schematic illustration of the feedback loop and the optical system.

*force micrographs thus show areas of a constant quantity, which does not necessarily follow the distance to the sample surface.* If the surface is chemically homogeneous and if only the van der Waals forces act on the tip, the AFM image shows the *topography of the surface.*

## 2.3 Preparation Tasks

- What are the underlying principles of AFM? What forces act between tip and sample? What does the corresponding potential look like?
- Briefly describe the experimental setup of the AFM. How is the information on the sample topography obtained?
- *Force distance behavior*: How does the deflection of the tip (proportional to the force) change when it approaches the surface? What difference do you expect for retracting the tip?
- What operation modes can be chosen? Briefly describe the different modes and their advantages/disadvantages.
- During this lab course we will use the tapping mode and the peak force tapping mode. How do these modes work in detail? *Tapping mode*: How is the resonance curve (amplitude vs. frequency) modified when the tip approaches the surface? Which parameter of the oscillation provides information on the height profile when the excitation frequency is kept constant? *Peak force tapping mode*: Which quantity is used as input for the feedback system? At which point of the approach–retract cycle does the maximum force on the cantilever occur? The maximum force can either be positive or negative. What are the main differences between both cases regarding the approaching procedure between cantilever/tip and sample?
- In our case the system controlls the distance between tip sample surface and tip. This is done by piezo-materials and a PID controller. How can piezo-materials be used in motion systems? What is a PID controller?
- What's the resolution of an AFM and how is it limited? How is the image influenced by the tip shape?
- What are the advantages of an AFM? E.g. in comparison with a SEM or STM?
- Get information from the internet on how the data is stored on a CD (How are 0 and 1 realized? How is the data stored? Keyword: Eight-to-Fourteen-Modulation).

# 3 Experiments and Evaluation

Goal of these experiments is to learn about the basic functioning and possibilities of the atomic force microscope. The structure of two calibration grids and different surfaces will be observed and quantified with respect to topography and adhesion.

A detailed description on how to operate the microscope and the meaning of the different scan parameters is given in chapter 4. In case of problems, have a look at sections 4.1 and 4.6.

**Important: All measurements for the whole lab course should be performed with a resolution of 512 x 512 points and a scan rate of 1 Hz.**

It takes quite some time to record an AFM image, but you can and you should use this time to do your evaluation.

## 3.1 Topographical Imaging in Tapping Mode

### 3.1.1 *P* and *I* Values

Start the microscope as described in chapter 4.1 and take a screenshot of the resonance curve. As first sample use the calibration grid TGZ02 (see figure 3.3).

Start scanning the surface of the calibration grid. Vary the values of the proportional gain $P$ and the integral gain $I$, while observing the scanning traces in the scope window (below image window). Try to find optimal values for both parameters. Take pictures for the optimal values you found and for too high and too low values for $I$, respectively.

Images that have to be taken:

- Resonance curve (old tip)
- Trace and retrace curve for optimal $P$ and $I$ values
- Trace and retrace curve for too high $I$ values
- Trace and retrace curve for too low $I$ values

Discuss the effect of high/low proportional and integral gain with respect to the image quality versus a reliable height measurement.

**Important: The $P$ and $I$ values have to be checked and potentially adjusted for every new sample.**

### 3.1.2 Tip Characterisation and Limitations of the AFM

In order to experience the limitations of the AFM two calibration grids with different step heights are measured.

Start with calibration grid TGZ02 and take one image with the scanning direction perpendicular to the steps, then change the scanning angle about 90° and take a second image with scanning direction parallel to the steps. Describe what you observe for the different scanning directions and discuss the reason for your observation.

Repeat the measurement with scanning direction perpendicular to the steps for the calibration grid TGZ01 (don't forget to readjust the $P$ and $I$ values). After you finished all measurements with the old tip, determine again the resonance curve and record it.

After that a new tip will be given to you by your supervisor. First record the new resonance curve and then investigate again the grid TGZ01 (perpendicular scanning direction).

All images that have to be taken for this part of the lab course are listed below:

- TGZ02 scanning direction perpendicular to the steps (10$\mu$m x 10$\mu$m) (old tip)
- TGZ02 scanning direction parallel to the steps (10$\mu$m x 10$\mu$m) (old tip)
- TGZ01 scanning direction perpendicular to the steps (10$\mu$m x 10$\mu$m) (old tip)
- Resonance curve (old tip)
- Resonance curve (new tip)
- TGZ01 scanning direction perpendicular to the steps (10$\mu$m x 10$\mu$m) (new tip)

To analyse the grid structure use the three images with perpendicular scanning direction and determine step width $w$, step height $h$ and the angle $\varphi$ (see figure 3.1).

Make five cross sections perpendicular to the steps and measure each value. Calculate the mean value and the standard deviation.

Compare the values for the step height and the step width to the values given by the manufacturer and discuss the relative errors comparing the two grids measured with the old tip.

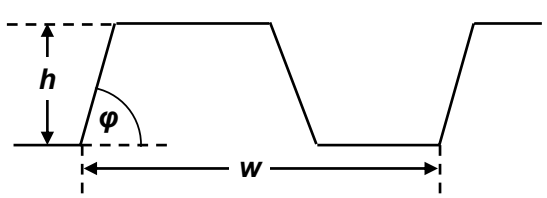



**Figure 3.1:** Values to be determined in the evaluation

Second, compare the values obtained for TGZ01 with the new and the old tip. Discuss these results in terms of tip radius, shape and inclination.

In addition compare the resonance curves with respect to the shape and the resonance position, use as well the resonance curve determined in 3.1.1. Are there any changes/differences?

### 3.1.3 Samples

#### CCD-Chip

**Important: The CCD-chip is a very high sample. Make sure you raise the AFM tip high enough before changing the sample!**

This sample is the CCD-chip of an old digital camera. Take an image of the surface.

- CCD-Chip (10$\mu$m x 10$\mu$m)

Measure (with a ruler) the size of the whole chip. Determine the size of one pixel from your measurement. Calculate the megapixels of this camera. When you do this, remember that the camera can take color pictures! How is the color information obtained?

#### Nano Lattice

The areas with the nano lattices are visible with bare eye as rainbow-coloured stripes when reflecting light on the sample. In the optical microscope the colors are not clearly visible but there are bright and dark stripes. The geometric properties of the stripes change continously from the dark to the bright side. Determine to which end of the rainbow the dark and the bright stripes correspond respectively. Choose two different stripes (one from the dark and one from the bright side) and take one image of each.

Images:

- Stripe from dark side (approx. 1.5$\mu$m x 1.5$\mu$m)
- Stripe from bright side (approx. 1.5$\mu$m x 1.5$\mu$m)

Characterize the structures you find (depths, diameter, distance between features) and compare the values obtained for the two different stripes. How do the geometric dimensions correlate to the color impression?

Imagine the lattice you observed being smaller (with a lattice constant of a few Ångstrom) and the lattice points being occupied by atoms of the same species. Which surface of an fcc crystal is then represented? Hint: Try to remember how the low-index surfaces fcc(100), fcc(110) and fcc(111) look like!

#### CD, DVD and Blue-ray Disc

In order to estimate the capacity of different common optical memory media, samples of a pre-pressed CD, DVD and Blue-ray Disc have been prepared for you. Take an overview image (10$\mu$m x 10$\mu$m) of each sample, think about the best scanning direction (parallel or perpendicular to the spiral lines) to determine the length of a pit.

- CD surface with best scanning direction (10$\mu$m x 10$\mu$m)
- DVD surface with best scanning direction (10$\mu$m x 10$\mu$m)
- Blue-ray Disc surface with best scanning direction (10$\mu$m x 10$\mu$m)

Identify the three different memory types on the basis of your images.
Measure (with a ruler) the area of the media where data is stored. From this area you can calculate the length of the total track using the distance between two spiral lines. Determine the length of the shortest pit and compare this to literature data. How is the digital information (1 and 0) stored on a CD and how many channelbits are coded in the shortest pit? Furthermore, try to estimate the capacity of a CD using your results. (This might be helpful: `http://rz-home.de/~drhubrich/CD.htm#Code`)
Estimate the capacities of a DVD and a Blue-ray Disc. Is the data storage organized in the same way as for the CD?

#### Optional Task

Now prepare a sample of a burned CD surface on your own by retracting a small square of the dye layer. Again take an overview image (10$\mu$m x 10$\mu$m).

- Burned CD surface with best scanning direction (10$\mu$m x 10$\mu$m)

What are the differences between the burned and the pre-pressed CD sample (only qualitatively)? What are the reasons for that?

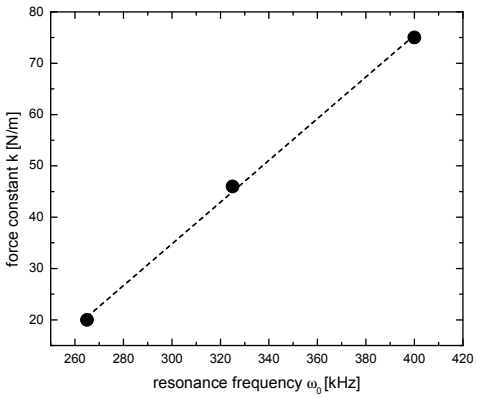



**Figure 3.2:** Determine the force constant

**Additional Samples**

You are encouraged to bring along some samples of your own. The size of the samples should not exceed 10 mm ×10 mm ×4 mm and the structures should not be higher than 500 nm.

## 3.2 Force-Distance Curve

Use a mica surface to get a force-distance curve for approaching and retracting the surface. First you determine again the resonance curve for the tip you are currently using. Then start imaging the surface in tapping mode and switch during acquisition into the ramp mode. Before you do that read again section 4.3. Set the sensitivity to obtain metric deflection data by marking the linear part of the force curve in the TM deflection window. The experiments should be performed on two different objects: an old and a new mica sample, where the influence of the water layer can be identified. Take one image of the force distance curve for each surface, choose the scaling parameters in a way that the interesting part of the curve is maximized.

Images:

- Resonance curve for the tip right before the measurement
- Force Distance Curve for old mica surface
- Force Distance Curve for cleaned mica surface

Discuss the different parts of the curves and the difference between approaching and retracting. Additionally, the difference between the two samples should be identified and quantified by calculating the force that acts on the tip. Therefore determine the difference in deflection between the minimum and the constant part of the curve for the retraction. This difference has to be multiplied by the force constant that can be determined with the help of figure 3.2.

## 3.3 Peak Force Tapping - Nanomechanical Imaging

In this part of the lab course, height and adhesion information should be imaged simultaneously for an organic sample. Set up the AFM to run in the Peak Force QNM (Quantitative Nanomechanical Mapping) mode (as described in 4.4) and start scanning. Record

- a topographic image and
- an adhesion image

of the chosen site. Measure the height and the adhesion contrast between the observed materials. How many materials can be distinguished?

**TGZ01**

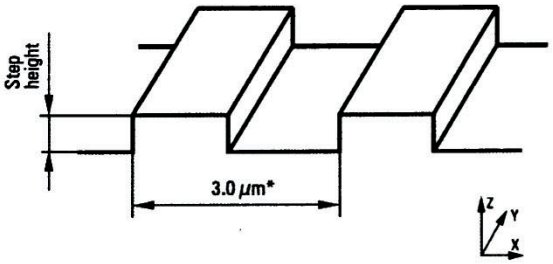
**Grating Characteristics:**
Active area: ..... 3x3 mm
Step height: ..... 18 nm
Accuracy: ..... 1.0 nm

**TGZ02**

**Grating Characteristics:**
Active area: ..... 3x3 mm
Step height: ..... 102 nm
Accuracy: ..... 1,5 nm

**TGZ03**

**Grating Characteristics:**
Active area: ..... 3x3 mm
Step height: ..... 493 nm
Accuracy: ..... 6 nm




* given for information only

Silicon calibration gratings of the TGZ series comprise a one-dimensional array of rectangular steps with a calibrated height value. The gratings are intended for Z-axis calibration of SPM scanners.

**Figure 3.3:** Data sheet for the calibration grids TGZ01 and TGZ02

# 4 Technical Support

This part will give a technical introduction in the use of the AFM type *MultiMode* by *Bruker*. It is meant to be a guide during the execution of the lab course, hence it is not necessary to read it in extension when preparing for the lab, but it should be read at the beginning of the lab course after the introduction of the tutor. Most parts of this section are taken from [10]. The AFM manual is also available on the AFM computer.

Throughout the entire tutorial expressions which are related to the control software or the AFM itself will be given in sans serif font.

## 4.1 Start-up (Tapping Mode/Ramp Mode)

1. Switch on the main power using the button behind the screen.
2. Turn on the computer.
3. Turn on the AFM controller behind the desk.
4. Open the program NanoScope 9.1.
5. Select the experiment category Tapping Mode, the experiment group Tapping Mode in Air and load the experiment FP. All the functions available in this experiment are described in the tables 4.1, 4.2, 4.4 and 4.3.
6. Place the sample on the magnetic sample holder above the piezo.
7. Put the cantilever in the cantilever holder.
8. Place the cantilever holder in the AFM head and fix it with adjustment I (as schematically shown in fig. 4.1).
9. Move the sample to the area of interest with adjustments IIa and IIb using the optical microscope. (Steps 7 and 8 are only necessary when a new cantilever is used.)
10. Locate the laser light on the cantilever tip. Before, verify that the mode switch on the multimode SPM's base is switched to AFM & LFM. Align the laser spot on the cantilever by the laser position adjustments IIIa ($x$-axis) and IIIb ($y$-axis). The maximized value for the SUM should be approximately 7 Volts.
11. Adjust the photo-diode positioner (adjusters IVa and IVb) to set the reflected laser beam in the middle of the diode. The values vertical and horizontal should be close to zero.
12. Put the mode switch on the multimode SPM's base to TM AFM.
13. Search for the resonance frequency of the cantilever (see table 4.4). Auto Tune will automatically search for this frequency. Exit will automatically set the value for drive frequency.
14. Recheck all control panel parameters. The feedback gains and the scan rate are the most important parameters. Start with the integral gain set to 4 and the proportional gain set to 5. The scan rate should be set below 2 Hz. Use a scan size of 10 µm to get an overview.
15. Focus with the optical microscope on the surface, e.g. an edge of the surface.
16. Use the step motor to bring tip and surface closer together. The AFM head has to be totally horizontal. If all goes well after re-engaging, a well-formed cantilever tip will begin to appear on the display monitor. Take care! If the sample is in focus, the tip has to be a little bit above the focus otherwise it will crash. Then use the Engage button (see table 4.2) to make the tip contacting the surface.
17. After the tip contacts the sample, the AFM will automatically start scanning the sample.
18. If the image doesn't look like it should, you can try to adjust the scan controls described in the following section to improve the quality of the image.
19. In order to switch to the Ramp Mode, use the Ramp button in the workflow toolbar (see table 4.2).

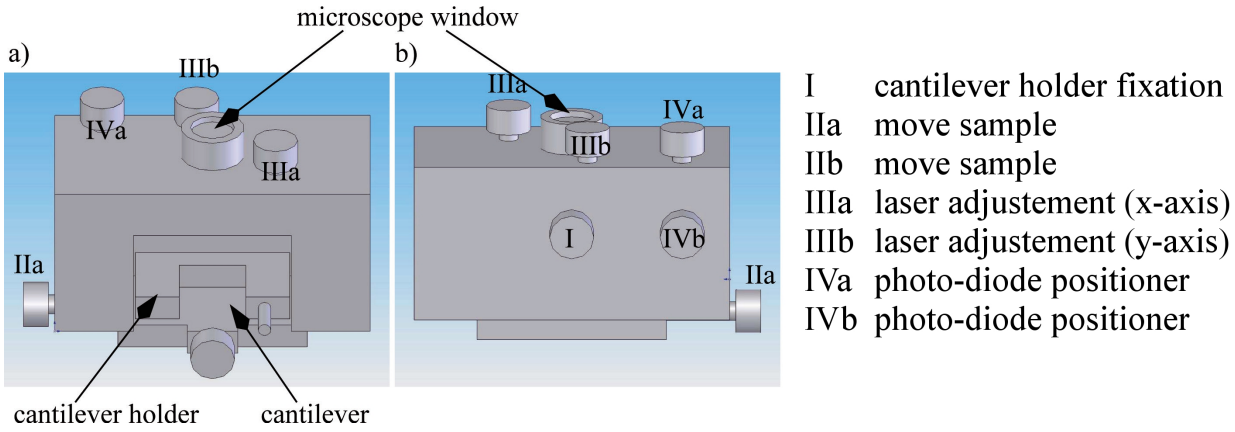



**Figure 4.1:** Schematic image of the AFM head, a) front and b) rear view. For the description of the parts see text.

**Table 4.1:** Description of the NanoScope toolbar buttons for the experimental workspace handling in the main screen.

| | |
|---|---|
|  | Select an experiment workspace to load. |
|  | Open NanoScope files. |
|  | Save current experiment workspace. (Does not save recorded images!) |
|  | Display the NanoScope help. |

**Table 4.2:** Description of the workflow toolbar buttons in the main screen.

| | |
|---|---|
|  | Displays information on the alignment: Crosshair, Tuning,... |
|  | Compare current scanning parameters to the default values. (Changed parameters are highlighted in green.) |
|  | Engage: Approch the surface automatically and start scanning. |
|  | Scanning mode. |
|  | Ramp mode. |
|  | Stops scanning and retracts the tip from the surface. *To change the sample, retract much more using the* UP *button.* |

**Table 4.3:** Description of the NanoScope toolbar capture buttons in the main screen.

| | |
|---|---|
|  | Capture mode: Save the next complete, uninterrupted frame or curve to a file. |
|  | Capture continuous: Continuously saves every frame. |
|  | Capture now: Immediately saves the current uninterrupted image before the frame is complete. |
|  | Abort the capture process. |
|  | Sets the capture filenames to the current date and time. |
|  | Select a directory to store the captured files. |
|  | Show/hide the file browser. |

**Table 4.4:** Description of the NanoScope toolbar buttons for scanning in the main screen.

| | |
|---|---|
|  | Display only basic scanning parameters. |
|  | Display an expanded set of scan parameters. (To be used during the lab course!) |
|  | Turn video monitor on and off. |
|  | Real time status window: Displays the real time z-piezo voltage. |
|  | Search the resonance frequency and set the drive frequency. |
|  | Record force distance curves at specified points. (Advanced!) |
|  | Thermal tune: Measures the energy of the cantilever oscillation due to thermal noise and calculates the spring constant. (Advanced!) |
|  | Capture and store specific controller signals at high rates. (Advanced!) |
|  | Open the Force Monitor (Peak Force Tapping and QNM Mode only). |
|  | Enable Peak Force Capture: Capture a force curve at every pixel (Peak Force QNM Mode only). |
|  | Automatically scales the vertical scan axis. |
|  | Start scanning at the bottom of the scanning area. |
|  | Start scanning at the top of the scanning area. |
|  | Reverse the scanning direction from the current position. |
|  | Start scanning at a user-specified line. (Line #0 : bottom of the scanning area) |
|  | Scan a number of frames specified with the Scan Single Frame Number parameter. |
|  | Continuous scanning. |
|  | Stop scanning *and keep the tip in contact with the sample.* |

## 4.2 Parameters for Tapping Mode

### 4.2.1 Scan Controls:

**Scan Size:** Size of the scan along one side of the square. If the scan is non-square (as determined by the aspect ratio parameter), the value entered is the longer of the two sides. Maximum value: $\sim 10\,\mu m$.

**Aspect Ratio:** Determines whether the scan is to be square (aspect ratio 1:1), or non-square. Default value 1:1.

**X offset, Y offset:** These controls allow adjustment of the lateral scanned area and the center of the scanned area. These values can be chosen between 0 and maximal $\sim 10\,\mu m$, depending on the scan size (piezo deflection in $x$- and $y$-direction is max. $\sim 10\,\mu m$).

**Scan Angle:** Combines $x$-axis and $y$-axis drive voltages, causing the piezo to scan the sample at varying $x$-$y$ angles. Value between 0 and 360 degrees. The scan angle value may affect the scan size.

**Scan Rate:** The number of lines scanned per second in the fast scan ($x$-axis on display monitor) direction. In general, the scan rate should be decreased as the scan size is decreased. Scan rates of 1.5–2.5 Hz should be used for larger scans on samples with tall features. High scan rates help to reduce drift, but they can only be used on very flat samples with small scan sizes. Minimum/maximum value: 0.1–5 Hz.

**Tip Velocity:** The scanned distance per second in the fast scan direction (changes as the scan rate changes).

**Samples/Line:** Number of imaged points per scanned line (default value 512).

**Lines:** Number of scanned lines. This value is the same as samples/line for aspect ratio of 1:1 (default value 512).

**Slow Scan Axis:** Starts and stops the slow scan ($y$-axis on display monitor). This control is used to allow the user to check for lateral mechanical drift in the microscope or assist in tuning the feedback gains. Always set to enable unless checking for drift or tuning gains. Default value: enable.

### 4.2.2 Feedback Controls

**Integral Gain and Proportional Gain:** Controls the response time of the feedback loop. The feedback loop tries to keep the output of the SPM equal to the setpoint reference chosen. It does this by moving the piezo in $z$ to keep the SPM's output on track with the setpoint reference. Piezoelectric transducers have a characteristic response time to the feedback voltage applied. The gains are simply values that magnify the difference read at the A/D converter. This causes the computer to think that the SPM output is further away from the setpoint reference than it really is. The computer essentially overcompensates for this by sending a larger voltage to the $z$ piezo than it truly needed. This causes the piezo scanner to move faster in $z$. This is done to compensate for the mechanical hysteresis of the piezo element. The effect is smoothed out due to the fact that the piezo is adjusted up to four times the rate of the display rate. Optimize the Integral Gain and Proportional Gain so that the height image shows the sharpest contrast and there are minimal variations in the amplitude image. It may be helpful to optimize the scan rate to get the sharpest image. Default values between 1 and 10.

**Amplitude Setpoint:** The setpoint defines the desired voltage for the feedback loop. The setpoint voltage is constantly compared to the present RMS amplitude voltage to calculate the desired change in the piezo position. When the SPM feedback is set to amplitude, the $z$ piezo position changes to keep the amplitude voltage close to the setpoint; therefore, the vibration amplitude remains nearly constant. Changing the setpoint alters the response of the cantilever vibration and changes the amount of force applied to the sample. Default value 250 mV.

**Drive Frequency:** Defines the frequency at which the cantilever is oscillated. This frequency should be very close to the resonant frequency of the cantilever. These value is around 300 kHz for the cantilevers used.

**Drive Amplitude:** Defines the amplitude of the voltage applied to the piezo system that drives the cantilever vibration. It is possible to fracture the cantilever by using too high drive amplitude; therefore, it is safer to start with a small value and increase the value incrementally. If the amplitude calibration plot consists of a flat line all the way across, changing the value of this parameter should shift the level of the curve. If it does not, the tip is probably fully extended into the surface and the tip should be withdrawn before proceeding. Default value 500 mV.

**SPM Feedback:** Sets the signal used as feedback input. Possible signals are Amplitude (default), TM deflection and Phase.

### 4.2.3 Channels

**Data Type:** Height data corresponds to the change in piezo height needed to keep the vibrational amplitude of the cantilever constant. Amplitude data describes the change in the amplitude directly. Deflection data comes from the differential signal off of the top and bottom photo-diode segments.

**Data Scale:** This parameter controls the vertical scale corresponding to the full height of the display and colour bar.

**Data Center:** Offsets the centerline on the colour scale by the amount entered.

**Line Direction:** Selects the direction of the fast scan during data collection. Only one-way scanning is possible.
Range of settings:

- Trace: Data is collected when the relative motion of the tip is left to right as viewed from the front of the microscope.
- Retrace: Data is collected when the relative tip motion is right to left as viewed from the front of the microscope.

**Scan Line:** The scan line controls whether data from the Main of Interleave scan line is displayed and captured.

**Realtime Plane Fit:** Applies a software levelling plane to each realtime image, thus removing up to first-order tilt. Five types of plane-fit are available to each real-time image shown on the display monitor.
Range of settings:

- None: Only raw, unprocessed data is displayed.
- Offset: Takes the $z$-axis average of each scan line, then subtracts it from every data point.
- Line: Takes the slope and $z$-axis average of each scan line and subtracts it from each data point in that scan line. This is the default mode of operation, and should be used unless there is a specific reason to do otherwise.

- Bow: Centers data and removes the tilt and bow in each scan line, by calculating a second order, least-squares fit for the selected segment then subtracting it from the scan line.

**Offline Plane Fit:** Applies a software levelling plane to each offline image for removing first-order tilt. Five types of plane-fit are available to each offline image.
Range of settings:

- None: Only raw, unprocessed data is displayed.
- Offset: Captured images have a DC $z$ offset removed from them, but they are not fitted to a plane.
- Full: A best-fit plane that is derived from the data file is subtracted from the captured image.

**High-pass filter:** This filter parameter invokes a digital, two-pole, high-pass filter that removes low frequency effects, such as ripples caused by torsional forces on the cantilever when the scan reverses direction. It operates on the digital data stream regardless of scan direction. This parameter can be off or set from 0 through 9. Settings of 1 through 9, successively, lower the cut-off frequency of the filter applied to the data stream. It is important to realize that in removing low frequency information from the image, the high-pass filter distorts the height information in the image.

**Low-pass filter:** This filter invokes a digital, one-pole, low-pass filter to remove high-frequency noise from the Real Time data. The filter operates on the collected digital data regardless of the scan direction. Settings for this item range from off through 9. Off implies no low-pass filtering of the data, while settings of 1 through 9, successively, lower the cut-off frequency of the filter applied to the data stream.

**Table 4.5:** Description of the menu buttons in the ramp mode.

| | |
|---|---|
|  | The tip is continuously lowered and raised by a distance equal to the Ramp size parameter. |
|  | Lowers and raises the tip once by a distance equal to the Ramp size parameter. |
|  | Halts all tip movements. |
|  | Continuous Approach: The tip lowers to the surface and raises in a controlled series of steps. When tip deflection exceeds the Threshold Step amount, it halts and the resulting force curve is displayed. (Advanced!) |
|  | The tip is lowered to the surface and raised in a single, controlled step. It halts if the surface is encountered by the tip, causing deflection exceeding the Step threshold amount. The resulting force curve is displayed. (Advanced!) |
|  | Starts automatic ramping as defined in the Auto Panel. (Advanced!) |
|  | Update the cantilever deflection sensitivity: Calibration of deflection to a metric scale. |

# 4.3 Parameters for Ramp Mode

## 4.3.1 Main Control

**Ramp Output:** Defines the variable to be plotted along the $x$-axis of the scope trace. Default Z.

**Ramp size:** This parameter defines the total travel distance of the piezo. Use caution when working in the ramp mode. This mode can potentially damage the tip and/or surface by too high values for the ramp size.

**Ramp Position:** This parameter sets the offset of the piezo travel (i.e., its starting point). It sets the maximum voltage applied to the $z$ electrodes of the piezo during the force plot operation. The triangular waveform is offset up and down in relation to the value of this parameter. Increasing the value of the Ramp Position parameter moves the sample closer to the tip by extending the piezo tube.

**Ramp Rate:** sets the rate with which the $z$-piezo approach/retract the tip. Default value: 1 Hz.

**X offset and Y offset:** controls the center position of the scan in the $x$- and $y$- directions, respectively; same as in image mode. Range of settings: $\pm 220$ V.

**Number of samples:** Defines the number of data points captured during each extension/retraction cycle of the $z$-piezo. Default value: 512 .

**Average Count:** sets the number of scans taken to average the display of the force plot. May be set between 1 and 1024. Usually it is set to 1 unless the user needs to reduce noise.

**Spring Constant:** This parameter relates the cantilever deflection signal to the $z$ travel of the piezo. It equals the slope of the deflection versus $z$ when the tip is in contact with the sample. For a proper force curve, the line has a negative slope with typical values of 10–50 mV/nm; however, by convention, values are shown as positive in the menu.

**Display Mode:** The portion of a tip's vertical motion to be plotted on the force graph.
Range of settings:

- Extend: Plots only the extension portion of the tip's vertical travel.
- Retract: Plots only the retraction portion of the tip's vertical travel.
- Both: Plots both the extension and retraction portion of the tip's vertical travel.

**Plot Units:** This item selects the units of the parameters, either metric lengths volts or force.

**X Rotate:** allows the user to move the tip laterally, in the $x$-direction, during indentation. This is useful since the cantilever is at an angle relative to the surface. One purpose of X Rotate is to prevent the cantilever from ploughing the surface laterally, typically along the $x$-direction, while it indents in the sample surface in the $z$-direction. Ploughing can occur due to cantilever bending during indentation of due to $x$-movement caused by coupling of the $z$ and $x$ axes of the piezo scanner. When indenting in the $z$-direction, the X Rotate parameter allows the user to add movement in the $x$-direction. X Rotate causes movement of the scanner opposite to the direction in which the cantilever points. Without X Rotate control, the tip may be prone to pitch forward during indentation. It typically ranges between 7 and 25 degrees. Normally, it is set to about 12 degrees.

**Amplitude setpoint:** Same as in image mode.

### 4.3.2 Channels

**Data Type/Data Scale/Data Center:** Same as in image mode.

**X Data Type:** Type of data that the channel data is being compared to. This data displays on the X-axis of the scope grid.

**TM Deflection Sensitivity:** TM Deflection Sensitivity relates the vibrational amplitude of the cantilever (in nm) to the amplitude signal (in V) from the controller. To calculate the TM Deflection Sensitivity, measure the slope of the RMS amplitude versus the Z piezo position when the tip is in contact with a very hard, stiff sample. The NanoScope software automatically calculates and enters the value from the graph. You must properly calibrate the TM Deflection Sensitivity before amplitude data can be correctly displayed in nm.

**Amplitude sens.:** This item relates the vibrational amplitude of the cantilever to the $z$ travel of the piezo. It is calculated by measuring the slope of the RMS amplitude versus the $z$ voltage when the tip is in contact with the sample. The NanoScope system automatically calculates and enters the value from the graph after the operator uses the mouse to fit a line to the graph. This item must be properly calculated and entered before reliable deflection data in nanometers can be displayed. For a proper force curve, the line has a negative slope with typical values of 10–50 mV/nm; however, by convention, values are shown as positive in the menu.

## 4.4 Start-up (Peak Force QNM Mode)

The Peak Force QNM (Quantitative Nanomechanical Mapping) Mode uses the Peak Force Tapping feedback (see 2.1.4) to extract the height profile of the

sample as well as nanomechanical properties, such as adhesion. *Due to the oscillation of the sample at acoustic frequencies (which moves the sample), it is necessary to fix the sample to the holder.*

1. Follow the instructions 1. - 4. in section 4.1.
2. Select the experiment category Mechanical Properties, the experiment group Quantitative Nanomechanical Mapping and load the experiment PeakForce QNM in Air - FP. All functions available in this experiment are described in table 4.4.
3. Follow the instructions 6. - 12. and 15. - 17. in section 4.1.
4. If the image doesn't look like it should, check if the force monitor displays a reasonable force-distance behavior. You can try to adjust the scan controls described in section 4.5 to improve the quality of the image.

### 4.4.1 QNM Calibration:

In order to enable the calculation of meaningful nanomechanical data, it is necessary to calibrate several properties of the cantilever and the tip (see 2.1.4).

**Deflection Sensitivity:** Switch to the Ramp mode, perform continuous ramping and update the sensitivity.

**Spring Constant:** After the calibration of the deflection sensitivity, the spring constant of the cantilever is determined by the Thermal Tune procedure: Withdraw the tip and open the Thermal Tune window. Acquire Data will record deflection data based on thermal noise. Calculate PSD will adjust the power spectral density bin width, Fit Data will fit a Lorentzian model to the data, and Calculate Spring k will finally determine the spring constant.

**Tip Radius:** Enter the tip radius given in the manufacturer's datasheet.

## 4.5 Parameters for Peak Force QNM Mode

### 4.5.1 Scan Controls:

**Peak Force Setpoint:** The setpoint for the peak force. If the deflection sensitivity is calibrated, the force (in Newtons) will be displayed.

### 4.5.2 Feedback Controls:

In this mode, all important Peak Force Tapping feedback parameters are automatically controlled and should therefore changed only in consultation with the tutor.

### 4.5.3 Channels:

**Height:** Height information on the sample surface.

**Peak Force Error:** Feedback signal in the Peak Force QNM mode.

**Adhesion:** Adhesion force information on the sample surface.

**Dissipation:** Information on the energy dissipated in each cycle of extension and retraction of the tip.

## 4.6 Trouble Shooting

It happens rather often that one thinks to have checked all control panel parameters correctly but does not get an image. Here a list of the most common errors.

- Right input channel (see monitor). Height signal for constant height and deflection signal for constant deflection mode.
- Correct gains for proportional gain and integral gain. These parameters have to be between 1 and 10, depending on the softness/hardness of the sample. Start with the integral gain set to 4 and the proportional gain set to about 5.
- Is the height scale justified correctly for the different channels?
- What is the size of the scan area? The scan size can be varied. Use a scan size of 10 µm to get an overview.
- Is the piezo in its limit between + and −220 V? This can be checked by the green/red setpoint line in the approach/retract bar. This green/red setpoint line should not be on the left or on the right site of the bar. This can be changed by the Amplitude Setpoint parameter.
- Does the force used to image the probe destroy the probe? Play around with the Amplitude Setpoint parameter and the Drive Amplitude parameter. The slope of the force curve shows the sensitivity of the Tapping Mode measurement. In general, higher sensitivities will give better image quality. The Ramp mode enables one to plot the cantilever amplitude versus the scanner

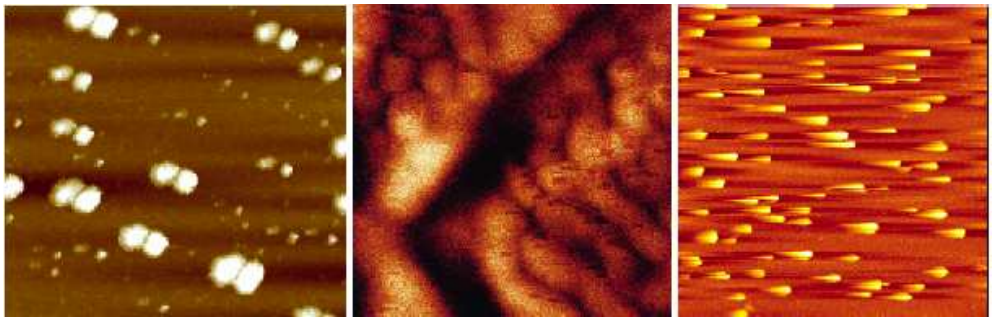


**Figure 4.2:** Artifacts can occur due to a defective tip. Left: a multiple tip occurs as a pattern; middle: repeating patterns due to several tip areas scanning at the same time; right: silverfish structures due to high setpoint

position (= force curve). The curve should show a mostly flat region where the cantilever has not yet reached the surface and the sloped region where the amplitude is being reduced by the tapping interaction. To protect the tip and sample, take care that the cantilever amplitude is never reduced to zero. Adjust the setpoint until the green setpoint line on the graph is just barely below the flat region of the force curve. This is the setpoint that applies the lowest force on the sample.

## 4.7 Poor Image Quality

### Contaminated tip

Some types of samples may adhere to the cantilever and tip (e.g. certain proteins). This will reduce resolution giving fuzzy images. If the tip contamination is suspected to be a problem, it will be necessary to protect the tip against contamination.

### Dull tip

AFM cantilever tips can become dull during use and some unused tips may be defective. If imaging resolution is poor, try changing to a new cantilever.

### Multiple tip

AFM cantilevers can have multiple protrusions at the apex of the tip which result in image artifacts. In this case, features on the surface will appear two or more times in the image, usually separated by several nanometers (see figure 4.2 left). If this occurs and this effect doesn't disappear after some time, change or clean the AFM tip.

### Repeating pattern

If a repeating pattern appears, more tip areas scan simultaneous (see figure 4.2 middle). Such a cantilever has to be changed.

### Too low setpoint

Some structure shows up that does not exist (e.g. circles). Change the amplitude setpoint to a better value.

### Too high setpoint

If silverfish structures appear (see figure 4.2 right), the adjustment of the integral and proportional gain can help, but if the setpoint is not adjusted correctly, increasing the gain will worse the noise of the image.

### Too high drive frequency

The image surface looks destroyed; e.g. holes appear. These holes are artifacts and disappear when the drive frequency is lowered.

### Other error sources

- Scanner beeps loud: decrease immediately the gains; the scanner is over-driven.
- The image seems only noise: Adjust Amplitude Setpoint.
- Strong drift of signal: withdraw the tip from the surface and engage again.

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