Atomic Force Microscopy

Dr. Robin Ohmann
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Manual: Masterpraktikum
University of Siegen

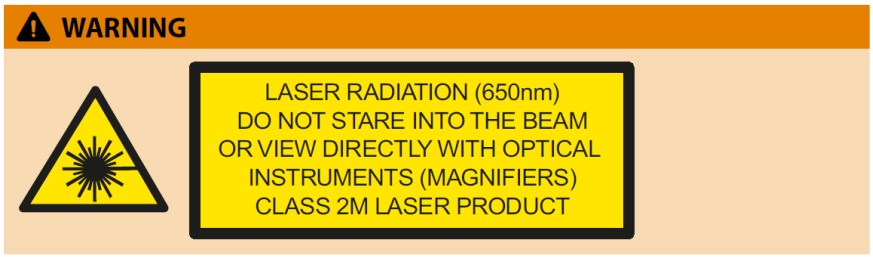

Image on title page: www.nanosurf.com

Location of lab: Room ENC-B 027
AG Experimentelle Nanophysik
Prof. Carsten Busse

Contents

1. Introduction

In this lab course, you will learn about atomic force microscopy (AFM). The atomic force microscope was invented by Gerd Binnig, Calvin Quate and Christoph Gerber in 1986 [1]. It belongs to the group of scanning probe methods, where a tip is moved across a surface measuring the topography and other properties of the surface. In the case of an AFM the interaction between tip and sample is measured. The main interactions are the attractive van der Waals interaction and the Pauli repulsion. Additionally, electrostatic interactions, magnetic interactions as well as chemical interactions contribute to the detection signal. The tip can be in direct contact or a few nanometers away from the surface. In contrast to scanning tunneling microscopy (STM), not only conducting, but also non-conducting samples can be measured. This makes AFM a useful tool not only in physics and chemistry, but also for biological applications. In Figure 1 several examples of AFM images are displayed. While the main usage is to measure the surface topography, several other modes exist allowing to measure elastic properties, such as the stiffness of a sample, or the mapping of magnetic domains or manipulate the surface.

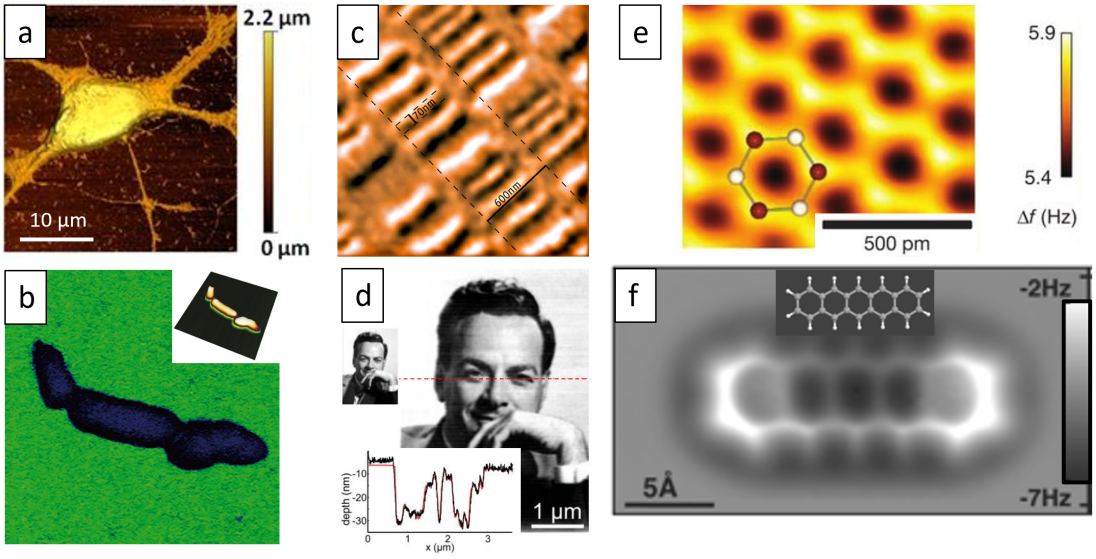

Figure 1.1: Atomic force microscopy images: a) Topography image of a neuron [2], b) elasticity map of a bacteria (inset: topography) [3], c) magnetic force microscopy image of a hard disk drive platter [4], d) topography image of a pattern of a photograph (inset) of Richard Feynman fabricated by thermal scanning probe lithography [5], e) and f) frequency shift maps obtained by non-contact atomic force microscopy using a qPlus sensor of e) HOPG (highly-oriented pyrolitic graphite) [6] and f) pentacene molecule [7].

2. Atomic Force Microscopy

2.1 Theory

The most important interactions between tip and sample are the van der Waals interaction and the Pauli repulsion. These occur between two atoms or molecules and depend on the distance between them. The van der Waals interaction is caused by induced dipoles in atoms or molecules and is always attractive. The Pauli principle states, that not two or more fermions (for example electrons) can have the same quantum numbers. When two atoms or molecules come close to each other the wavefunctions can overlap leading to a repulsion. Adding both interactions leads to the Lennard-Jones potential, which is a simplified model potential approximating the interactions of two non-charged atoms or molecules:

$$V(r) = 4\epsilon \left[\left(\frac{\sigma}{r}\right)^{12} - \left(\frac{\sigma}{r}\right)^{6} \right]$$

Here, r is the distance between the atoms or molecules, ϵ the depth of the potential minimum and σ the distance between the particles when the potential is zero $V(r = \sigma) = 0$. The positive term describes the repulsive interaction and the negative term the attractive interaction (see Figure 2.1). At a distance $r_m = \sqrt[6]{2} \cdot \sigma \approx 1,12 \cdot \sigma$ the minimum of the potential is located, which is the energetically preferred distance of the particles. The force between the particles is given by the first derivative of the potential:

$$F(r) = -\frac{\mathrm{d}V}{\mathrm{d}r} = 24\epsilon \left[\frac{2\sigma^{12}}{r^{13}} - \frac{\sigma^{6}}{r^{7}} \right] .$$

The force crosses zero at $F(r = r_m) = 0$, that is at the minimum of the potential. For all distances $r < r_m$ the force (interaction) is repulsive and for all distances $r > r_m$ attractive forces are acting between the particles. That is, for the force positive and negative values distinguish between repulsion and attraction, while for the potential it is the left or right of the minimum at which point the first derivative of the potential changes sign. Additionally to the van der Waals interaction and the Pauli repulsion other interactions can occur depending on the material of tip and sample. These are for example magnetic interactions or electrostatic interactions.
To measure the interaction between tip and sample, the tip is attached to a flexible cantilever or the cantilever is directly fabricated in one piece, where the end of the cantilever has the shape of a tip. The forces acting on the cantilever lead to a bending of the cantilever following Hooke's law:

$$F = -\Delta x \cdot k \,,$$

where Δx is the displacement and k the spring constant of the cantilever (see Fig. 2.2).

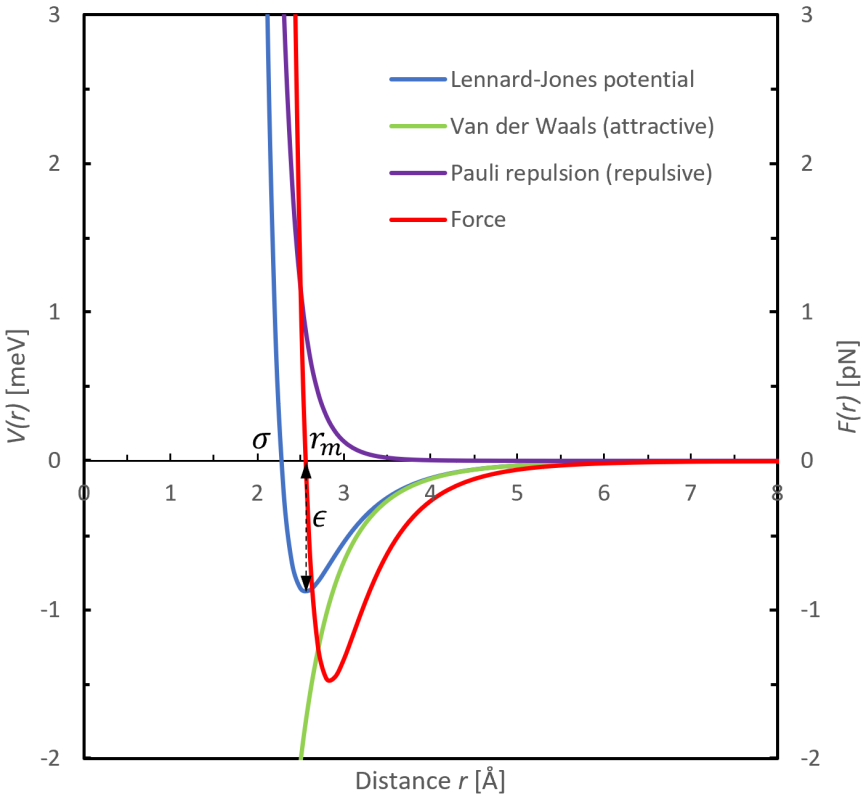

Figure 2.1: Lennard-Jones potential as a function of distance between two particles. As an example, the relationship between two He atoms is shown (where $\epsilon_{\mathrm{He}} = 0.88$ meV and $\sigma_{\mathrm{He}} = 2.28$ Å). Furthermore, the van der Waals interaction term and the Pauli repulsion term are shown as well as the force between the two atoms as a function of distance. For the latter, the right vertical axis applies.

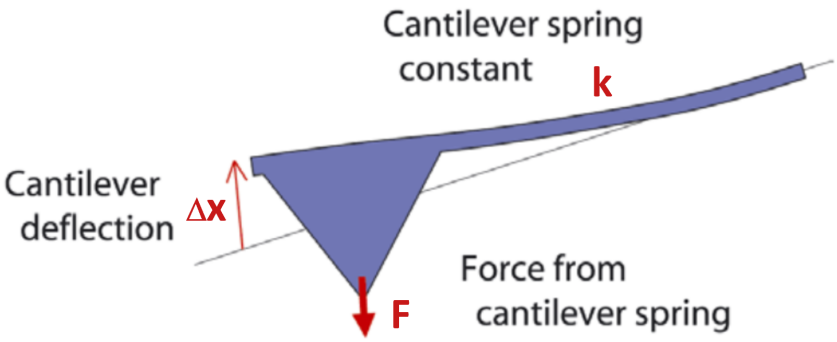

Figure 2.2: Schematic of the cantilever illustrating Hooke's law [8].

2.2 Working principle

The schematic setup of an AFM is depicted in Figure 2.3. The sample is scanned with a cantilever, at which a tip is attached interacting with the sample. The relative movement between sample and cantilever is carried out with piezoelectric elements. Depending on the construction of the AFM either the sample or the cantilever is moved. The piezoelectric elements allow a precise control of the movement with nanometer precision. By applying a voltage to the piezoelectric elements these deform following the piezoelectric effect and thus lead to a movement of the cantilever or the sample in all directions. To detect the bending of the cantilever due to the interaction with the sample, typically a laser is used, which gets focussed on the cantilever and the reflection of the laser beam off the cantilever is measured with a four quadrant photodiode. When acquiring a topographic image the image is scanned line by line, which is characteristic for scanning probe methods.

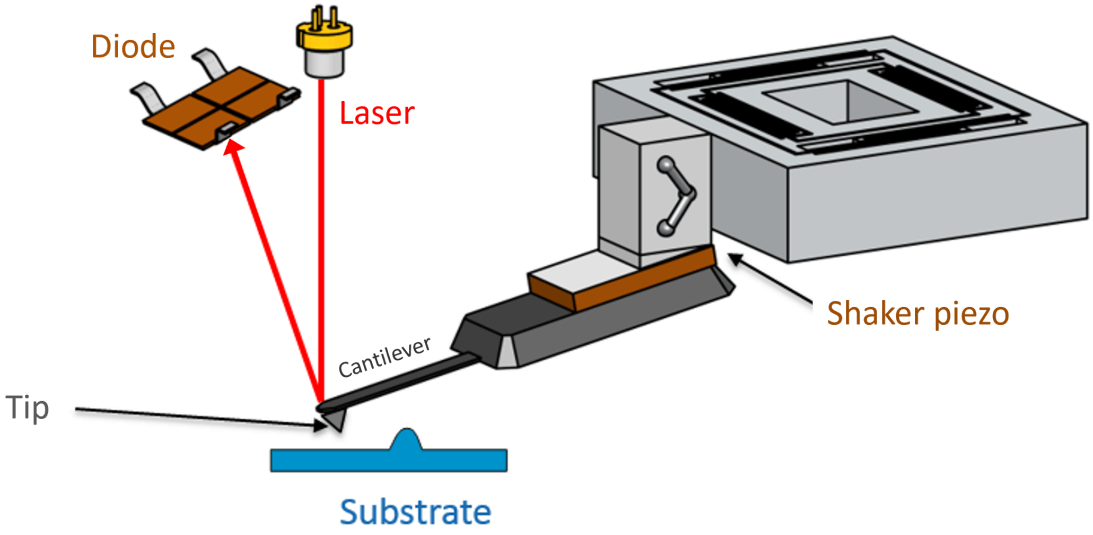

Figure 2.3: Schematic setup of an AFM [10].

2.2.1 Microscopy

Static force mode

In static force mode or contact mode the tip is in contact with the sample and therefore in the repulsive regime. While scanning a feedback loop meachnism maintains a constant force by adjusting the z-height with the piezo elements. This change in z-height is the topographic signal. Note, if the surface is composed of more than one material, it may occur that even if the atoms are arranged in the same geometrical plane a different z-height is measured due to the different interactions with the tip of the different materials.

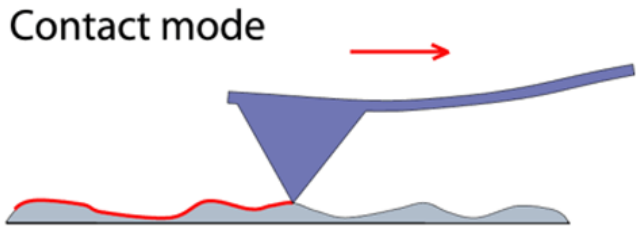

Figure 2.4: Schematic of contact mode of an AFM [8].

Dynamic force mode

In dynamic force mode the cantilever gets excited with a shaker piezo and oscillates close to the sample. In case the tip touches each time the sample this is referred to as intermittent contact or tapping mode (see Figure 2.5). If the tip is a little further away from the sample and does not touch the sample one speaks of non-contact mode. In the Masterpraktikum as dynamic mode the intermittent contact mode is used. Hereby, the cantilever gets excited close to its resonance frequency.

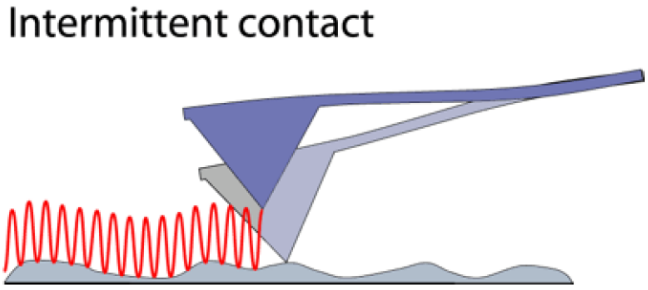

Figure 2.5: Schematic of the dynamic force mode intermittent contact mode (tapping mode) of an AFM [8].

Phase imaging mode

The response signal determined by the actual mechanical movement of the cantilever exhibits a phase shift with respect to the excitation signal driving the cantilever in the dynamic force mode. This phase shift can be recorded in an extra phase channel and is given by:

$$\Delta x = A \sin(2\pi f t + \phi)$$

where Δx is the deflection of the cantilever, A the amplitude, f the frequency, t the time and ϕ the phase shift. The phase shift depends on the interaction between tip and sample and also on the mechanical properties of the sample. Depending on adhesion, stiffness, dissipation and viscoelasticity of the sample the phase can change, without any change in topography (see Figure 2.6). Due to the multiple ways of changing the phase the actual phase shift is a convolution out of all the properties and it can therefore be challenging to interpret the phase contrast maps.

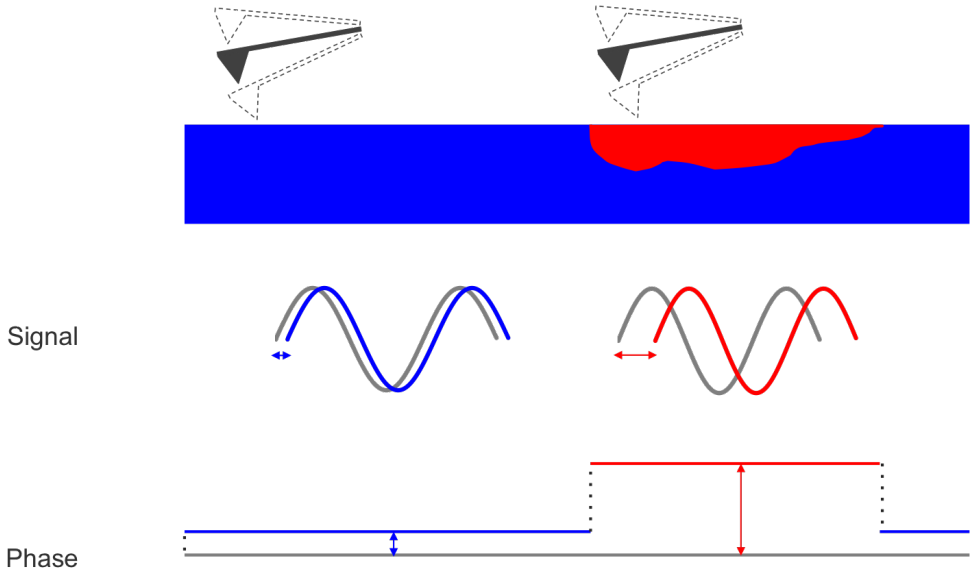

Figure 2.6: Schematic of the phase imaging mode of an AFM [9].

3. Experimental setup

3.1 NaioAFM

The NaioAFM (see Figure 3.1 orange box) consists of the scan head, including the cantilever, laser system and piezoelectric elements, a sample stage with magnetic coupling of the sample holder as well as an $x-y-$table with positioning screws [11]. The NaioAFM rests on a vibration isolation table to mechanically damp external vibrations.

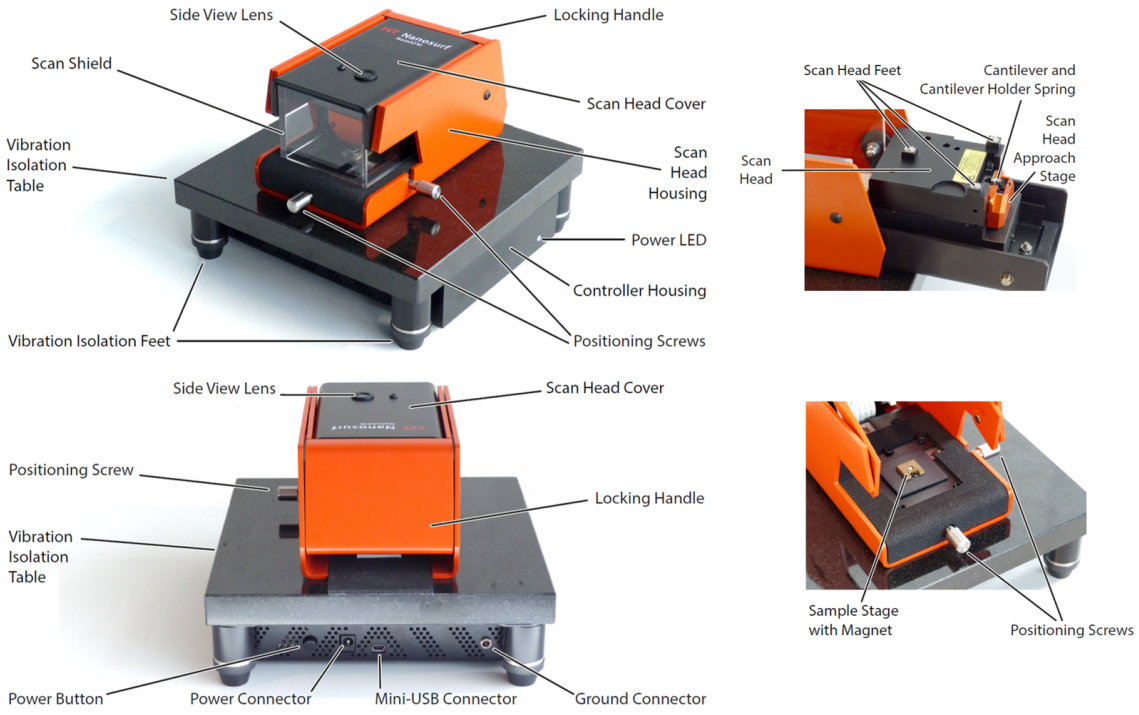

Figure 3.1: Setup of NaioAFM [11].

3.2 Software

To control the NaioAFM the software **Nanosurf Naio** is used. The main window is depicted in Figure 3.2.

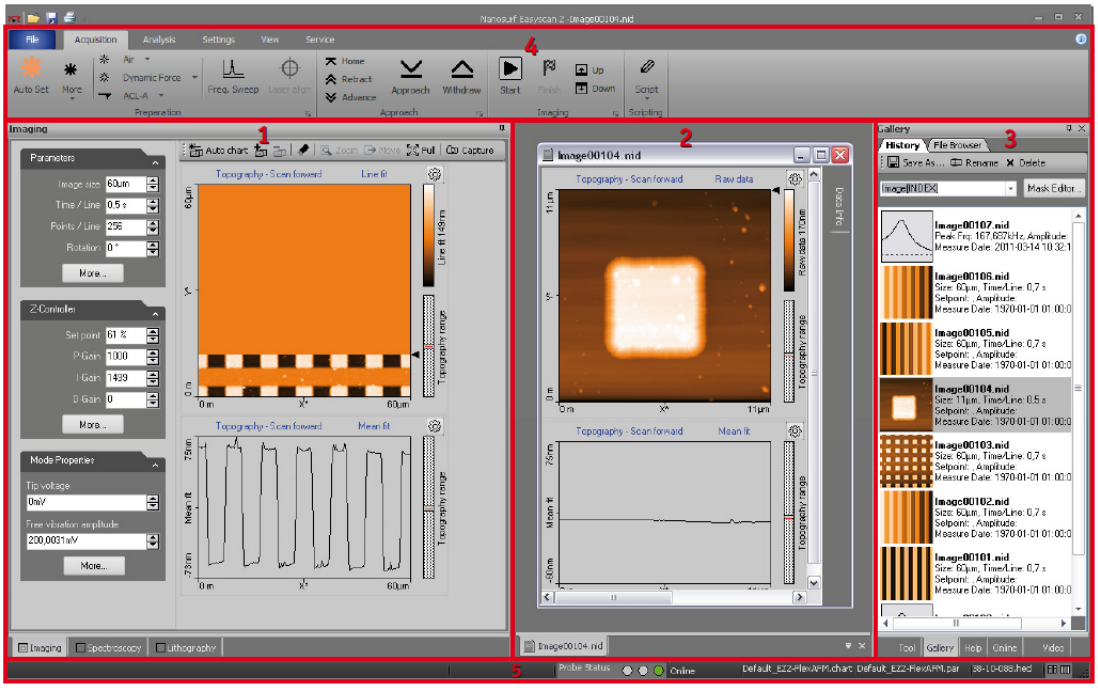

Figure 3.2: User interface of the software Nanosurf Naio (Version 3.8). Marked sections are: 1) Data acquisition pane, 2) Data viewing space, 3) Files, 4) Function selection bar and 5) Status bar [11].

The user interface contains five main areas, which are labeled in the image and described in detail in the following:

1. Data acquisition pane: In the left column parameters for scanning can be set. Important parameters are for example: **Image size**,**Time/Line**, **Points/Line** and **Setpoint**. More parameters can be adjusted via the button **More...**. In the right column the currently running measurement is displayed in real time. At the bottom there are three tabs allowing to select different modes: **Imaging** (atomic force microscopy), **Spectroscopy** and **Lithography**. The default setting is **Imaging**.

2. Data viewing space: By double click on one of the files displayed in area 3 the data files can be opened and examined. This is mainly for having a quick look at the acquired data, for detailed analysis an extra program such as WSxM [12] is needed.

3. Files: Display of files with file name, thumbnail and some scanning parameters, when at the bottom the tab **Gallery** is selected. With the button **Mask Editor...** the file name can be chosen. It is recommended to use the material of the sample as a filename and an automatic index with [INDEX].

4. Function selection bar: When **Acquisition** tab is active, it is possible to select operation modes, set the type of cantilever, acquire a resonance curve, coarse move in z-direction for approach and retraction and start a scan with the Play button and pause it with the Pause button, which appears after starting the scan. Two types of operation modes can be selected: **Static Force Mode** and **Dynamic Force Mode**. Pressing the **Freq. Sweep** button opens a new window, in which the resonance curve und the phaseshift can be determined. In the panel Approach the tip-sample distance can be manually increased with **Retract** or manually decreased with **Advance**. When pressing **Advance** one needs to take care not to crash the cantilever into the sample. With the button **Approach** the cantilever-sample distance is automatically decreased step by step and as soon as a large enough interaction is detected the approach is stopped and the scan is automatically started. Note, that before pressing approach it is advisable to set already the intended scan parameters (see Data acquisition pane).

5. Status bar: The **Probe Status** is displayed by colored filled circles:
green (●) cantilever-sample distance is within a measurable distance, yellow (●) cantilever-sample distance is too large, red (●) tip is inside the sample. On the right side of the status bar the currently used calibration file is displayed.

4. Experimental procedures

To measure surfaces with the NaioAFM a suitable cantilever as well as the desired sample need to be inserted. To bring cantilever and sample close enough to detect an interaction the cantilever is approached manually up to a reasonable close distance to the sample and then the automatic approach is started. As soon as the approach has finished the scan starts. Once a scan finishes the image is saved and can be used for data analysis.

4.1 Cantilever exchange

Following cantilevers are used in the Masterpraktikum:

- ContAl-G (static force mode)
  -Silicon tip with radius $R_{\mathrm{Tip}} < 10$ nm
  -Cantilever length $L = 450$ μm and width $W = 50$ μm
  -Resonance frequency $f_0 = 13$ kHz (6- 21 kHz)
  -Spring constant k=0.2 N/m (0.02-0.77 N/m)

- Tap190Al-G (dynamic force mode)
  -Silicon tip with radius $R_{\mathrm{Tip}} < 10$ nm
  -Cantilever length $L = 225$ μm and width $W = 38$ μm
  -Resonance frequency $f_0 = 190$ kHz (160- 220 kHz)
  -Spring constant k=48 N/m (28-75 N/m)

To exchange the cantilever the housing of the NaioAFM needs to be properly opened following the steps as depicted in Figure 4.1. Then the scan head containing the cantilever slot can be accessed. In order to prevent the cantilever from falling down a drop stop is to be inserted (see Figure 4.2 a). To unlock the cantilever place the insertion tool as shown in Figure 4.2 b pressing down the clamp. With the tweezer take out the old cantilever (see Figure 4.2 c). **Note, handling the cantilever with the tweezer is a very delicate process as the cantilever can be easily dropped or damaged.** Therefore, take extra precaution. It is advisable to already have the cantilever box close by so that you do not have to carry the cantilever over a long distance minimizing the risk. Also, take care that you apply always a suitable pressure on the tweezer not too much that the cantilever gets rotated or flipped, but also not too little that the cantilever drops. Once the old cantilever is properly stored in the box, take out the desired cantilever and insert it into the cantilever slot. The cantilever needs to be aligned as shown in Figure 4.2 e. Once the cantilever is in place remove the insertion tool and the drop stop and close the AFM again in the reverse order as shown in Figure 4.1.

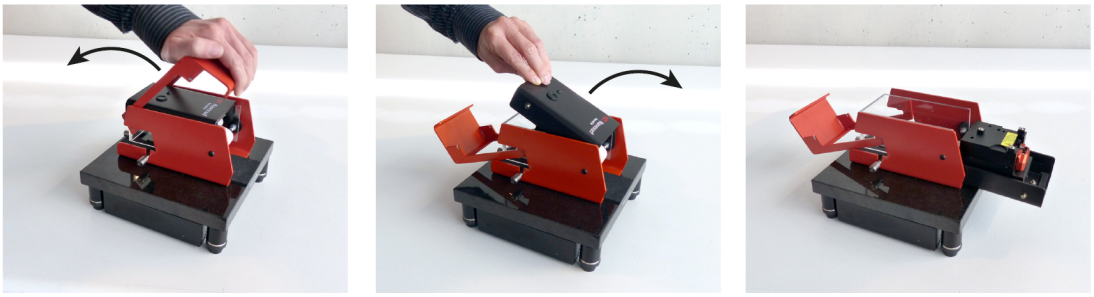

Figure 4.1: Opening of the AFM to exchange cantilever.

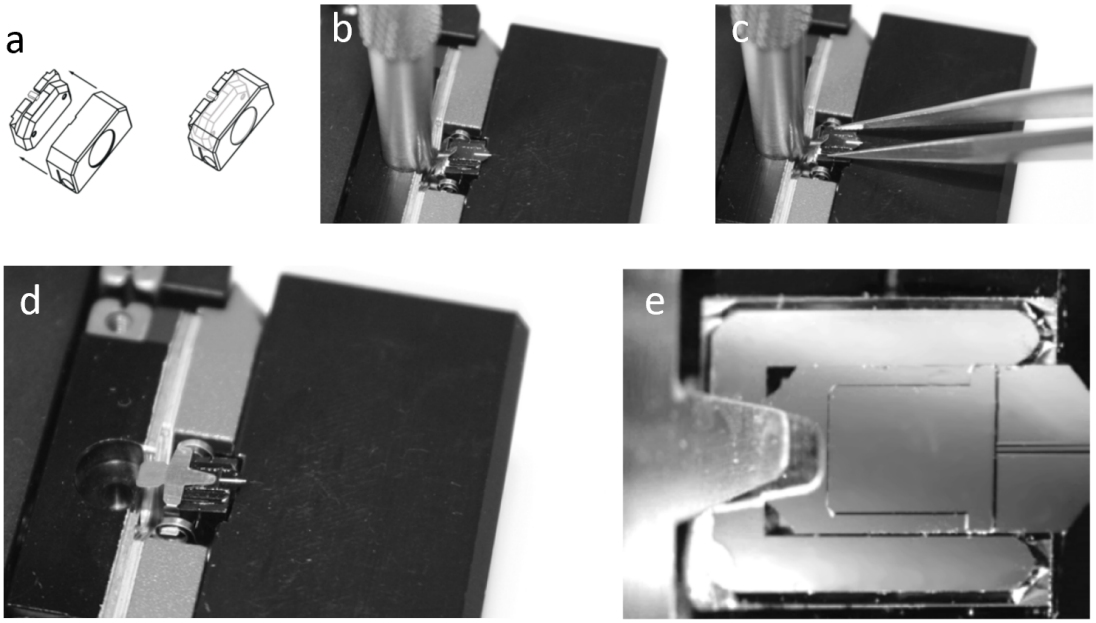

Figure 4.2: Cantilever exchange: a) Insertion of the drop stop, b) pressing the clamp with the insertion tool to release cantilever, c) take out cantilever with the tweezer, d) new inserted cantilever, e) correct position of cantilever.

4.2 Sample exchange

For the sample exchange the NaioAFM needs to be opened similar to the cantilever exchange except with an additional fourth step as shown in Figure 4.3. After the opening the sample stage can be accessed. To remove the sample pinch the sample holder with the tweezer and slightly tilt the sample holder so that the sample gets magnetically decoupled from the sample stage and then place the sample in the proper sample box. Then pick up the desired sample and place it on the sample stage. In case the sample holder is not properly in the center you can push it slighlty in place with the tweezer.

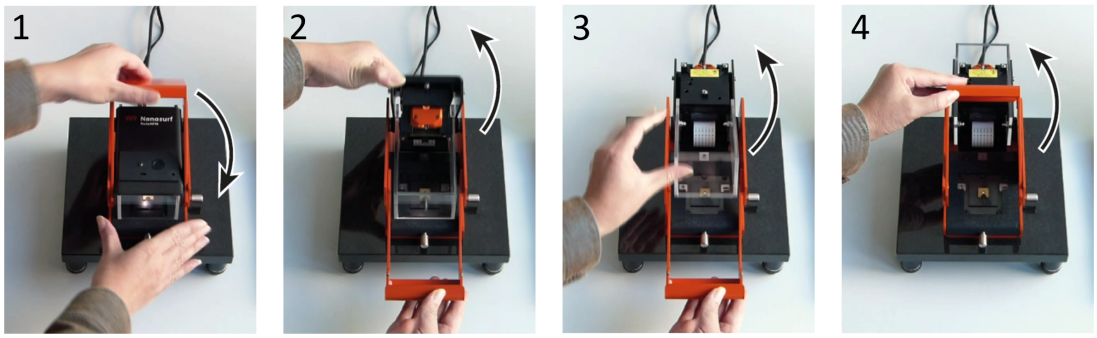

Figure 4.3: Opening of the AFM to exchange sample

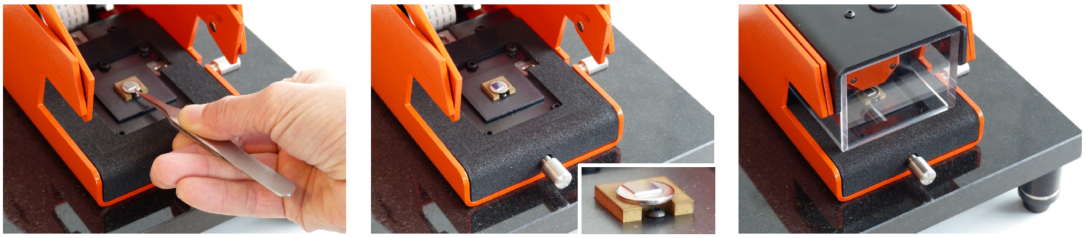

Figure 4.4: Sample exchange with a tweezer.

4.3 Sample preparation

Following samples are used during the course:

- DVD
- SBS (poly(styrene-butadiene-styrene)) -PS (polystyrene) polymer blend

A piece of a **DVD** is already mounted on a sample holder and is **ready to be used**.

The **SBS-PS polymer blend** needs to be freshly prepared using the following **procedure** (Note, in order to avoid contact to the solution gloves have to be worn.):

1. Cleaving of mica substrate:

Press a piece of adhesive tape (approx. 4 cm long) on top of the mica substrate (see Figure 4.5 a). Use your finger to gently attach the tape to the mica. While using the tweezers to hold the metal disc down to the table, peel off the adhesive tape from the substrate (see Figure 4.5 b). On the tape there should be now a round thin layer of mica (see Figure 4.5 c). In case, the layer is not fully round or not complete, repeat the cleaving again. After the successful cleaving, do not touch the clean mica surface.

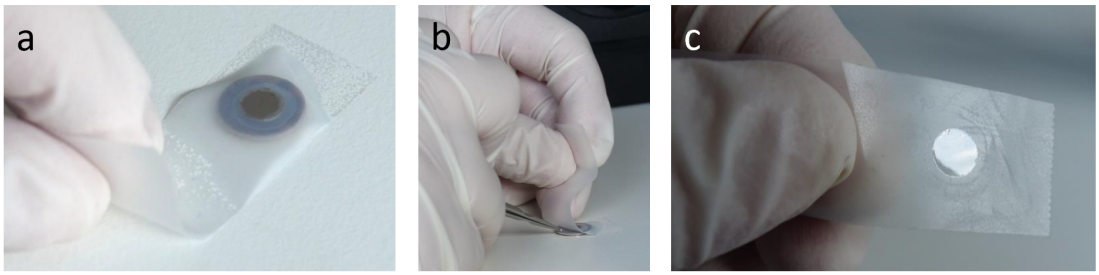

Figure 4.5: Cleaving of mica substrate using adhesive tape.

2. Drop-casting of polymer blend solution

Open the vial containing the SBS-PS polymer blend solution. Insert the narrow end of the yellow pipette into the solution (see Figure 4.6 a). A small amount of solution will enter the pipette due to capillary action (see Figure 4.6 b). Press your finger on top of the pipette and remove the pipette from the vial. Then hold the pipette close on top of the mica and release a drop of solution on mica (see Figure 4.6 c). The liquid will disperse on the substrate as shown in the inset of Figure 4.6 c. To remove excess solution grab the disc with the tweezers on the edge, tilt it by 45° and touch with tissue paper the edge of the disc to soak up the solution. After this procedure leave the sample for another minute to dry out so that all remaining solvent gets evaporated.

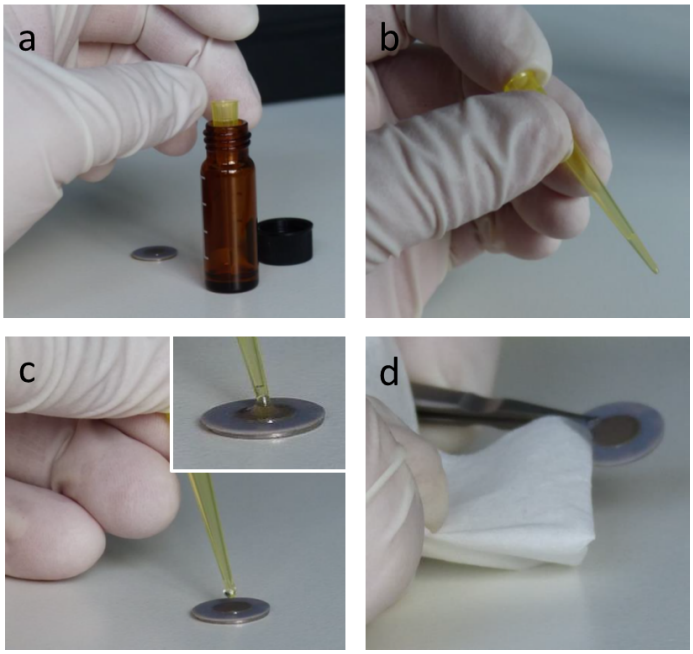

Figure 4.6: Application of polymer blend solution on mica.

4.4 Approach

The tip needs to be brought within a few nanometers close to the sample so that a sufficiently strong interaction can occur. The approach of the tip to the sample is done in two steps:
1. **Manual approach:** The tip is moved manually close to the sample by clicking on **Advance** (see Figure 4.7) (for large distances keep the button pressed). The distance to the sample can be monitored by looking through the lens. Once the tip is within a few times of the cantilever width the manual approach is to be stopped (see Figure 4.8). Do not go any closer as the cantilever can then be crashed into the sample and can be damaged.

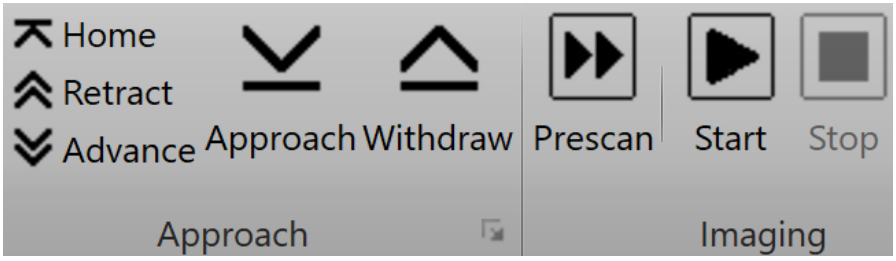

Figure 4.7: Buttons for approach and imaging

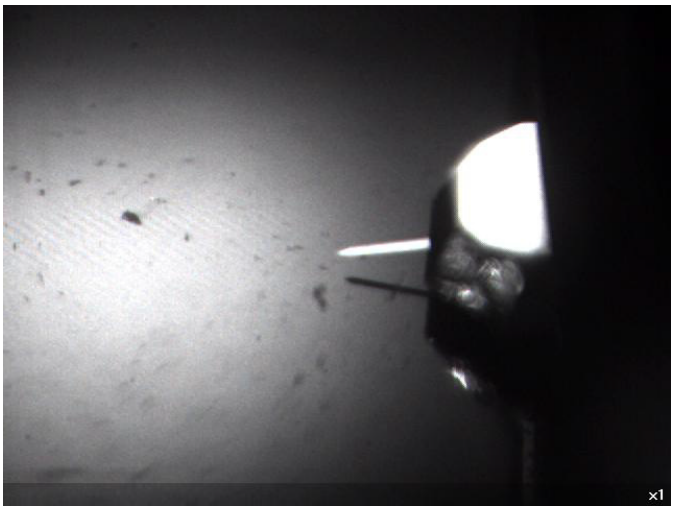

Figure 4.8: Side view of cantilever and sample after manual approach

2. **Automatic approach:** To prepare for the automatic approach first in the function selection bar of the software (see Figure 3.2 panel 4) the desired mode and the used cantilever need to be selected. As directly after the approach the scan is automatically started also the setpoint parameters for the scan (see section 4.5) need to be set beforehand. Once everything is set the automatic approach can be started by clicking on

Approach. The automatic approach brings the tip step by step closer to the sample by first extending the z-piezo until a large enough signal is detected and if this is not the case the z-piezo is moved back to the initial position and one step with the coarse movement is performed. This process is repeated until a large enough signal is detected. Then a window will show up with **Approach done**, the status light will turn green and in the standard setting the scan is automatically started.

4.5 Scan

The scan can be started, paused or stopped via the buttons in the imaging panel (see Figure 3.2). During the scan the feedback loop is typically turned on adjusting the z-piezo in such a way that a set setpoint is kept constant. The chosen setpoint depends on the measurement mode. In the static force mode the cantilever deflection can be set and in the dynamic force mode the amplitude can be set. The unit of the amplitude is in per cent, which refers to the amplitude far away from the sample, which would be 100 %.

4.6 Image processing and analysis

The data is stored in the format *.nid. For analysis the program WSxM is recommended (http://www.wsxm.eu) , however it is possible to use other programs such as Gwyddion. The *.nid files can be opened with these programs. Useful image processes in WSxM, which can be accessed via the icons or under the tab Process are: **Plane (Local)**, **Zoom** and **Profile**.

5. Exercises

5.1 Experimental

5.1.1 DVD

Measure the structure of a DVD in a) Static Force Mode and in b) Dynamic Force Mode.
Note: Before changing the cantilever measure its properties (see section 5.1.2.).

5.1.2 Cantilever

Measure the resonance curves of the two cantilevers (ContAl-G and Tap190Al-G) and the phase signal.

5.1.3 SBS-PS polymer blend on mica

Measure the topography and phase contrast using dynamic force mode and phase contrast imaging of a polymerblend of SBS (poly(styrene-butadiene-styrene) and PS (polystyrene).

5.2 Analysis

5.2.1 DVD

Determine the depth, width and length of the grooves. Compare both measurement techniques. Calculate the storage density of the given DVD.

5.2.2 Cantilever

Determine the resonance frequency and the q-factor of the two cantilevers. Present the frequency and phase shift curves in a proper range.

5.2.3 SBS-PS polymer blend on mica

Determine the location of the two different materials and mark them in the images.

6. Hints for preparing the report

- Recommended software for analysing the data is WSxM [12] or Gwyddion [13], which are freely available.
- Present both raw and optimized images.
- All relevant parameters should be either included in the image (e.g. scale bar, height scale) or in the caption (e.g. set point,...).
- Content of report:
  -Introduction
  -Theory AFM
  -Experimental setup
  -Experimental procedure
  -Results
  -Discussion and interpretation of results
  -Summary
  -Bibliography
- Please, hand in (electronically via email) the first version of your report to the supervisor within 4 weeks after the experiment. The supervisor will correct your report once and will send it back to you with comments and suggestions for revision. Please, hand in the final revised version no later than 6 weeks after the experiment, which will then be marked by the supervisor. Note, the oral exam approx. 45 min will be half of your mark and the report the other half. In case a report is handed in later than six weeks the initial draft will be marked or if no initial draft has been sent the experiment will be marked with not passed.

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