

Atomic Force Microscopy

Instructions to the Advanced Lab Course

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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 an example. Since this kind of microscopy is widely used in research areas dealing with structures on the nanometer scale, it might serve as a prototypical 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 Chapters 1 to 3 of this tutorial manual 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 on the internet. 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.

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

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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 scanning tunnelling microscopy (STM) [1, 2]. This technique exploits the fact that a tunneling current through a potential barrier of the width d is proportional to e^{-d} (provided a constant and final height of the potential barrier). Using a sharp tip close to the surface, the tunnelling current between the tip and the surface atoms in the vicinity of the tip can be measured by applying a voltage between the tip and the surface, hence scanning the height of the surface. The strong dependence of the current on the distance d allows to detect changes in the distance on a sub-atomic scale. To control such distances, electro-mechanical devices of high precision are required. This task is commonly solved by piezo-ceramics. Furthermore, it has to be taken into account that the tunnelling current is also a function of the chemical properties of the surface. For more details and information on the STM, see e.g. [3, 4].

In contrast to STM, not only conducting, but also non-conducting samples can be measured non-destructively and with high precision by an atomic force microscope (AFM) (also known as scanning force microscope (SFM)), which was developed in 1986 by Binnig, Quate and Gerber [5]. This makes AFM a useful tool not only for physical and chemical, but also biological applications [6].

1.2 The Scanning Force Microscope

The following section is mostly taken and adapted from [7].

Both the design and development of the AFM are very closely connected to those of the STM, as the central component of these microscopes is basically the same. A fine tip is positioned at a small characteristic distance away from the sample. The height of the tip above the sam-

ple is adjusted by piezoelectric elements. For STM, the tunneling current gives the information about the surface properties, whereas in AFM the interaction of forces between the tip and the surface are measured. 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.

In general, STM can sense a rich variety of forces. In the non-contact mode (for distances greater than 1 nm between the tip and the sample surface), van der Waals, electrostatic, magnetic or capillary forces are producing the images. Meanwhile, repulsion forces are dominating in the contact mode. Since the AFM does not require a current between the sample surface and the tip for its operation, it can move into potential regions inaccessible to the STM, such as samples which would otherwise be damaged irreparably by the tunnelling current of the STM. Insulators, organic materials, biological macromolecules, polymers, ceramics and glasses are among many materials that can be imaged in different environments, such as liquids, under vacuum or at low temperatures.

In the non-contact mode, the surface analysis with atomic resolution can be obtained. However, the sample has to be prepared under ultra-high vacuum (UHV) conditions in this case. It has been shown that it is possible to achieve a sufficient resolution in the tapping mode (a modified non-contact mode) under ambient conditions, similar to STM investigations, single vacancies or their agglomeration [8]. Besides this, a non-contact mode has one further advantage over the contact mode: The surface of a very soft and rough materials is not influenced by frictional and adhesive forces as opposed to scanning in contact mode, i.e. the surface is not “scratched”. Additionally to height profiles, state-of-the-art AFM allows for the imaging of nano-mechanical properties, such as adhesion or Young’s modulus.

2 Basics

2.1 Theoretical Principles

The following subsection are mostly taken and adapted from [7].

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 a 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 ($\propto d^{-7}$). This arises from the fact that the interaction between dipole moments occurs through the exchange of virtual photons, as indicated in Fig. 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 com-

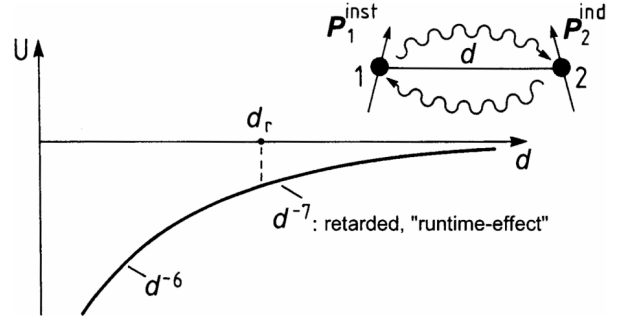


Figure 2.1: Van der Waals potential U between two atoms. d_r is the critical distance above which the transit time effects weaken the interaction. Figure is taken unchanged from [7].

parison 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 are:

- **Single atom over half space**

A single atom at a distance d relative to the half-space (see Fig. 2.2) leads to an interaction potential U of

$$U = -\frac{C\pi\rho}{6} \frac{1}{d^3}, \quad (2.1)$$

where C is the interaction constant of the van der Waals potential and ρ 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 a distance d (distance between the sphere surfaces, as shown in Fig. 2.2), one obtains an interaction potential U of

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

where H is the so-called Hamaker constant. It can be defined for a van der Waals body-body interaction as $H = \pi^2 \times C \times$

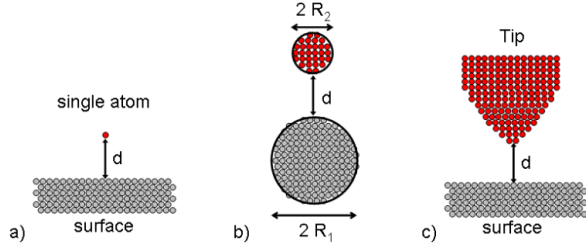


Figure 2.2: Schematic illustration of the three described interactions cases: **a)** sphere with half-sphere, **b)** two spheres and **c)** sphere (of a tip) with half-space.

$\rho_1 \times \rho_2$ with ρ_1 and ρ_2 as the number of atoms per unit volume in the two interacting bodies and C as the coefficient in the particle-particle pair interaction [9]. It 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-sphere**

If a sphere with a radius R has a distance d from a half-sphere (see Fig. 2.2), an interaction potential U of

$$U = -\frac{HR}{6} \frac{1}{d} \quad (2.3)$$

is obtained from Eq. 2.2. This case describes the geometry in a SFM best and is therefore 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 STM to achieve a high resolution of the SFM. 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 due to

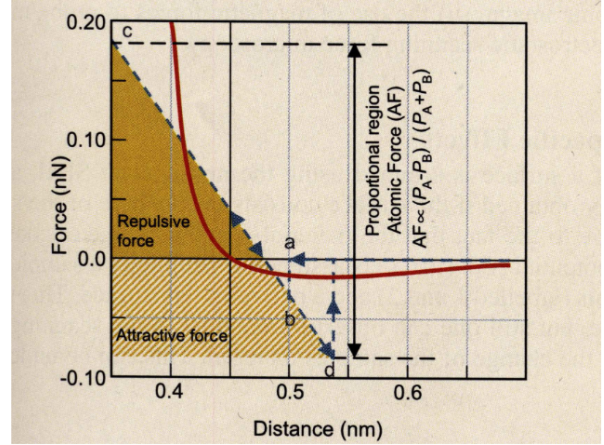


Figure 2.3: The cantilever force (dashed line) as a function of the tip-sample distance. Figure is taken unchanged from [7].

attractive forces is negligible. After the cantilever is brought closer to the surface of the sample (at point “a” in Fig. 2.3), 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 (at points “b-c” in Fig. 2.3). Within this range, the position of the laser beam on four-quadrants of the photo diode (see Section 2.2.1), which is proportional to the force, is a linear function of the distance. On reversal, this eristic shows a hysteresis. This means that the cantilever loses contact with the surface at a distance (at point “d” in Fig. 2.3), which is much larger than the distance on approaching the surface (at point “a” in Fig. 2.3).

In general, the forces acting on the flexible cantilever lead to a bending of the cantilever following Hooke’s Law (see Fig. 2.4):

$$F = -\Delta x \cdot k, \quad (2.4)$$

where Δx is the displacement and k the spring

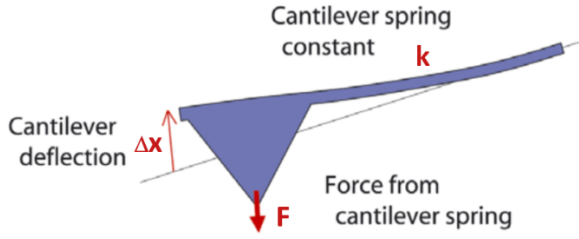


Figure 2.4: A schematic of the cantilever illustrating the Hooke's law. Figure is taken unchanged from [10].

constant of the cantilever [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 to obtain the mechanical properties of the surface, e.g. Young's modulus or adhesion force, the so-called Derjaguin-Muller-Toporov model (DMTM), describing the adhesive contact mechanics of touching solids, is used [11]. The force on the tip F_{tip} is then given by

$$F_{\text{tip}} = F_{\text{adh}} + \frac{4}{3}Y\sqrt{rd^3}, \quad (2.5)$$

where r is the tip radius and d the indentation. Therefore, by measuring the force on the tip, both the adhesion force F_{adh} and the reduced Young's modulus Y can be calculated via a fit.

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 and dynamic modes. Another classification could be contact, non-contact and intermittent-contact modes, which is related to the working regimes (see Figure 2.5). In the following, the three commonly used techniques, namely static mode (contact mode) and dynamic modes (non-contact mode, tapping mode and peak force tapping mode) will be shortly described. Further information can be found in textbooks or review articles.

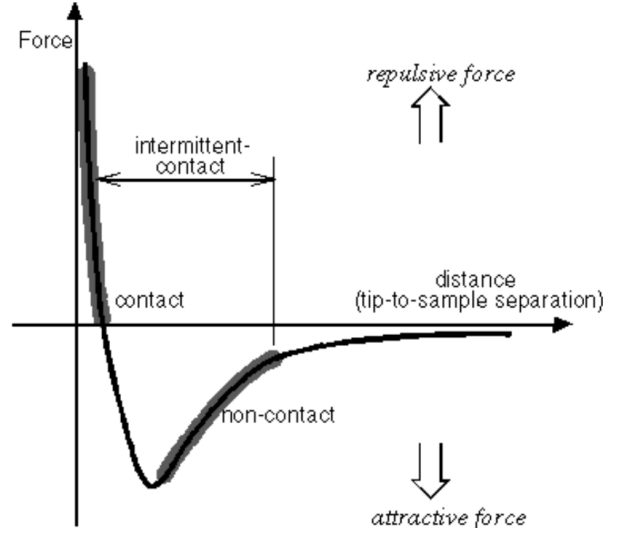


Figure 2.5: A typical force-distance curve with added classification of the AFM operation modes within the working regime of the van der Waals potential.

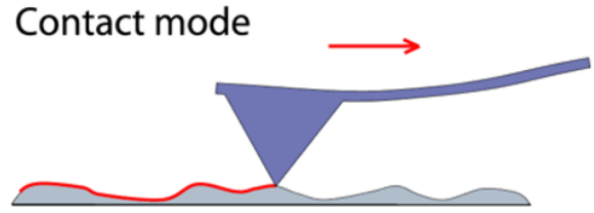


Figure 2.6: Schematic of the contact mode of an AFM. Figure is taken unchanged from [10].

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, the frictional forces appear which can be destructive and damage the sample or/and the tip. A representation of this measuring mode can be found in Fig. 2.6.

Constant height mode: This mode is particularly 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 (de-

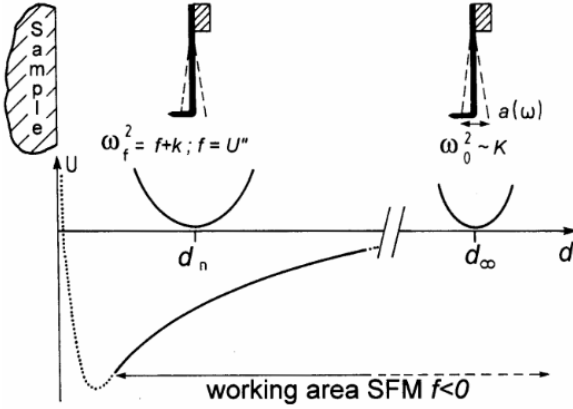


Figure 2.7: A schematic representation of the effect of the van der Waals interaction potential on the vibration frequency of the spring with a tip. As the tip approaches the surface, the resonance frequency of the leaf spring is shifted. Figure is taken unchanged from [7].

scribed in Section 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 (Fig. 2.7). 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 Fig. 2.8. In Fig. 2.8, ω_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 sim-

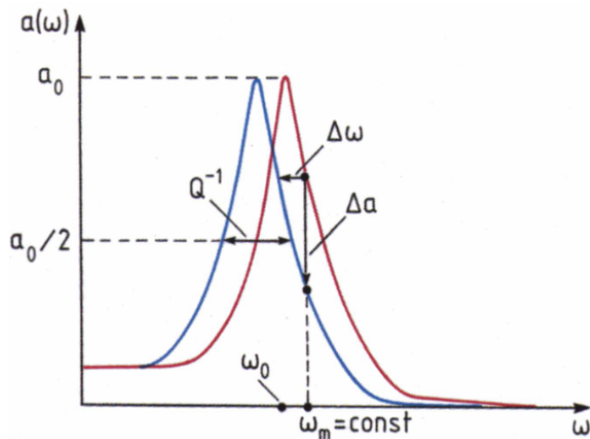


Figure 2.8: Resonance curves of the tip with and without a van der Waals potential. The interaction leads to a shift of the resonance frequency $\Delta\omega$ with the consequence that the tip excited with the frequency ω_m has a vibration amplitude $a(\omega)$ attenuated by Δa . Figure is taken unchanged from [7].

ilar 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. An illustration of this mode is shown in Fig. 2.9.

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 Chapter 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

Intermittent contact

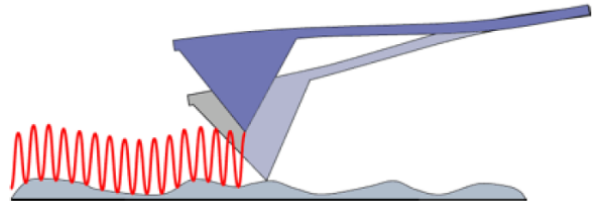


Figure 2.9: Schematic of the dynamic force mode, here intermittent contact mode (tapping mode) of an AFM. Figure is taken unchanged from [10].

properties (e.g. adhesion) in addition to the topography of the sample surface.

2.2 Operation Principles

2.2.1 Optics

The AFM reconstructs the topography of a material components by analysing the deflection of the tip at the end of a cantilever (spring). Today, the most common method for detecting the deflection of the cantilever is by using an optical lever detection method which includes measuring the position of a reflected laser-beam on a photosensitive detector.

The back of the cantilever is coated with a reflective metal like aluminium or gold such that the laser is reflected off the back and falls off on a mirror which then reflects it on a photodiode. In this particular AFM, a four-quadrant photodiode is used, as indicated in Fig. 2.10.

The photodetector generates a voltage proportional to the amount of light on it. When the light spot is centred, the voltage in the upper and lower half of the photodetector is equal; hence, the deflection is zero. Upon changing the cantilever angle, the signals in the top and the bottom halves changes, and one observes a non-zero deflection. This relative change in deflection is then translated to height profiles in AFM. The signals on the left and the right of the detector give a measure of the twist of the cantilever.

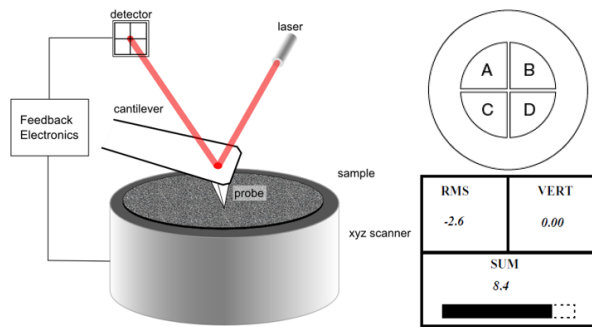


Figure 2.10: **Left:** A schematic illustration of the optical setup present in the AFM. **Right:** A schematic illustration of the used four-quadrant photo-diode on top and a representation of SUM signal at the AFM base on bottom.

While setting up the calibration in AFM, one would observe an increased signal at the electronic output of the detector located at the AFM's base in case of the laser spot falling on the detector. This output is quantified as SUM, which indicates the total voltage generated by the photodetector. It is recommended to perform calibration in the **AFM** & **LFM** mode, such that one would get the voltage difference between the top and bottom segments as well as the left and right segments. Depending on the cantilever used, the SUM should be between 4.0 – 9.0 V. In general, a SUM signal of above 7.0 V is considered adequate for the tips used during the FP.

In TM AFM mode, RMS amplitude value is displayed in Volts instead of the horizontal signal. This is because, as the cantilever oscillates in the air at its resonant frequency in the tapping mode, the reflected laser beam is deflected in a regular pattern over the photo-diode, hence generating a sinusoidal electric signal which is converted to the RMS value.

2.3 Feedback control

For the realisation of a SFM, the force measurement must be supplemented by a feedback control (see Fig. 2.11), in analogy to the STM. The

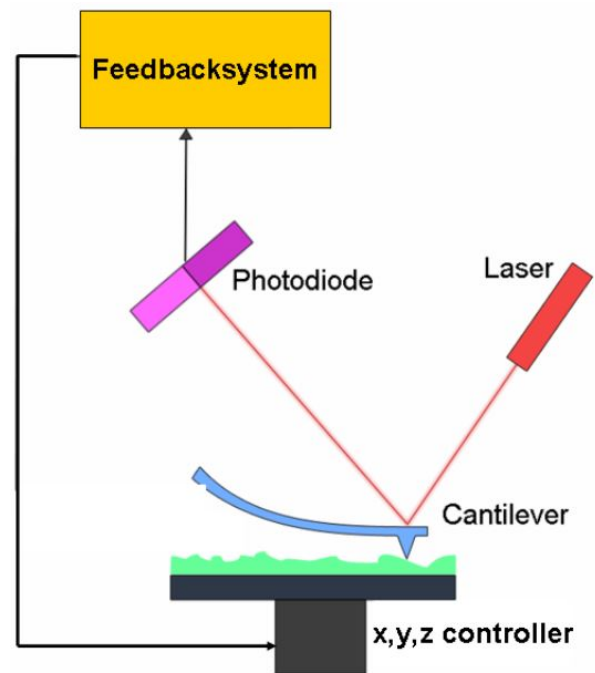


Figure 2.11: A schematic illustration of the feedback loop and the optical system.

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 toward the cantilever until the controlled quantity has reached the setpoint value again. *The scanning 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 on the surface.

2.4 Preparation Tasks

- What are the underlying principles of the AFM? What forces act between the 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 behaviour*: 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 controls 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 on 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). Here you can find useful information.

3 Experiments and Evaluation

The goal of these experiments is to learn about the basic functioning and possibilities of the AFM. The structures of two calibration grids and different surfaces will be observed and quantified with respect to their 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 ??.

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 also should use this time to do your evaluation.

3.1 P and I values

Start the microscope as described in Chapter 4.1 and take a screenshot of the resonance curve. For your first sample, use the calibration grid TGZ02 (see Figure 3.1).

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 both too high and too low values for I , respectively.

Images that have to be taken:

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

Discuss the effect of high/low proportional and integral gains 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.

3.2 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 the 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 possible reasons for your observations.

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

Afterwards, a new tip will be given to you by your supervisor. First record the new resonance curve and then re-investigate the grid TGZ01 (scanning direction perpendicular to the steps).

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

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

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

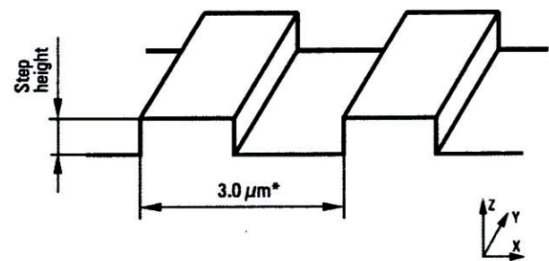
TGZ03

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 3.1: Data sheet for the calibration grids TGZ01 and TGZ02

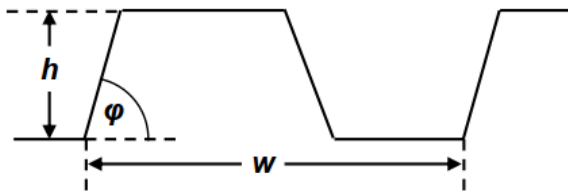


Figure 3.2: Values to be determined in the evaluation

In order to analyse the grid structure, use the three images with perpendicular scanning direction and determine the step width w , step height h and the angle φ (see Figure 3.2).

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.

Secondly, compare the values obtained for TGZ01 with the new and the old tips. 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, consider using the resonance curve determined in 3.1. Are there any visible changes/differences?

3.3 Samples

3.3.1 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 ($10\mu\text{m} \times 10\mu\text{m}$)

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?

3.3.2 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 continuously 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. $1.5\mu\text{m} \times 1.5\mu\text{m}$)
- Stripe from bright side (approx. $1.5\mu\text{m} \times 1.5\mu\text{m}$)

Characterize the structures you find (depth, 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!

3.3.3 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 ($10\mu\text{m} \times 10\mu\text{m}$) of each sample, think about the best scanning direction (parallel or perpendicular to the spiral lines) to determine the length of a pit. Images:

- CD surface with best scanning direction (10 μ m x 10 μ m)
- DVD surface with best scanning direction (10 μ m x 10 μ m)
- Blue-ray Disc surface with best scanning direction (10 μ m x 10 μ 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. Estimate the capacities of a DVD and a Blue-ray Disc. Is the data storage organized in the same way as for the CD (hint: check for the EFM mechanism)?

3.3.4 Burned CD

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 μ m x 10 μ m).

- Burned CD surface with best scanning direction (10 μ m x 10 μ m)

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

3.3.5 Additional Samples

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

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 [12]. 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

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 figures 4.2,4.3,4.4 and 4.5
6. Use a tweezer to place the sample on the magnetic sample holder above the piezo. **If the sample is thicker than the previous one it might be necessary to bring up the stage**
7. If a new cantilever is needed: Ask your tutor for a new one.
8. **Is the distance between sample and AFM head enough? Pay attention and make sure that the tip does not touch the surface!** Place the cantilever holder in the AFM head and fix it with adjustment 1 (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.
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 figure 4.5). **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. In the next step, the tip is brought down to the sample. **Attention: When done wrong this can destroy the tip!** To start, the microscope must be focused on the surface, at the position where the tip is. Then move **up(!)** the microscope a bit so that the surface is no longer focused. As a last step move down the tip until the tip is in focus and stop.
17. Use the **Engage** button (see figure 4.3) to make the tip contacting the surface. 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.

4.2 After the measurement

After a measurement has finished, click **withdraw** to disengage the tip from the surface. After that, the sample can be moved to have a look at another region. The sample or tip is removed the following way:

1. Bring up the tip to ensure that there is enough distance between the surface and the tip.
2. Release the cantilever holder with adjustment screw I.
3. Remove the cantilever holder and position it in a way that you cannot accidentally touch the tip.
4. Remove the sample with a tweezer.

4.3 Data evaluation

The data is evaluated with the Nanoscope analyser. It has many functions, most of them are rather self-explanatory. The most important functions are **Plane fit** and **Section**, which can both be found in the top panel. **Plane fit** allows to select different parts of the Surface that should have the same height to flatten the data. This should be performed at the beginning of every image evaluation. **Section** is the easiest way to measure the height profile along a straight line. If you want to evaluate the data at home, Gwyddion is a free software that can also be used for SPM data evaluation. It has comparable and sometimes even more functionality.

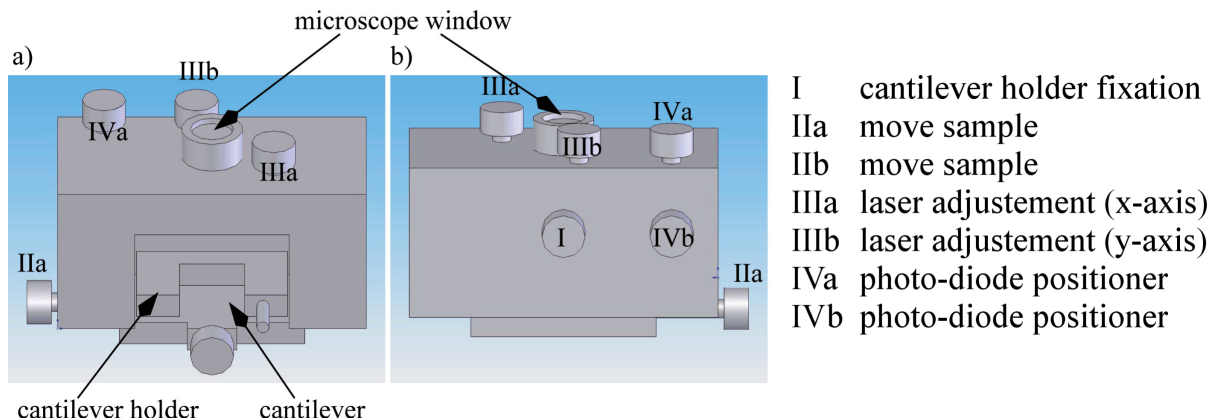


Figure 4.1: Description of the NanoScope toolbar buttons for the experimental workspace handling in the main screen.

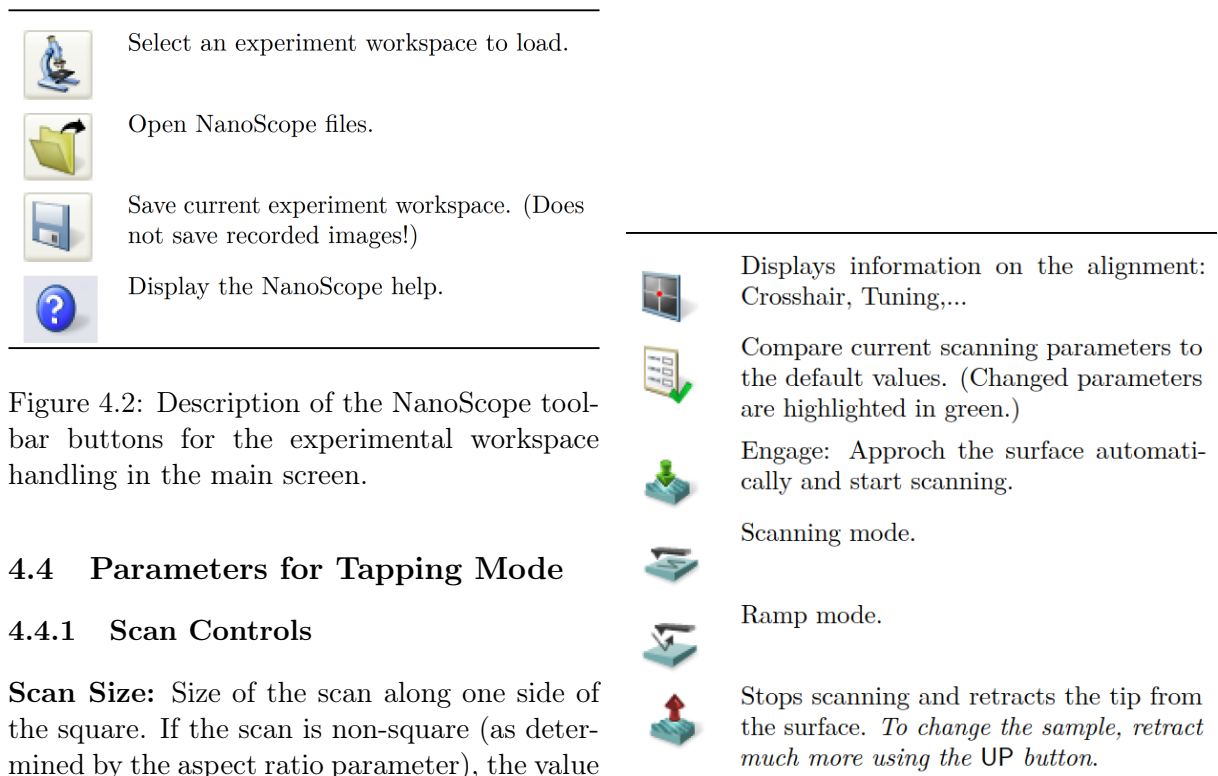


Figure 4.2: Description of the NanoScope toolbar buttons for the experimental workspace handling in the main screen.

4.4 Parameters for Tapping Mode

4.4.1 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: $\approx 10 \mu\text{m}$.

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 $\approx 10 \mu\text{m}$,

Figure 4.3: Description of the workflow toolbar 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.

Figure 4.4: Description of the NanoScope tool-bar capture buttons in the main screen.

depending on the scan size (piezo deflection in x- and y- direction is max. $\approx 10 \mu\text{m}$).

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



















	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.

Figure 4.5: Description of the NanoScope tool-bar buttons for scanning in the main screen.

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.

4.4.2 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

4.4.3 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 or 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.

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