# F29: Atomic Force Microscopy - Short Evaluation

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## Abstract

**In this experiment the physical basics and variety of modes an Atomic Force Microscope (AFM) provides are tested. In the first part, the influence of technical parameters and the limits of resolution of the AFM are investigated. Afterwards, the AFM is used for scanning the surface of different objects, e.g. optical data storage media and a camera's CCD chip.**


# 1 Theoretical Basics

## 1.1 Introduction to SPM

Generally, scanning probe microscopy (SPM) can be performed in several ways [1]: For example, a scanning tunnelling microscope (STM) measures the tunnelling current between a tiny tip (cantilever) and the surface of the investigated object to deliver information about the height profile [2]. In this experiment we will use a atomic force microscope (AFM) which gains information about surface of objects by measuring the force between the tip of the microscope and the surface. Apparently, the SFM does not require a conducting surface as the STM to carry out measurements and it cannot damage the sample by applying too high voltages. Instead, the SFM senses the electromagnetic forces (e.g. van der Waals or electrostatic forces) following from approaching the sample's surface with a cantilever.

## 1.2 Van der Waals interaction

On short time scales the positive nucleus and the electrons of an an atom form a dipole due to the electrons' movement. These short-lived dipoles interact with the atoms in the environment so that an attractive force is created. For more than two atoms attracting each other by this so-called van der Waals interaction the whole force between to objects can be calculated by an integral. The tip above the sample can be approximated as a sphere with the radius R and the atom number density $\rho_1$ while the sample below can be seen as a half-space with the density $\rho_2$. The van der Waals potential $U_{vdW}$ which depends on the distance d between these two is given by

$$U_{vdW} = -\frac{\pi^2 C \rho_1 \rho_2}{6} \cdot \frac{R}{d}. \tag{1}$$

C is a constant quantizing the strength of the interaction.

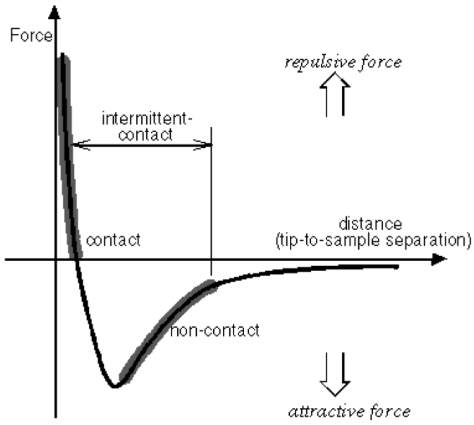



Figure 1: Force-distance curve showing the non-contact distance where van-der-Waals force is attracting and the repulsing Pauli barrier in bold respectively. Taken from [2]

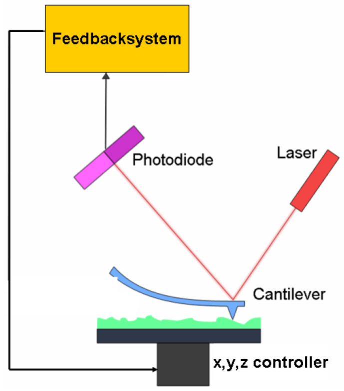



Figure 2: Experimental setup: The sample (green) is investigated by the cantilever (blue). Its height is measured by the laser and photodiode system (red and violet) providing input for the feedbacksystem that controls the motion of the sample. Taken from [2].

## 1.3 Force distance behavior

Although the van der Waals interaction attracts two objects, Pauli's principle leads to an repulsive force that outweighs the attraction for short distances. The force, dependend on the distance, is shown in figure 1. If the tip approaches the surface, the attraction pulls it to the surface. As the force's sign changes (see figure 1) the tip touches the surface. For closer distances the tip experiences a strong repulsion. On its way out of the sample the tip does not lose contact to the surface at the same cantilever position as where it started touching it during approaching but loses contact at a slightly higher position. This hysteresis is due to adhesion forces of the surface.

## 1.4 Operation Modes

In general, the microscope can be driven in static and dynamic modes. In the first one either the distance to the sample or the force on the tip is kept constant by a feedback circuit and the profile information is extracted from the deflection of the cantilever or the movement of the tip respectively. The cantilever's deflection needs to be measured in scales of 0.001 nm. A laser combined with photo diodes is used for this task. The rough setup of the experiment is shown and explained in figure 2.

In the dynamic modes the cantilever is forced to oscillate either at a certain frequency $\omega$ or with a certain amplitude $a(\omega)$. If the distance to the sample is large enough, the behavior is simply a harmonic oscillator with a typical Lorentzian resonance curve. For shorter distances the interaction between the sample and the tip influences the oscillation leading to a shift in the resonance curve. Therefore, either the amplitude changes if the frequency is kept constant or the frequency changes if the amplitude is kept constant. The information about the sample's profile are then gained by these changes.

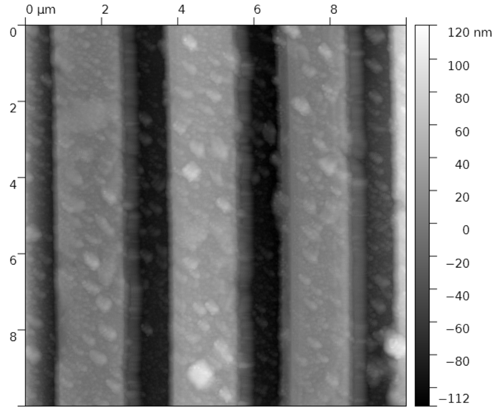



Figure 3: Picture of sample TGZ02 taken with an old tip and the best P and I values. The line structure is clearly visible

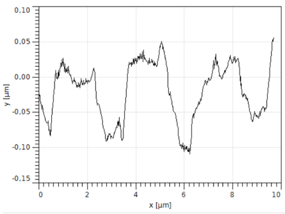
Dynamic methods split up in non-contact mode, tapping mode and peak force tapping mode which use slightly different distance and frequency adjustments. During the experiment, the last two will be chosen since they provide adequate precision with a reasonable low risk of damaging the sample. The peak force tapping mode taps the sample just like in the tapping mode but uses far lower frequencies. Additionally, it records a force-distance curve for every pixel in which the maximum force can be measured and used as parameter for the feedback circuit.

# 2 Measurements and Results

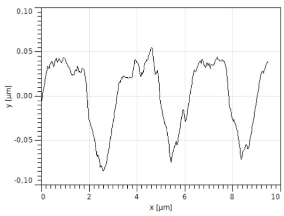
## 2.1 P and I Values

At first, a resonance curve was measured which looks like a typical Lorentzian curve with a resonance frequency at $\omega_0 = (383.85 \pm 0.50)\,\mathrm{kHz}$.

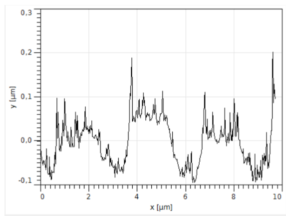
In the tapping mode the height of the sample TGZ02 is investigated using a PID controller. The respective resistance value to determine how much the proportional (P) and the integral (I) controlling elements contribute to the total controlling were varied to find the best combination. $P = 5$ and $I = 9$ were chosen to deliver the best results which can be seen in figure 3. The impact of different I values on extractable height profiles are visible in figure 4 a)-c). Too low I values smooth the profile so that the profile which looks almost like a rectangle becomes triangular while too high I values show a lot of details swallowing the main structure. In the pictures badly chosen I values caused a decrease of contrast.



(a) I = 9



(b) I = 1



(c) I = 30

Figure 4: Height profiles with different I values, P = 5. Too high I values show too many details while too low ones swallow them

Variations of the P value were executed as well and it turned out that mainly the ratio of P and I values impacts the results rather than the absolute values. P and I values were optimized for every new sample and tip in this experiment.

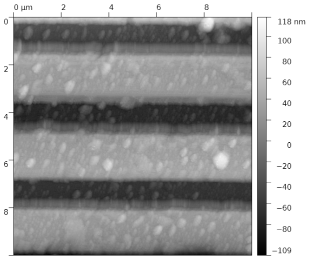


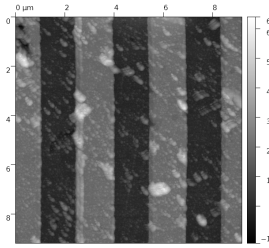
Figure 5: Picture of sample TGZ02 taken with an old tip, parallel scanning provides poorer resolution

## 2.2 Tip characterisation and limitations of the AFM

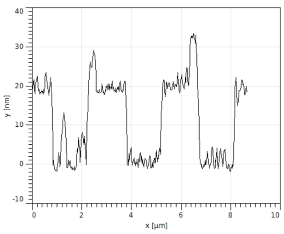
Next, two different samples and two different tips (old and new) were used for the measurements. Perpendicular scanning to the line structure gives clearer and better measurable results, since the higher and lower parts of the lines are easier found if the tip confronts them directly. Parallel scanning supports smearing of the contrast and therefore lowers the picture's quality as can be seen in figure 5.

The second sample TGZ01 shows the same structure as TGZ02 before. This can be seen in figure 6. Using a new tip delivered optically even better results which becomes obvious when comparing figure 6 a) - d) although the sample shows huge artefacts and seems to be seriously damaged.

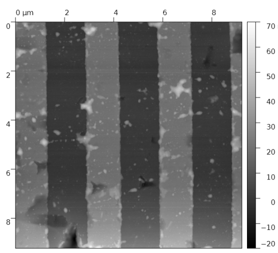
To determine the structure values height h, width w and angle $\phi$ (shown in figure 7) several measurements were taken and the statistical means and errors were calculated. The results are shown in table 1. $h_x$ is the width where the profile changes its height i.e. the slope is not 0. The width of the lines is - as expected from the pictures - only slightly shorter in the TGZ01 sample, while its height is much lower. The new tip got the angles more precisely wich could be explained by a sharper tip. However, during investigating the height structures in TGZ01 of only a few Nanometer the limits of resolution either of the software or the tip were reached, what means: When measuring the $h_x$ value only three different values in the range from 78 nm to 102 nm were measured. This observation shows that the limits of resolution were reached and explains the large errors for $h_x$ as well.



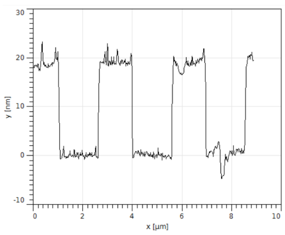
(a) Old tip



(b) Old tip



(c) New tip



(d) New tip

Figure 6: Pictures and height profiles with old and new tip which clearly delivered better results.

| TGZ- sample | $w$ [$\mu$m] | $h$ [nm] | $h_x$ [nm] | $\phi[\circ]$ |
|---|---|---|---|---|
| 02 | $2.98 \pm 0.01$ | $101.8 \pm 1.7$ | $228 \pm 9$ | $24.06 \pm 0.93$ |
| 01(old) | $2.92 \pm 0.03$ | $22.1 \pm 0.9$ | $94 \pm 5$ | $13.23 \pm 1.16$ |
| 01(new) | $2.89 \pm 0.01$ | $24.3 \pm 1.7$ | $79 \pm 5$ | $17.10 \pm 3.48$ |

Table 1: Parameters of the line structure in TGZ01 and TGZ02.

A comparison of the resonance curves of the old and the new tip shows that the new tip's resonance frequency is lower ( $\omega_0 = (301.10 \pm 0.50)$kHz) than the old one's. The

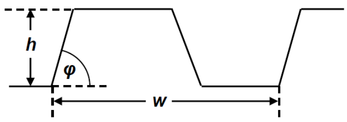



Figure 7: Height profile structure of TGZ01 and TGZ02. Taken from [2].

resonance frequency of a harmonic oscillator grows as its mass is reduced. The mass reduction of the tip may have an impact on the difference of the resonance frequency in the same way.

## 2.3 CCD Chip

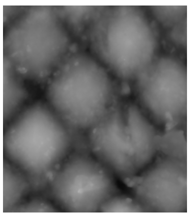


Figure 8: Picture of a camera's CCD chip.

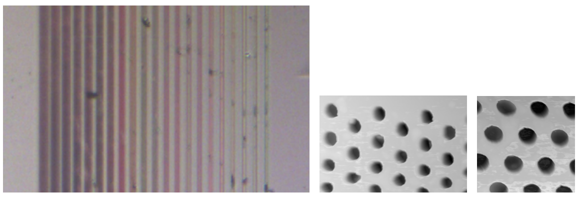
After taking a picture of a camera's CCD chip (figure 8) we measured the size of one pixel which includes four rough squares in the picture, since the chip uses four detectors to cover the whole range of wavelengths for the visible light. The area of one pixel was measured as $(2.52 \pm 0.04) \cdot 10^{(-11)}\mathrm{m}^2$. After determining the size width and height of the whole chip we calculated the total number of pixels on the CCD chip which was found to be

$$N_{pixel} = \frac{A_{total}}{A_{pixel}} = (1.07 \pm 0.15) \cdot 10^6. \quad (2)$$

Although modern camera's or even smartphones deliver pictures in significant higher qualities one needs to consider that resolution of CCD chips has enormously increased in the past years so that the result above is reasonable for a chip produced a decade or longer ago.

## 2.4 Nano Lattice

The AFM also allowed us to measure the size of holes in a Nano Lattice which shows tiny lines of holes with different sizes reflecting different colours and making the lattice look similar to a rainbow. The main aspect we learn from these measurements is that the brighter lines (orange - red) are due to smaller holes than the darker, blue stripes. Smaller holes cause larger distances in between the holes, since the hole density is kept constant throughout all stripes. These distances in between the holes, namely the areas in the stripes with a flat surface, are assumed to work like an optical lattice because larger distances reflect waves with larger wavelengths similar to the double slit.



(a) Nano Lattice (b) Picture of a bright stripe (c) Picture of a dark stripe

Figure 9: Pictures of the Nano Lattice (a) and scans of the holes in the bright and dark stripe.

## 2.5 Optical Data Storage Media

After taking pictures of optical data storage media like CDs or DVDs one can measure the size of the structures which become visible in the pictures. The information on these media is encoded in pits and lands in a polycarbon foil. A change from pit to land or vice versa symbolizes a 1 in binary code and no change a 0. If the way the data on the CD is modulated is considered one can measure the size of one bit on the medium and therefore, calculate the total data capacity. For instance, the width of the spiral-shaped lines of the pits and lands is approximately $w_{bit} = 1190\text{nm} \pm 15\%$ and the length of one bit can be calculated as $l_{bit} = 550\text{nm} \pm 20\%$. The largely estimated errors are caused by the low quality of the picuture and variance in bit sizes. Using these values the capacity of a whole CD can be estimated as following:

$$\begin{aligned} N_{data} &= \frac{\pi(r_o^2 - r_i^2)}{w_{bit} \cdot l_{bit}} \cdot \frac{1}{8 \cdot 10^6} \cdot \frac{8}{14} \\ &= 935\text{MB} \pm 21\% \end{aligned} \tag{3}$$

The last factor is due to the Eight-to-Fourteen-Modulation [3]. The smaller sizes of bits on DVD and Blu-Rays cause correspondingly larger capacities.

# 3 Summary and Critical Comment

In this experiment the atomic force microscope and its different modes to scan surfaces were become acquainted. Thereby, the previous expectations about the resonance and force-distance curves were proved. Investigations on two calibration grids showed the precision and the limits of the resolution of the microscope as well: The experimental setup could not deliver measured values precisely anymore as soon as we approached length scales of a few nanometers. The parameters from table 1 are decent taking into account their three sigma environments and the given data sheet about the calibration grids as well.

Moreover, it turned out that perpendicular scanning to the interesting structures was the best choice to get sharp shapes in the pictures, as one would intuitively assume.

During the investigation of micro structures of a CCD Chip and the polycarbonat foil of optical data storage media all calculations led to reasonable results about the resolution and the data capacity respectively. One Megapixel seems to be quite low compared to modern standards but the chip was probably produced several years ago. Exact determination of the data capacity of CDs etc. was complicated by the poor picture quality and lack of sharp structures.

Furthermore, some issues arised during placing the tip into its mount in the beginning.
Lastly, the microscope was not able to engage the sample's surface properly anymore and showing an error in the corresponding software instead. That is why the last measurements were aborted.
However, he experiment enabled us to gain extensive knowledge about the basic methods of an AFM.

# References


[1] `https://en.wikipedia.org/wiki/Scanning_probe_microscopy`

[2] J. Vogt. *Atomic Force Microscopy F29.* revised in 2017. `http://www.kip.uni-heidelberg.de/ag_pucci/teaching/afm`

[3] `https://de.wikipedia.org/wiki/Eight-to-Fourteen-Modulation`