Atomic Force Microscopy

Contents

Atomic Force Microscope

In an Atomic Force Microscope (AFM), one type of Scanning Probe Microscope the force acting between a probe and the sample is measured. After developing the Scanning Tunneling Microscope (STM) Gerd Binnig and Heinrich Rohrer continued to develop another scanning probe microscopy method the atomic force microscope (AFM), since only conductive samples could be probed with the scanning tunneling microscope [1]. Instead of the tunneling current, the force acting between the sample and the probe is monitored. Binnig and Rohrer used the same high precision scanners to determine the position of the probe, but used different probes.

Scanning probe microscopy is a general term that describes imaging methods that form images using a physical probe instead of optical or electrooptical methods. The sample is scanned and at discrete points measurements are taken. All the measurements are put together to assemble a digital picture.

Theory

In an atomic force microscope a cantilever, clamped on one end with a needle-like tip attached to its free end, is used to sense the forces acting between the probe and a sample of interest. A laser, focused on the backside of the cantilever is used to sense the deflection of the cantilever from its equilibrium position as shown in figure A1. This technique of

detection is called the optical beam deflection method and is the most common technique to sense cantilever bending. Four segment photodiodes allow the detection not only of normal bending but also of torsional deformation caused by lateral forces acting on the tip. In the following description the equilibrium position of the cantilever will be referred to as $h(t)$. The gap between the cantilever tip and the sample is expressed by $z(t)$ leading to the description of $d(t) = z(t) - h(t)$ for the deflection of the cantilever from its equilibrium position.

An atomic force microscope can be operated in a lot of different environments. Samples can be probed in air or liquid, as well as vacuum. It can also be combined with other measurements to quantify different physical parameters such as the work function, at the same time.

Since forces acting between the tip and the sample are generally more long ranged than the tunneling current, the resolution that can be achieved is usually lower than in scanning tunneling microscopy. Nevertheless it is also possible to achieve atomic resolution in atomic force microscopy, but this requires specialized equipment.

Atomic force microscopy measurements can be performed in different modes, which are either static, or performed with an oscillating probe tip. One has also to distinguish between imaging and spectroscopy modes. Images are obtained in constant height mode or with a feedback control to keep the force-, amplitude-, phase- or frequency constant. In spectroscopy mode one is interested in converting the observables of an atomic force microscopy experiment into a tip sample interaction as a function of the tip-sample distance.
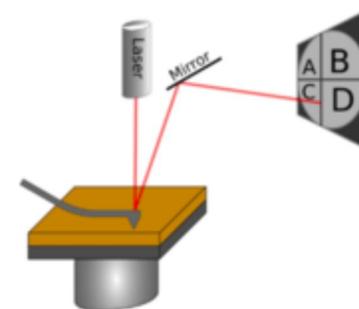

Figure A1: Schematic setup of an atomic force microscope utilizing the optical beam deflection method to determine the cantilever bending. Lateral displacement of the beam is derived from $(I_A + I_C) - (I_B + I_D)$, the in plane component can be derived from $(I_A + I_B) - (I_C + I_D)$.

Relevant Forces and Interactions

The tip-sample interaction is the result of the superposition of different forces. Forces can be categorized by their range of interaction and differ in their origin. If the range of interaction reaches from 1-100nm, the force is called long range. Sub-nm forces are referred to as short-range forces. As a result of the spatial range, the number of tip atoms which significantly contribute to the interaction varies. The number of tip atoms contributing to short-range interactions is usually small compared to the number of atoms contributing to long-range interactions.

Pauli Repulsion

At very small tip-sample separations the Pauli principle leads to a repulsive fore. The quantum mechanical Pauli exclusion principle states that two identical fermions, e.g. electrons, cannot occupy the same quantum state simultaneously.

Van Der Waals Forces

The long ranged electric Van der Waals interaction occurs between all atoms and molecules and is given by : $F_{VdW} = \dfrac{A_H R_p}{6d^2}$ for a sphere or particle (radius R_p) interacting with a flat, planar surface over the distance d. The Van der Waals force originates from dipole moments that can either be induced or permanent. Only when averaged over time a fully occupied electron shell has no net dipole moment. Fluctuations of the electron density in particles cause dipoles that polarize adjacent molecules and induce dipoles depending on their dielectric properties. This means, that even noble gases exhibit Van der Waals forces in their liquid state. In the case of a particle interacting with an infinite surface, Van der Waals forces show a quadratic decay with respect to the distance. The van der Waals interaction depends on the surrounding medium as well as the interacting phases. It is to note that the medium dependent Hamaker constant which contributes to the van der Waals force linearly is in the order of 10−19 J[2] in air whereas in water it is 2.2 × 10−20 J[3] for the in- teraction between two mica surfaces for example.

Electric Forces

At larger tip-sample separations the long range electric forces dominate. The quadratic dependence of electric forces with respect to distance can be described by Coulombs law and originates from static surface charges, differences in the work function or an externally applied voltage. In addition to the tip-sample force, the cantilever itself can contribute to the interaction with the sample due to the long range of interaction. Electrostatic forces can either be attractive or repulsive depending on the algebraic sign of the charge. As an approximation the system sample-interspace-tip can be described as a capacitor with the capacitance C, where the voltage ΔV is applied to it. The potential difference $\Delta V = V_{tip} - V_{sample}$ can originate form static surface charges,differences in the work function or an externally applied voltage.

Capillary Forces

Capillary forces originate from a thin layer of water located on the surface of the investigated surface when measuring in air. The layer occurs at room conditions in every laboratory, while the thickness depends on the humidity and the surface properties. By forming a liquid meniscus between tip and sample the water provokes an additional attractive force as soon as the tip is in contact with the liquid.

Quasi static atomic force microscopy

Quasi static atomic fore microscopy images or contact mode atomic force microscopy images describe an imaging technique where the tip scanned across the surface while monitoring the bending of the cantilever. Measurements are mostly performed in the repulsive regime, which means the tip needs to be in firm contact with the surface. Physical contact always causes damage or at least temporary deformation of the sample and wear-out on the tip apex. An additional obstacle is caused by the force gradient of the attractive tip-sample interaction while approaching the surface. When the tip is positioned far away from the surface, no force is acting on the cantilever figure A2(1)(5). If the force exceeds the restoring force of the cantilever, the lever bends towards the surface to jump into contact which is called "jump-to-contact" figure A2(2). While further approaching, the cantilever gets bend away from the surface figure A2(3). While retracting the cantilever, adhesion forces acting between the sample and the probe also cause a bending of the lever towards the surface. As soon as the cantilever restoring force exceeds the adhesion forces, the contact is lost abruptly which is called *snap-out" figure A2(4).* This disparate behavior causes a hysteresis which shows the

mechanical instabilities of the cantilever. In addition the area marked in green indicates, that the surface was plastically deformed.

In imaging mode the hysteresis is not influencing the measurement, since it is performed in the repulsive regime entirely. For force spectroscopy however, where the cantilever deflection is monitored as a function of the tip-sample distance, it is huge drawback. In the area of interest close to the surface a stable measurement can not be performed.

Using cantilevers of higher stiffness could improve the stability but would make the measurement less sensitive at the same time. The analysis of the force distance curves however is straightforward, since the cantilever can be treated like a Hooks law: $F_{TS} = kd$ Other than using cantilevers of high stiffness, an other option is to switch to dynamic methods to probe the tip sample interaction.
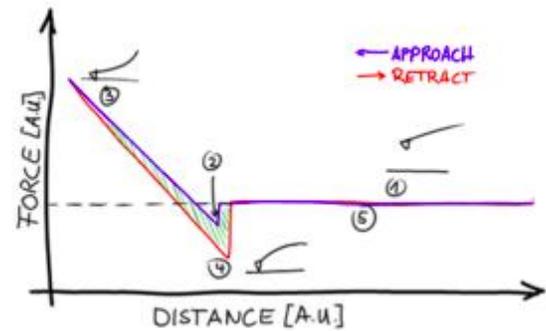

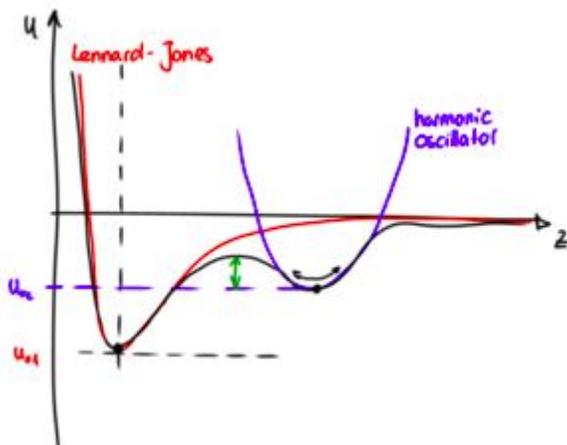Figure A2: Scheme of a force distance curve with plastic deformation

Figure A?: Lennard Jones potential combined with harmonic oscillator potential
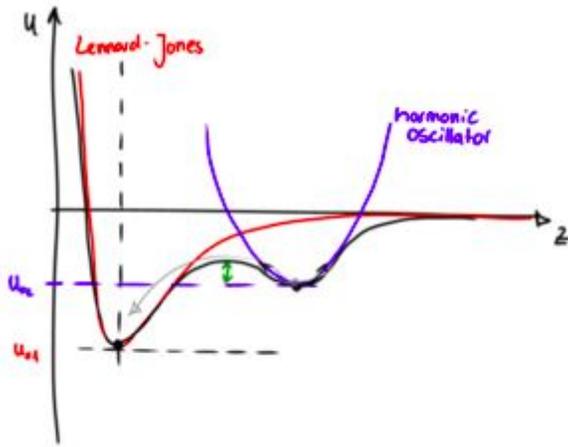
Figure A?: Lennard Jones potential combined
with harmonic oscillator potential when tip is
closer to the surface

Dynamic Atomic Force Microscopy

The cantilever motion can be described by the equation of motion:

$\frac{1}{\omega_0^2}\ddot{d}(t) + \frac{1}{Q\omega_0}\dot{d}(t) + d(t) = F_{Drive}(t) + F_{ts}(t)$, where d is the deflection, ω_0 is the resonance frequency

and Q expresses the quality factor (Gütefaktor). $F_{Drive}(t)$ is the excitation applied to the cantilever, and $F_{ts}(t)$ the force acting between the tip and the sample. $F_{ts}(t)$ includes both, conservative (electrostatic forces e.g.) and dissipative (damping e.g.) contributions. Conservative forces depend on the gap between the tip and the sample, while dissipation forces change depending on the direction of motion.

Far away from the surface, $F_{ts}(t)$ is equal to zero and the equation of motion becomes the linear equation of motion of the classical driven harmonic oscillator:

$$\frac{1}{\omega_0^2}\ddot{d}_{free}(t) + \frac{1}{Q\omega_0}\dot{d}_{free}(t) + d_{free}(t) = F_{drive}(t).$$

If a linear system is driven with a sinusoidal signal $F(t) = cos(\omega t)$, it will respond at the same frequency $d(t) = Acos(\omega t + \phi) = Re\left[\hat{d}(\omega)e^{i\omega t}\right]$, with a scaling factor of the amplitude $A(\omega)$ and a phase shift $\phi(\omega)$.
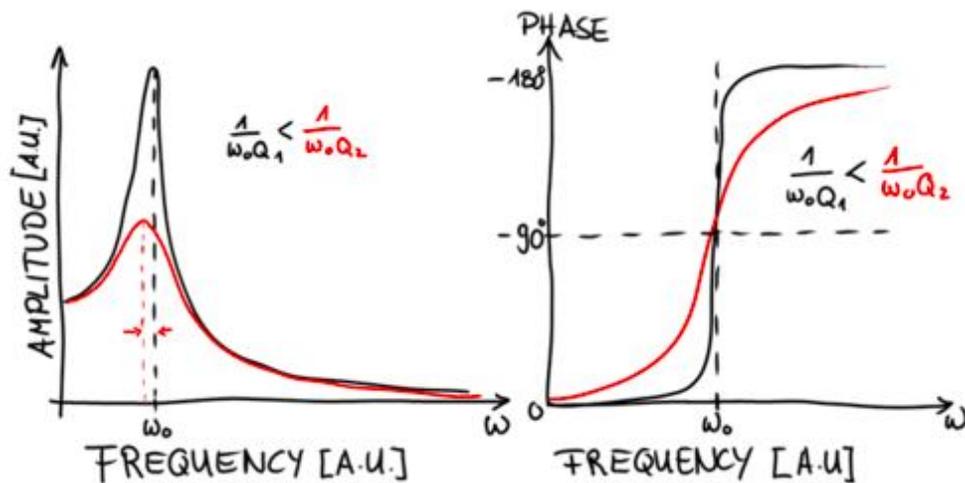
Figure A?: Scheme of amplitude and phase of the harmonic oscillator

Amplitude Modulation

Amplitude modulation atomic force microscopy (AM-AFM) was already introduced by Binnig et. al. [1] when they first presented the atomic force microscope. In AM-AFM the cantilever is excited with a fixed amplitude and frequency close to or at its free resonance frequency ω_0. The feedback is performed on the amplitude A to track the position of equal interaction in imaging mode. The driving signal will not be influenced, even if the tip motion is heavily distorted. Therefore the normal motion of the cantilever can be restored properly after a distortion, making this mode of measurement very stable. When performing dynamic spectroscopy measurements with AM-AFM, the amplitude is monitored as well as the shift in phase between the excitation signal and the cantilever response as a function of the cantilever base position.

The shift in phase as well as the reduction in amplitude originate from a combination of dissipative and conservative interaction. Hence both interactions can not be separated clearly.

Feedback

In most scanning probe microscopes a feedback loop is used to control the separation between the tip and the surface by keeping the for example deflection, amplitude, tunneling current or phase constant. A feedback loop enables the possibility to monitor the tip sample distance at which a certain variable is constant instead of interpreting different values of observed quantities. The topographic images obtained with a feedback loop is often referred as a the physical topography of the surface.

In the case of an AFM usually a PI-Controller (Proportional-Integral-Controller)is used. The controller is used to keep one observable constant during a measurement, even if perturbations occur. In our case the feedback uses the P- and the I-gain to regulate the height. Please check the basic theory of a PID controller (**only section about the theory**) to get a feeling about the working principle of the feedback loop: Section PID controller theory (http://en.wikipedia.org/wiki/PID_controller) (P- and I- gain are essential to perform the experiment!) A PI-controller calculates an error value $e(t)$ as the difference between the desired setpoint and the measured signal. Based on the error signal it then applies a correction based on the proportional and integral terms. To obtain the output signal of the p-controller the error signal is multiplied with the proportional gain factor K_p. The proportional controller accounts for present values and responds fast. In contrast the integral controller summs up the occurring error for a given period of time.

In the AFM the two controllers are combined and the overall correction signal is obtained by adding the two correction terms: $u(t) = K_p \cdot e(t) + K_i \int_0^t e(\tau)d\tau.$

Calibration

In general one is interested in a conversion of the deflection voltage $[V]$ into an interaction force $[N]$. Knowing the spring constant of the cantilever as well as the inverse optical lever sensitivity is crucial for force conversion algorithms. In literature several methods for the determination of the spring constant have been described. In the practical course the cantilever which is used in the measurement is pressed against a hard surface. The repulsive part of the curve can than be used to derive the sensitivity of the setup. Afterwards the thermal noise of the probe is recorded. The thermal noise is the Fourier spectrum of the thermal fluctuations of the cantilever \comment{citep{Sader1999} citep{Higgins2006}}. Thermal noise force is frequency independent and can be calculated from $\sqrt{k_B T/k}$ \comment{citep{Butt1995}}. From the Fourier spectrum the quality factor of the cantilever can be obtained as well as the resonance frequency, to then determine the spring constant. Additional parameters which are necessary to calculate the cantilever spring constant with this method are the length and the width of the cantilever and the Reynoldsnumber of the surrounding medium. This method can only be applied for rectangular cantilevers but does not require knowledge about the thickness of the cantilever which is inconvenient to obtain. The accuracy of the thermal calibration is within 5 to 20%.

Lennard Jones Potential

In order to relate the measured forces acting on the tip with physical properties we need models that relate the quantities with each other. One such model is the Lennard-Jones model. It is an empirical description of the atom-atom interaction and is given as: $U(z) = 4U_0 \left[\left(\dfrac{\sigma}{z} \right)^{12} - \left(\dfrac{\sigma}{z} \right)^6 \right].$

U_0 describes the depth of the potential well, σ is the finite distance at which the inter-atomic potential is zero. σ can be predicted by the lattice constant a_0, when no net stress is acting onto the material.
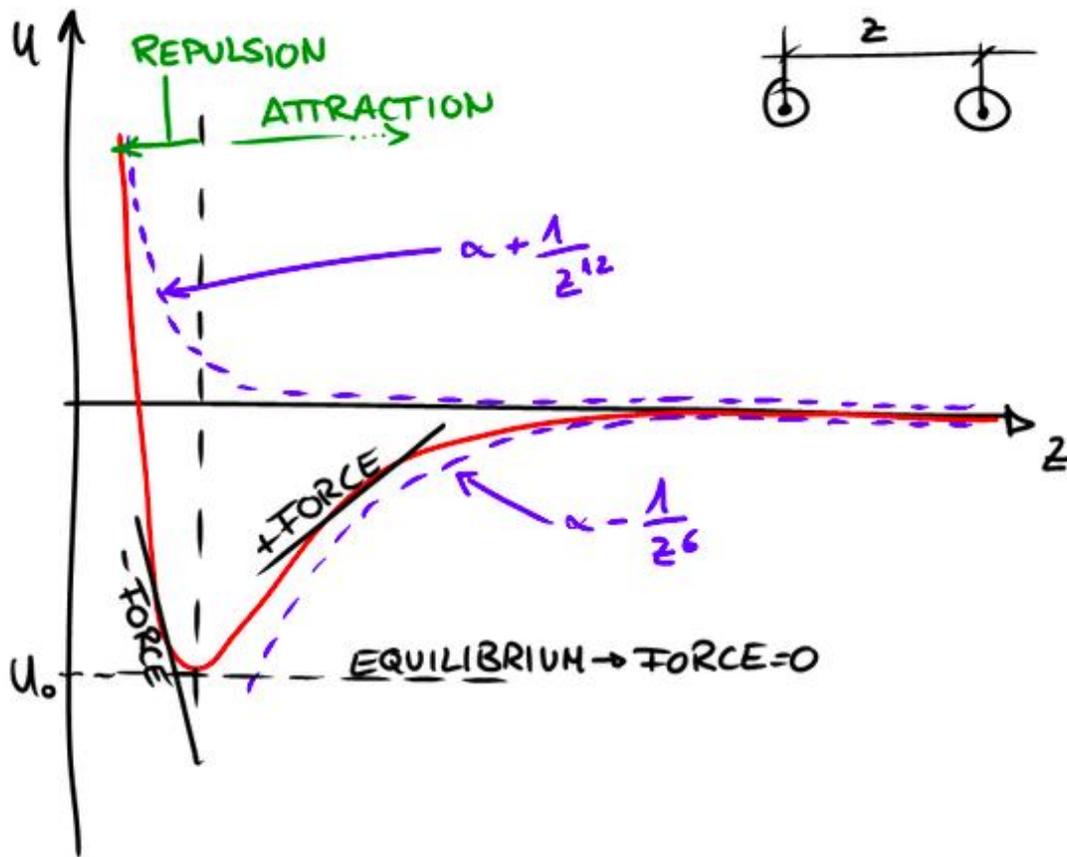
Figure A4: Scheme of the Lennard Jones potential

Elastic modulus

In order to describe the elasticity of a material, the elastic modulus or the youngs modulus is used[2]. The definition is a more general description of Hooks law. The elastic modulus for homogeneous isotropic materials can be calculated by deviding the tensile stress $\sigma(\epsilon)$ by the extensional strain ϵ:

$$E = \frac{\sigma(\epsilon)}{\epsilon} = \frac{\frac{F}{A}}{\frac{\Delta l}{l}}.$$

F represents the normal foce, A is the surface area of one side of the cube with the length of the edge l that is elongated by dl. This leads to the units $\left[\frac{N}{m^3}\right]$

The softer a material the smaller the elastic modulus.

Another quantity that is important in the discussion of elasticity is the Poisson ratio. Whenever stress is applied to a solid this causes deformations. In a cube with the dimensions $w * d * l$ the Poisson ratio is given as: $\nu = \frac{-\frac{\Delta d}{d}}{\frac{\Delta l}{l}} = \frac{-\frac{\Delta w}{w}}{\frac{\Delta l}{l}}$ Whenever the cube is elongated by Δl in the direction of the force, the Poisson ratio indicates how much the cube changes in the directions of w and d longitudinal to the force.

Hertz Model

In order to describe the tip-sample interaction when the tip is in contact with the sample we use the Hertz model. The Hertz model assumes the tip is not deformable, no additional forces act between the tip and the sample and the sample is an isotropic, linear solid of infinite dimensions in a half space. With these assumptions in the elastic modulus can be fitted from the data using the Hertz model. First of all we have to specify our indentation tip.

When intending an elastic half space with a solid cone shaped tip, the indentation depth and the contact radius can be described by

$$d = \frac{\pi}{2} a \tan \theta$$

θ describes the angle between the surface and the sides of the cone.

The overall acting force can be described by:

$$F = \frac{2}{\tan \theta \cdot \pi} E d^2 .$$

For parabolic shapes

$$F = \frac{4 \sqrt{R_c}}{3} \frac{E}{1-\nu^2} \delta^{3/2} .$$

$$a = \sqrt{R_c \delta} .$$

Pyramid (four sides):

$$F = \frac{E}{1-\nu^2} \frac{\tan \alpha}{\sqrt{2}} \delta^2 .$$

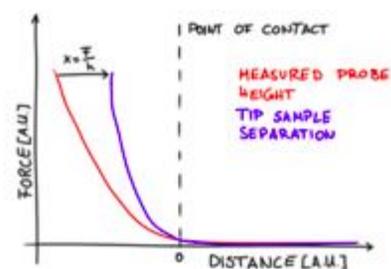$$a = \frac{\tan \alpha}{\sqrt{2}} \delta .$$

Figure A5: Sketch on tip sample separation
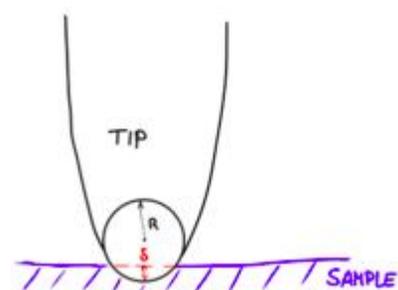
Figure A6: Sketch on the tip radius and indentation

Data Evaluation

To evaluate AFM images we usually use the free software gwyddion which can be installed via opsi. This only works for basic images. If you have data recorded in advanced modes it can be beneficial to use the software provided by the respective AFM manufacturer. There are a few things we like to keep in mind when evaluating AFM images in general. Some of the descriptions mentioned below are based on gwyddion. For a more general, detailed but short description I recommend the book by Peter Eaton and Paul West [4].

- Usually samples are slightly tilted during an AFM measurement. You can correct that by subtracting a mean plane. A common image artifact is a surface that appears bend. This can be fixed by removing a polynomial background.
- When recording an image the height information usually is represented by relative values. Images are easier to read when you shift the minimum data value to zero. In case you have some outliers or want to shift the zero value you can go to : Data Process-->Multidata-->Arithmetic and subtract a constant value there.
- When exporting your file, please make sure that in the final picture one can read the labels and you include a scale bar or ticks.

A procedure to edit most AFM pictures in gwyddion (2.54):

- Open gwyddion. Buttons will be indicated with "xx" in this tutorial, following a drop down menue with -->.
- Open the file via drag and drop or File--> open.
- Via Info--> show data browser you can see and select channels of your image.
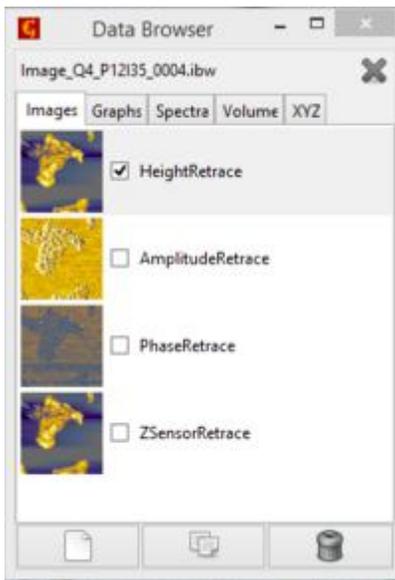
- You can alter the color scheme by clicking on the color bar.
- Use a linear colormap (like Shame or Sky). In addition do not use a nonlinear mapping with this linear colormap (spacing of values should be even and not adapted).
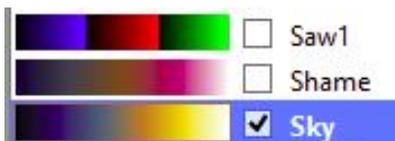
- Select the channel that you want to look at. In this case we select the HeightRetrace channel. As you can see there are some streaks in the image.
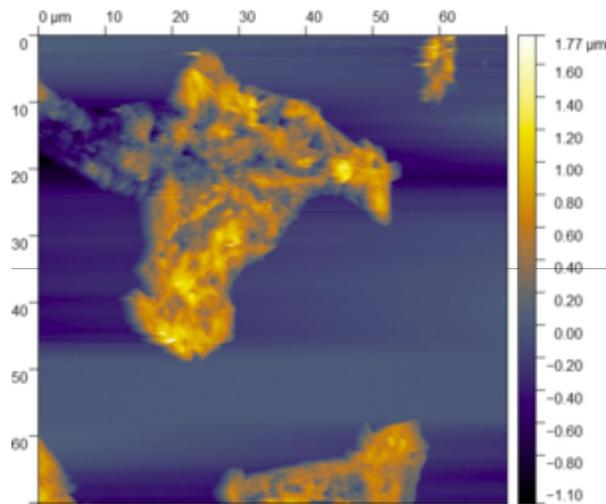
We assume that our substrate is flat in this image. We do not observe an overall tilt in this image. If you can observe such a tilt you can get rid of it by: Data Process--> Level --> Plane Level or find the respective button in the "Data Process" area ("Level Data by mean plane subtraction").

- To get rid of the streaks, mask any features that are not your flat background substrate.

To mask you can try out different procedures. The most effective way may vary from image to image. 1) Mask by hand: Go to tools--> Edit mask. With the drawing and filling tools you can select the area that is not background. 2)Data Process--> Mask --> Mark with you can mark certain areas using masks that you exported earlier or other channels to

mark certain areas in your height image.
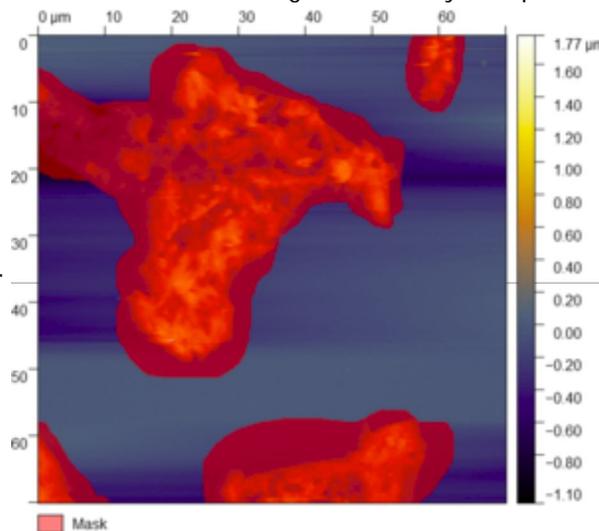
- Now align the rows using: Data Process--> Correct Data --> Align Rows or the respective button("Data Process" area " "Alighn rows using various methods"). Exclude the masked region and set the method to polynomial (first degree). To start with. You can also try out different methods here.
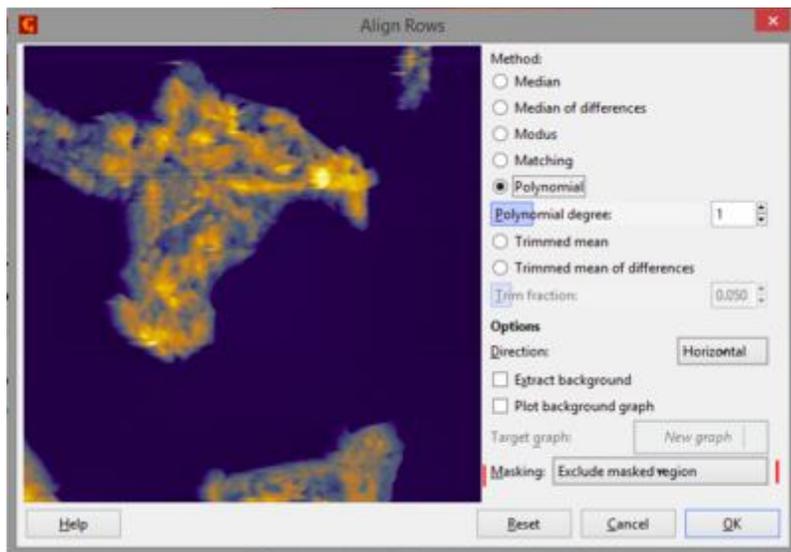
- Before removing the mask you can save the mask via: Data Process--> Mask--> Extract Mask if you want to use it again later. Remove the mask via "Remove" in the mask editor.

- Now we want to alter the range displayed in the image to provide a better contrast and make the features that we want to highlight appear more prominent. Also we want to make the image as easy to read as possible. Shift the minimum value of your image to zero by using "Shift minimum value to zero" in dhe Data Process area. Open the color range via the " Stretch color range to part of the data". You can now play around with the color range.
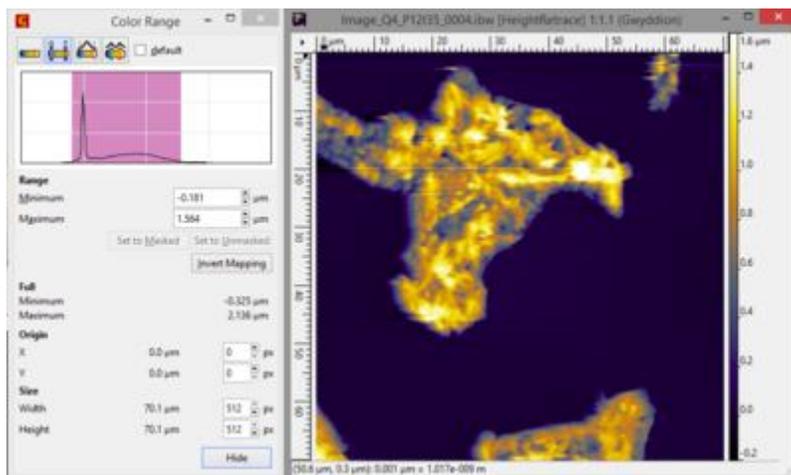
- Sometimes there are outliers in the image or you want to display a different color range. You can manually select a "base" value for your image. Go to Data Process--> Multidata--> Arithmetic and Write d1 - "x" as an expression with x being the value you want to subtract from your image in meters. Adapt the color range in your new image.

- To save all the alterations to the image go to File-->Save as and save the image as a *.gwy file. To export the image also go to File--> save as and export it as a *.png for example. Please make sure that when exporting you always add a scale bar or ruler around the image as well as the color bar.

Additional steps:

If you want to plot a profile line select " Extract profile along arbitrary lines" in the area "Tools". Draw a line in the image. You can alter the thickness of that line, including more data. Click on Apply in the window that showed up. In this new window you can now measure via "Measure distances in the graph". To export the data do a right click in the plot. You can export the image or the data (to plot it yourself) as a text file.

Links

1. G. Binnig and C. F. Quate. Atomic force microscope. Physical Review Letters, 56(9):930–933, 1986
2. Arturo M. Baró and Ronald G. Reifenberger. Atomic force microscopy in liquid : biological applications, Wiley-VCH, Weinheim, 2012
3. Hans-Jürgen Butt, Karlheinz Graf, and Michael Kappl. Physics and chemistry of interfaces, Wiley-VCH, Weinheim, 2., rev. and enl. ed. edition, 2006.
4. Eaton, Peter and West, Paul. Atomic force Microscopy;New York, Oxford University Press Inc., (2010)

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