

Experiment: Atomic Force Microscope (AFM)

1. Overview

The development of the atomic force microscope in 1986 was based on the scanning tunnelling microscope already presented in 1981. While the latter is dependent on conductive samples due to the quantum mechanical tunneling of electrons, the atomic force microscope permits also the use of non conductive samples. In this experiment you will learn the operation of an atomic force microscope and measure the surface topography of two samples as well as examine the effect of electrostatic forces.

2. Introduction

In the scanning tunneling microscope (STM) a fine metal tip is brought so close to the surface of a conductive sample that a quantum mechanical tunneling current through the vacuum barrier between tip and sample occurs. With this tunneling current the distance between tip and sample can be controlled very accurately (see Appendix 1, page 2-5). Since this principle is limited to conductive tips and samples, the atomic force microscope was developed a few years later. Here a fine tip is attached to the end of a cantilever, which controls the force between sample and tip in the so-called "contact mode" (see Annex 1, page 7-10). A further development of this contact mode is the non-contact mode with the so-called NC-AFM (see Annex 1, page 10-11). Here the cantilever oscillates near its resonance frequency. This resonance frequency depends sensitively on the force acting between tip and sample. If now a specific shift with respect to this resonance frequency is held constant, the surface topography can be measured by scanning the tip over the sample.

It is possible to map the atomic structure of surfaces with both STM and AFM. It must be noted, however, that the STM images are a convolution of the electronic structure of the tip and the surface. In the NC-AFM the force gradient between tip and sample controls the distance, therefore a surface of constant force gradient is being mapped. Based on the AFM a number of methods have been developed in recent years that combine different methods of measurement with the AFM and thus allow the measurement of sample properties with high lateral accuracy. For example there is the "scanning capacitance microscope" for the determination of capacity and the Kelvin "probe force microscope" for the determination of work functions.

3. Fundamentals of Atomic Force Microscopy

A typical AFM consists of a cantilever with a small tip at the free end, a laser, a 4-quadrant photodiode and a scanner (see Annex 1, page 9, figure 1-5). The laser beam is focused onto the back of the free end of the cantilever and from there reflected to a 4-quadrant photodiode. Using this position sensitive photodiode the bending of the cantilever can be measured very precisely.

In the NC-AFM the cantilever is now excited to oscillate near its resonance frequency. This is done via a piezo element by applying a small ac-voltage with the corresponding frequency. When the tip is now brought so close to the sample that an interaction between tip and sample occurs, the forces cause a shift of the resonance curve of the cantilever. If the cantilever is excited at a fixed frequency, the amplitude of the oscillation changes. This change is detected by the photodiode and a controller changes the distance between tip and sample so that the preset amplitude remains constant.

The force between tip and sample consists of several contributions:

$$F_{total} = F_{chem} + F_{vdW} + F_{electr.} + F_{magn.} , \quad (1)$$

where $F_{chem.}$ is the short-range chemical force, F_{vdW} the van der Waals interaction, $F_{electr.}$ the electrostatic force and $F_{magn.}$ the magnetic force. The typical characteristics of the forces as a function of the tip-sample distance are shown in Annex 1, page 6, figure 1-4. The repulsive part at small distances is due to the interaction between the positive nuclei and the overlap of electron shells (Pauli principle). The repulsive and attractive forces at small distances can be described by a Lennard-Jones potential:

$$U_{chem.} = 4 \varepsilon \left[\left(\frac{\sigma_0}{r} \right)^{12} - \left(\frac{\sigma_0}{r} \right)^6 \right] , \quad (2)$$

where ε is the strength of the potential at the minimum and σ_0 denotes the distance where the potential is zero. Below this distance the forces are repulsive.

The van der Waals force F_{vdW} is an attractive electrostatic force between permanent and temporary dipoles. Permanent dipoles are molecules which exhibit a permanent polarity due to the electronegativities of the atoms and their spatial structure, whereas temporary dipoles are uncharged molecules with a constantly changing charge distribution. Since the range of the van der Waals forces is limited, the interaction between tip and surface can be well approximated by a sphere in front of an infinitely extended surface:

$$F_{vdW} = \frac{H R}{6 d^2} , \quad (3)$$

here H is the Hamaker constant, R is the radius of the tip and d is the tip-sample distance. The magnitude of the Hamaker constant is about 10^{-19} J. For a tip with radius $R = 30$ nm and a distance of $d = 5$ Å the force in vacuum is $F_{vdW} \approx 2$ nN.

The magnetic forces are only relevant for magnetic tips and samples and can be neglected in the context of this experiment.

The oscillation of the cantilever can be approximated by the equation of motion for a damped harmonic oscillator with forced oscillation:

$$m \ddot{z} + \gamma \dot{z} + k z = F_0 \cos(\omega t) + F_{total} , \quad (4)$$

where z describes the tip-sample distance, γ the damping of the oscillation, k the spring constant of the cantilever and $F_0 \cos(\omega t)$ represents the excitation of the oscillation through the piezoelectric element.

The oscillation frequency therefore is about: $\omega_0 = \sqrt{\frac{k}{m}}$.

The interaction between tip and sample can now be included by a change in the spring constant of the cantilever:

$$k_{eff} = k - \frac{\partial F_{total}}{\partial z} . \quad (6)$$

The new resonance frequency is then shifted with respect to the free resonance. In the case of small oscillation amplitudes (more precisely: the variation of the force gradient should be small over the oscillation amplitude) this shift $\Delta\omega_0$ can be approximately described by:

$$\Delta\omega_0 = - \frac{\omega_0}{2k} \frac{\partial F_{total}}{\partial z} . \quad (7)$$

The resonance curve of the oscillating cantilever thus shifts when additional forces between tip and sample appear. This is reflected by a reduction in the oscillation amplitude when exciting with a fixed excitation frequency.

According to Annex 3 the force gradient can be derived from a change in the amplitude:

$$\frac{\partial F_{total}}{\partial z} = k \left(\frac{1 - 2a^2 + \sqrt{4Q^2(a^2 - 1) + 1}}{2(Q^2 - a^2)} \right), \quad (8)$$

where $a = A_0/A'$ = amplitude of the free resonance / feedback amplitude and Q is the quality factor of the cantilever.

In the AFM the sample is mounted on a scanner that allows to precisely move the sample relative to the tip and thereby allows scanning of the tip across the sample surface. The scanner consists of several piezo elements, which move the sample by applying a controlled voltage (see Appendix 1, page 35-38). It should be noted, however, that a piezoelectric element does not perfectly convert the voltage into an extension in a linear way. Hence the following effects have to be taken into account when analyzing AFM images: non-linearity, hysteresis, creep, aging and cross-coupling. Details of these piezoelectric effects are found on pages 39-48 in Annex 1. Furthermore you can obtain various artifacts in the picture, e.g. by a not very sharp tip. Details can be found on pages 70-75 in Annex 1.

4. Electrostatic forces in AFM

The electrostatic forces in the AFM are a result of the tip and the sample forming a capacitor. Thus the force can be described as:

$$F_{electr.} = \frac{1}{2} \frac{\partial C}{\partial z} V_{eff}^2, \quad (9)$$

where C is the capacitance of the tip-sample capacitor and V_{eff} represents the entire applied voltage. In addition to the voltage V_{bias} which is applied from outside also the contact potential V_{CPD} is taken into account, it is: $V_{eff} = V_{bias} + V_{CPD}$. The contact potential results from the difference of work functions of tip and sample: $V_{CPD} = (\Phi_{Probe} - \Phi_{Spitze})/e$. Therefore the contact potential is dependent on the sample under the AFM tip.

An examination of Eq. (9) shows that the contact potential can be determined experimentally. If we introduce a spectroscopic measurement, i.e. if we measure the tip-sample force as a function of the applied voltage, we obtain a minimum of the force for the voltage that corresponds to the contact potential (see Annex 4). The behavior of the curve should correspond to a parabola. To perform this spectroscopy experiment the controller which controls the tip-sample distance must be turned off.

Another form of voltage spectroscopy is obtained when the distance controller is switched on. Then the electrostatic forces which result from the applied voltages respectively are balanced by a corresponding change in the tip-sample distance. In this case a distance-voltage curve with a constant force gradient is measured instead of the force. This creates the possibility to determine the term $\partial C/\partial z$ from Eq. (9). As described in Annex 5 the shape of the distance-voltage curve depends on the geometry of the tip.

5. Experimental setup

For the AFM experiment a microscope by Nanotec is used. Excerpts from the manual of the microscope are provided in Annex 2 and should be used for preparation of the experiment. A brief overview will be given here.

The AFM consists of a configuration that contains the scanner (see Fig 2-1 and 2-2). The sample is mounted on the piezo which is found in the center. The AFM-head is shown in Fig 2-3 and 2-4 and includes the laser, the photodiode and the holder for the cantilever with tip (see Fig 2-6).

The adjustment of the laser is explained on page 28 – 30 and how to approach the tip to the sample on pages 30 – 31. Previously, however, the AFM head needs to be properly positioned over the sample (see page 32 - 34).

Detailed instructions for setting the laser and to operate the WSxM software are given on pages 46 – 63.

On pages 71 - 74 recording pictures is described and several menu items and controls of the software are given on page 90 – 98.

ATTENTION: For the detection of the cantilever oscillation a laser of protection class 2 is used. Never look directly into the laser beam. When the AFM-head is moved (e.g. from holder to microscope) the laser should always be switched off.

6. Tasks

1. Preparation

- a. Study the basics of atomic force microscopy and the experimental realization of the AFM
- b. Study Annex 1 and 2 as well as papers 3,4 and 5

2. Experimental preparation

- a. After the preparation discussion you will mount the cantilever on the AFM head together with your supervisor.
- b. Adjust the laser on the cantilever (see p. 46-50, Appendix 2)
- c. Adjust the photodiode (see p. 51-52, Appendix 2)
- d. Install the first sample (sample A) by positioning the metal disc with the sample in the center of the scanner
- e. Position the AFM-head over the sample (see p. 52-53, Appendix 2)
- f. Find the resonance of the cantilever (see p. 54-55, Appendix 2). For this, set the excitation voltage to such a value that an oscillation amplitude of approximately 1 V results. The amplitude can be read from the upper graph.
- g. Approach the tip to the sample (see p. 55-63, Appendix 2). Approaching is done by “move”. The parameters for this are: engine speed: 50, motor step: 25, amplitude reduction to range: 8%, amplitude reduction to slow down: 2%. The set point for approaching and measuring should be about $\frac{1}{2}$ - $\frac{3}{4}$ of the adjusted oscillation amplitude.

3. Mapping of the surface topography of sample A (Au on graphite)

- a. Select the feedback parameter P and I so that neither oscillations occur nor the controller reacts too slowly (see p. 58, Annex 2)
- b. Start scanning by setting the scan size to approximately 1000 nm. The scales of the oscilloscope and view images can be automatically adjusted with the “magic stick”. Select the “tilt” so that a scan line is more or less flat. As a typical scan frequency choose 0.5 Hz.

- c. Now select an appropriate size for an overview (e.g. $4 \times 4 \mu\text{m}^2$), restart the scan by “up” or “down” and save the image
 - d. Now zoom into the image (e.g. $1 \times 1 \mu\text{m}^2$) on an appropriate spot with both graphite and gold islands
 - e. Run spectroscopy experiments at various points in this image (see task 4 and 5)
4. Voltage spectroscopy **without** feedback
- a. Place the tip in each case on at least 3 points on the gold islands. For this, press the button “M” in the main-scan window. The scan will stop and a cross appears that can be used to place the tip at any point in the image.
 - b. Open the dialog window for IV spectroscopy and set the desired values (number of views = 1, channel = amplitude, samples/point = 20, number of IVs = 10, 256 points per spectrum).
 - c. Take at least 3 voltage spectra at the 3 points on the gold islands. The voltage should be varied in a range from approximately -2 V to +2 V. The feedback should be turned off, so that the tip-sample distance is not regulated. The time for a spectrum should not exceed 2 seconds. Note which spectrum was taken at which point or select the file names accordingly.
 - d. Repeat the voltage spectroscopy at 3 different locations between the gold islands on the graphite substrate.
 - e. **Evaluation:** Determine the contact potential of gold and graphite with respect to the used AFM tip from the recorded spectra by fitting with equation (9). Compare the values obtained with literature values for the work function.
5. Voltage spectroscopy **with** feedback
- a. Place the tip on at least 3 different points on the gold islands in each case
 - b. At each position take a voltage spectrum in which the voltage should be ramped through a range of about -6 V to +6 V. The feedback should be switched on so that the tip-sample distance is actively regulated while taking the spectrum. Adjust the time for the whole spectrum in a way that the curves for forward and backward spectrum lie on top of each other. For this, the value for "samples per point" should be in the order of 60. **The recording channel in this case is the topography!**
 - c. *Repeat the voltage spectroscopy at 3 different locations between the gold islands on the graphite substrate.*
 - d. **Evaluation:** From the curves determine the tip geometry in accordance with the procedure described in the paper by Olsson et al. in **Annex 4**. Do calculate the according size. Decide in advance which experimental variables you need for the calculation and record the data during the experiments!
6. Mapping of the surface topography of sample B
- a. To change the sample set the scan-size back to 0, withdraw the tip and replace the sample A with sample B. To withdraw press the button “withdraw” in the “approach” window. This button should be pressed for about 30 s. Then turn off the laser (in the window "photodiode") and turn on again only after the sample has been exchanged.

- b. Check the adjustment of the laser beam on the cantilever and the photodiode (see tasks 2 b and c). Approach (2 g).
- c. Select the feedback parameter P and I, so that neither oscillations occur nor the controller reacts too slowly (see p. 58, Annex 2)
- d. Select an appropriate size for an overview (e.g. $2 \times 2 \mu\text{m}^2$)
- e. Zoom into the image (e.g. $500 \times 500 \text{ nm}$)
- f. **Evaluation:** Determine on the basis of the recorded images the size of the used polystyrene spheres of the mask (see sample description below).

For the analysis of the images use the program "WSxM" by Nanotec. This program can be downloaded free of charge for evaluation at home from the website: www.nanotec.es. The spectra are stored as a text file and can be evaluated with appropriate programs.

Notes on the samples and the handling of the samples:

The samples have already been mounted on a sample holder. The holder consists of a magnetic metal plate which is placed on the scanner, where it is magnetically attached. The samples should be touched only with tweezers and never with fingers. Do not touch under any circumstances the surface of the samples (not even with tweezers) as this could lead to destruction of the surface structure.

Sample A consists of gold islands on a graphite substrate. A thin layer of gold (about 5 nm) was evaporated under vacuum on a HOPG sample (Highly Oriented Pyrolytic Graphite).

Sample B consists of small gold islands on a Si (111) substrate. Here the gold (nominal thickness of about 50 nm) was evaporated through a mask of polystyrene spheres and the polystyrene spheres were subsequently removed by adhesive tape, so that the gold that was deposited onto the substrate through the gaps between the spheres remains on the substrate.