Chapter 13
Static Atomic Force Microscopy

In static atomic force microscopy the force between the tip and sample leads to a deflection of the cantilever according to Hooke's law. This cantilever bending is measured, for instance, by the beam deflection method. The name static comes from the fact that the cantilever is not excited to oscillate, as in the dynamic modes of AFM. In the following, we will discuss the static mode, while the dynamic variants are considered in the subsequent chapters. The atomic force microscope (AFM) is alternatively known as the scanning force microscope (SFM). However, here we will use the more common name atomic force microscope. At the end of this chapter, we discuss how force-distance curves can be used to identify the tip-sample interaction regime in which subsequent imaging is performed.

13.1 Principles of Static Atomic Force Microscopy

In static atomic force microscopy, the sample is scanned in the xy-direction while the tip-sample distance is so small that the cantilever sensor can sense the tip-sample force. In the constant force mode of static atomic force microscopy, a certain setpoint value of the tip-sample force is selected via a certain deflection of the cantilever Δz, which is in turn realized by a corresponding sensor signal ΔV_{sensor}. The sensor signal is kept close to the setpoint value via a feedback loop as shown already in Fig. 1.7. When scanning, for example, over a step edge, the tip-sample force changes and thus the corresponding deflection Δz deviates from its setpoint value. The feedback electronics adjusts the z-signal controlling the tip-sample z-distance in order to restore the setpoint value of the cantilever deflection Δz. For ideal feedback, the deflection of the cantilever should always stay very close to its setpoint value. Topographic images are recorded by scanning the tip over the sample surface, while the feedback maintains constant cantilever deflection. The z-height contour corresponds to a contour of constant tip-sample force. For the setpoint value of the force, either a repulsive force or an attractive force can be selected.

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Static atomic force microscopy often operates in the repulsive regime of the force-distance curve. In this case, static atomic force microscopy is also known as contact mode atomic force microscopy. The terms static mode and contact mode (repulsive force regime) are often misleadingly used as synonyms. However, it is also possible to operate the static atomic force microscopy in the attractive (non-contact) regime. We will distinguish between the mode of operation: static (non-oscillating cantilever) or dynamic (oscillating cantilever), on the one hand, and the type of interaction probed: repulsive (contact) or attractive (non-contact), on the other hand.

In static atomic force microscopy, the z-position of the tip, i.e. the deflection of the cantilever, is given by a balance of forces. If the tip comes close to the sample, a force F_{ts} acts on the tip. This force leads to a deflection of the cantilever by Δz relative to the equilibrium of the free cantilever and to a corresponding force F_{cant}, as shown in Fig. 13.1. In equilibrium, the total force on the cantilever has to vanish as

$$F_{tot} = 0 = F_{ts} + F_{cant}, \tag{13.1}$$

with $F_{cant} = -k\Delta z$.

If we take a closer look at the force between tip and sample, F_{ts}, this force comprises several forces: the long-range attractive van der Waals force and the short-range repulsive forces. For the force between individual pairs of tip and sample atoms, we consider the Lennard-Jones potential plotted once more in Fig. 13.2b. The direction of the force on individual tip atoms resulting from the interaction with the sample is shown by arrows in Fig. 13.2a. For different atoms of the tip, forces with different strength and direction act depending on the distance to the sample. Tip atoms closer to the sample experience a net repulsive force (red in Fig. 13.2), while the atoms slightly farther from the sample experience only an attractive interaction (blue in Fig. 13.2). The total tip-sample force is obtained by integration.

Considering that the force between the tip and sample arises due to summation (integration) over billions of atoms in the tip (and in the sample) it might be feared that nanometer or even atomic resolution might never be reached. In this regard two things are helpful: (a) the long-range (attractive) interactions are much weaker than the short-range repulsive forces and (b) the distance dependence of the long-range forces is much weaker than that of the short-range forces. Thus the long-range forces result in a background force which is almost independent of the tip-sample distance,

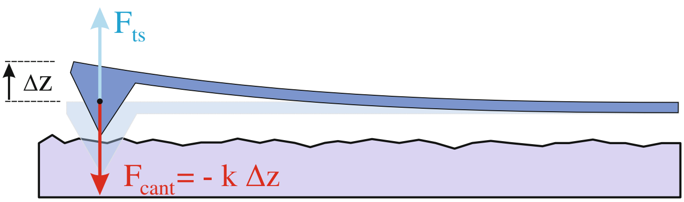

Fig. 13.1 Force equilibrium in static mode. The tip-sample force F_{ts} and the spring force of the cantilever compensate to a net vanishing force

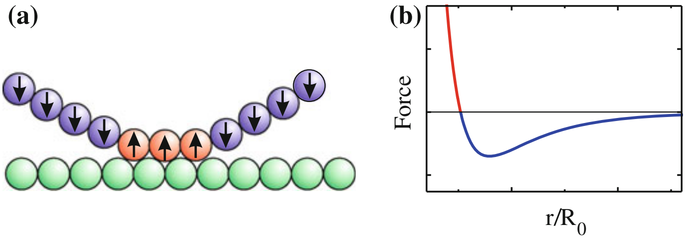

Fig. 13.2 Forces on the tip atoms due to interaction with the sample. For the atoms close to the surface the net interaction with the sample is repulsive (indicated in *red*). For larger distances to the sample, the interaction is attractive (indicated in *blue*)

if, for instance, the tip-sample distance changes by 1 Å. However, 1 Å change in tip-sample distance changes the short-range forces significantly, enabling nanometer or even atomic resolution, as we will see later.

There are cases in which the total interaction between tip and sample can still be attractive due to the long-range attractive forces, while it is already repulsive for the atoms at the tip apex. Since the tip-sample forces also act on the sample, the sample (and tip) can be deformed if these forces become strong. This deformation of tip and sample in the area of the repulsive interaction can establish a contact area consisting of several atoms and therefore inhibit true atomic resolution of single defects in the atomic lattice. This effect in contact atomic force microscopy is called the egg carton effect, since the atomic corrugations of tip and sample slide along each other like two egg cartons. Since the repulsive forces increase very strongly with decreasing tip-sample distance, images of constant repulsive force are often identified with the topography of the surface.

The non-monotonous distance dependence of the tip-sample force leads to the fact that for some forces (negative forces in Fig. 13.2b) two tip-sample distances exist for a certain force. As discussed in Sect. 17.3 in detail, this can lead to instabilities in feedback behavior if the tip unintentionally switches from one branch to the other branch with the opposite slope as a function of distance.

13.2 Properties of Static AFM Imaging

If static atomic force microscopy is operated in the contact mode, the tip is in direct contact with the sample and strong repulsive forces act between tip and sample. To avoid damaging the probed surface, the cantilever should be soft, i.e. the cantilever spring constant should be lower than the effective spring constant (force gradient) of the sample atomic bonds. As discussed in Sect. 11.2, under this condition snap-to-contact occurs, which is actually desired in the contact mode in order to maintain tip-sample contact during scanning.

The standard application of contact AFM is imaging the surface topography with a resolution in the nanometer range. Especially the direct determination of the height of image features is an advantage of AFM measurements. In other microscopy techniques such as optical microscopy or scanning electron microscopy, the lateral feature size is easily measured, but using these techniques does not give easy access to the true height of the imaged features.

Atomically "resolved" images using the contact mode AFM technique were first obtained on layered materials like graphite, boron nitride, mica, molybdenum selenide and others. These materials have the advantage that clean surfaces can be prepared under ambient conditions. While a corrugation with a periodicity of the atomic lattice is observed, defects of atomic size are never observed. This led to the conclusion that small flakes of the layered material are probably attached at the tip apex and that an egg carton effect prevents the detection of atomic size defects.

After the first successful applications of contact AFM to layered materials, it was natural to extend the investigations to non-layered materials. For these cases, the effect of dragging flakes of the layered materials over the surface does not occur. Inorganic crystals like NaCl or LiF were prepared in ultrahigh vacuum and imaged with contact AFM. Typical forces between the sample and the tip during imaging are set to approximately 10^{-8} N. The measured step heights range down to single atomic steps.

The contact zone between tip and sample in contact mode AFM is assumed not to be a single atom but consisting of many atoms. The tip is usually of a different material than the sample surface. Therefore, the tip atoms are not in registry with the sample surface structure and hence a superposition of tip and sample interactions, leading to an atomic resolution, is not expected. The usual understanding is that the atoms of the tip lock into the atomic lattice of the sample, so the atomic lattice of the sample is imaged. However, also on salt crystals like NaCl or LiF no single atomic defects were observed in contact mode AFM. Due to an egg carton effect between the sample and the contact area of the tip, it is possible to observe *atomic corrugation*, while no atomic scale defects are seen and correspondingly no true *atomic resolution* is possible.

Typical problems with contact mode AFM are that contact diameters lie in the range of 1–10 nm, limiting the lateral resolution. Moreover, the relatively high forces can lead to a destruction of soft (organic or biological) samples.

13.3 Constant Height Mode in Static AFM

Up to now we have considered the constant force mode of static AFM, the tip-sample force is controlled to a certain value given by the setpoint for the cantilever deflection. For the constant height mode we assume for the moment that the sample surface is aligned to the scanner, i.e. no scanning slope is present (cf. Chap. 7). In this case an xy-scan can be performed (starting with an initially preset tip-sample distance) and the change of the cantilever deflection is measured. In this case, no feedback is

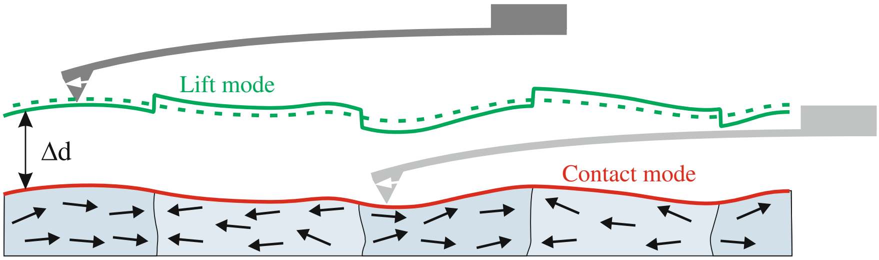

Fig. 13.3 Principle of the lift mode. In a first scan line, the topography is measured (contact mode). In a second scan line, the topography is retraced with an offset Δd (*dashed line*). The deflection due to the long-range magnetic interaction is measured relative to this retraced height (*solid line*)

involved and the scan can be performed fast. The constant height mode is mostly applied for long-range forces, i.e. electrostatic or magnetic forces.

Since it is difficult in practice to get rid of the sample tilt the actual experimental procedure is different from the principle described above. We consider here as an example a magnetic interaction sensed with a ferromagnetic tip, as sketched in Fig. 13.3. In order to be independent of variations in the topography every scan line is scanned twice. First the topography is measured using the contact mode, and in a second scan of the same line the measured topography is followed with an offset Δd relative to the previous scan line as shown in Fig. 13.3 by the dashed line. In this second line, the long-range magnetic interactions are detected by a corresponding deflection of the cantilever shown as a solid line in Fig. 13.3. The difference between the two signals (the dashed and solid line) corresponds to the magnetic signal. This kind of constant height mode is also called the lift mode.

13.4 Friction Force Microscopy (FFM)

When the tip is moved over the surface in contact mode, friction in the tip-sample contact will lead to a lateral force on the tip apex. If the scanning direction is sidewise to the cantilever length, this lateral force causes a torsional bending of the cantilever, which can be recorded in beam deflection microscopes as shown in Fig. 13.4. While a two electrode split photodiode was used in order to detect the vertical bending of the cantilever, quadrant photodiodes are used in order to measure also this torsional bending of the cantilever. In this way the local variation of friction can be studied with high resolution and for various values of external parameters like the load force or the scanning velocity. One great benefit of friction force microscopy (FFM) is that it is possible to measure whether wear has taken place in the course of the experiment by subsequent imaging of the relevant area.

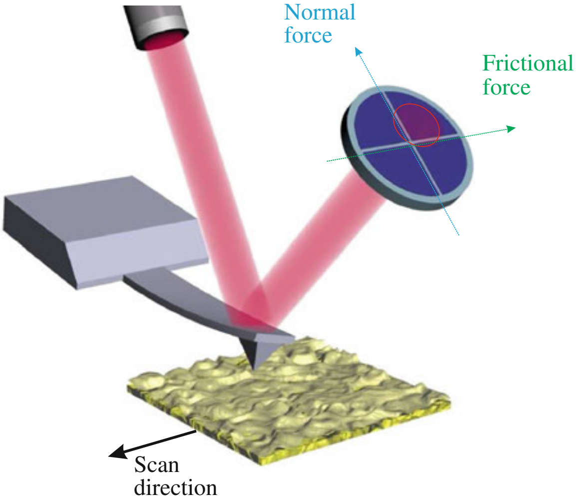

Fig. 13.4 Principle of the detection of frictional forces by the beam deflection method using a quadrant photodiode

13.5 Force-Distance Curves

Force-distance curves are measured by bringing the sample towards the cantilever tip and measuring the cantilever deflection which is proportional to the tip-sample force. These force-distance curves contain the following useful information: (a) The sensitivity of the detection method can be determined as described in Sect. 12.5. (b) Properties like the sample elasticity or the maximum tip-sample adhesion force can be accessed. (c) The working point (setpoint for the cantilever deflection signal) for subsequent AFM imaging can be characterized and chosen properly. For instance, when imaging is performed in the attractive force regime it can be determined how far the working point is located from the point of snap-to-contact. (d) A force-distance curve can be used to determine the tip-sample force-distance dependence, at least partly.

The aim is to obtain the tip-sample force $F_{ts}(d)$ as a function of the tip-sample distance d, as indicated in Fig. 13.5. What is actually measured when acquiring a force-distance curve is the deflection of the cantilever z_{tip} (which is proportional to the tip-sample force) as function of the z-position of the sample z_{sample}. This has the disadvantage that the tip-sample distance d is not only given by the intended z-motion of the sample (induced by a voltage at the z-piezo element) but also by an additional distance change due to the deflection of the cantilever as shown in Fig. 13.5. However, d can always be recovered as $d = z_{tip} - z_{sample}$. With the coordinate system in Fig. 13.5, the action (approach of the sample) and the reaction (deflection of the cantilever) are separated into two coordinates. Also experimentally, these two parameters are measured or set independently: z_{tip} is measured via the cantilever deflection, while z_{sample} is set via the applied z-piezo voltage. As the zero point for

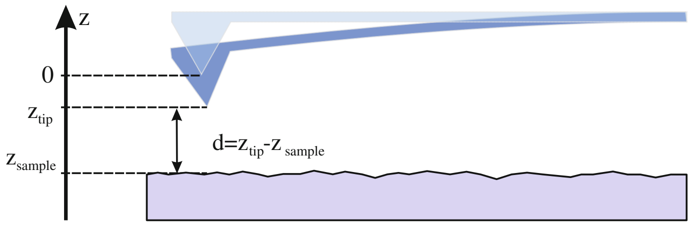

Fig. 13.5 Nomenclature for the coordinates used in force-distance curves

z_{tip} and z_{sample}, we choose the equilibrium position of the cantilever tip with the sample far away.

Figure 13.6a shows a schematic of a $z_{\text{tip}}(z_{\text{sample}})$ plot for the model force-distance curve which is shown in the inset. The blue curve corresponds to the approach of the sample towards the tip, while the red curve corresponds to the retraction of the sample. As the sample approaches the tip (increasing z_{sample} from the right to the left) the cantilever bends slightly towards the sample (negative z_{tip} values) due to the attractive force between tip and sample. At point c, the force gradient exceeds the value of the spring constant k (indicated by a dashed line in the inset). This leads to the previously discussed instability and to snap-to-contact (cf. Sect. 11.2). The cantilever jumps to point d. The maximal cantilever deflection at point c multiplied by the spring constant gives the maximum attractive force before snap-to-contact (usually quite small).

If the sample is moved further towards the tip, the point is reached where attractive and repulsive tip-sample interactions compensate each other and the tip-sample force vanishes. At this position, the cantilever is unbent ($z_{\text{tip}} = 0$). If the sample is pushed further towards the tip, the regime of repulsive tip-sample interaction is entered. In the repulsive regime the sample bends the cantilever upwards. As the repulsive force rises very sharply with decreasing tip-sample distance, both tip and sample move together ($\Delta z_{\text{sample}} \approx \Delta z_{\text{tip}}$ and $\Delta d \approx 0$) Specifically for a stiff sample with a high elastic modulus, the $z_{\text{tip}}(z_{\text{sample}})$ curve is a straight line with a slope of one, as shown in the left part of Fig. 13.6a. If the sample is soft, the slope can be (initially) smaller than one (due to an indentation of the tip into the sample), resulting in information about the elastic/plastic deformation of the sample (cf. Chap. 16).

If the direction of the sample motion is reversed, the tip motion follows the same straight line in the reverse direction (red line) for stiff samples. The repulsive tip-sample force decreases continuously and finally the attractive regime is entered again, where tip and sample adhere to each other as long as the tip-sample force gradient is smaller than the cantilever spring constant. If the force gradient becomes larger than the cantilever spring constant, the cantilever snaps out of contact (point f). The tip snaps back to a position where the deflection of the cantilever is close to zero (point a). Point f corresponds to the position at which the maximum attractive force (adhesion force) between tip and sample acts. Generally, for elastic samples

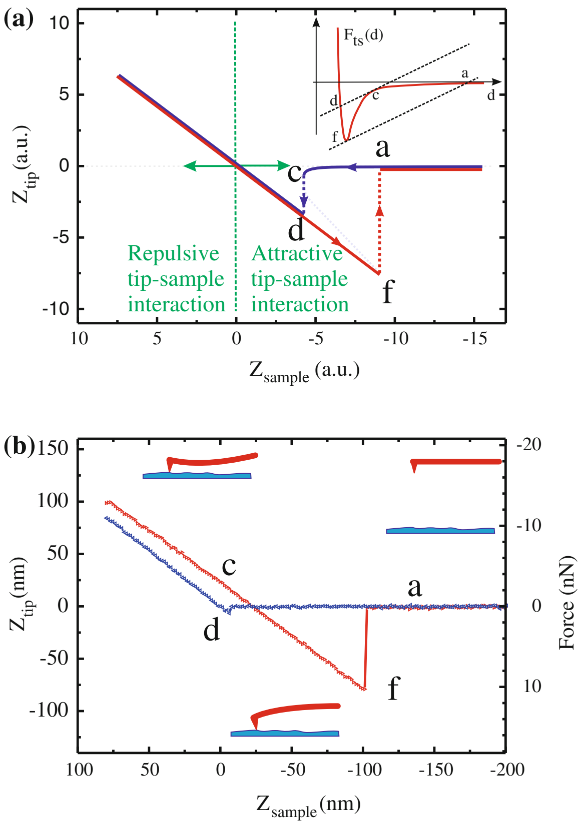

Fig. 13.6 a Schematic of a $z_{tip}(z_{sample})$ plot with the *blue curves* corresponding to an approach of the sample toward the tip, while the *red curves* correspond to a retraction of the sample. The nomenclature for the variables is the same as in Fig. 13.5. At points c and f, the tip-sample force gradient becomes equal to the spring constant of the cantilever and leads to an instability associated with snap-to-contact or snap-out-of-contact, respectively. **b** Experimentally measured force-distance curve obtained on a silicon wafer in a lab course at RWTH Aachen University. The cantilever spring constant was 0.13 N/m (The unusual coordinate system has negative z_{sample} values going to the right. This is, however, the way it is normally plotted)

the retraction curve and the approach curve are the same in the repulsive regime, while the retraction curve lies below the approach curve for a plastic deformation of the sample.

In Fig. 13.6b an experimental force-distance curve is shown which in principle resembles the behavior discussed above. The measured tip deflection is converted (via Hooke's law $F_{ts} = -kz_{tip}$) to a corresponding force, which is shown on the right axis in Fig. 13.6b. In the experimental $z_{tip}(z_{sample})$ plot, the jump to contact (from point c to point d) is small. The corresponding force (the attractive force before snap into contact) is about 1 nN. The maximal attractive force, which is reached at point f just before snap out of contact, can be extracted as 10 nN. Also the width of the attractive force minimum can be read from the difference in z_{tip} between point c and d. This shows that several important parameters of the force-distance curve can be extracted directly from the force-distance curve. In one respect, the measured force-distance curve does not follow the idealized expectation shown in Fig. 13.6a. The approach curve (blue) and the retract curve (red) do not coincide for positive sample distances in Fig. 13.6b. This effect arises due to hysteresis and creep effects of the piezoelectric actuators. For a quantitative analysis of the force-distance curves, those effects have to be carefully excluded.

In principle, the measured $z_{\mathrm{tip}}(z_{\mathrm{sample}})$ curve or the $F_{\mathrm{ts}}(z_{\mathrm{sample}})$ curve (right axis in Fig. 13.6b) can be translated into the more fundamental force-distance curve $F_{\mathrm{ts}}(d) = -kz_{\mathrm{tip}}$, with $d = z_{\mathrm{tip}} - z_{\mathrm{sample}}$. However, as can be seen from the inset in Fig. 13.6a, the force-distance curve between points c and f is inaccessible due to snap in and out of contact. Unfortunately, this is one of the interesting regions. For larger distances down to point c the tip-sample force is almost negligible, while for distances closer than point f, the force rises very steeply. The range in which the force-distance curve can be measured could be extended by using a cantilever with a larger spring constant. However, this has the drawback of reduced force sensitivity.

The importance of the force-distance curves for subsequent imaging lies in the fact that a particular point on the force-distance curve can be identified and that subsequent imaging of the sample can be performed at a defined position on this curve. This is important because the imaging in AFM depends critically on the applied force. For instance in imaging soft (biological) samples it is preferable to avoid strong repulsive forces between tip and sample as this leads to wear on soft sample structures. In order to achieve this the force-distance curve can be measured and the working point for imaging is selected close to point f in Fig. 13.6a, i.e. in the regime of attractive tip-sample interaction, thus avoiding large repulsive forces. However, since this condition is close to snap-out-of contact, there is a danger of leaving the desired imaging conditions by snap-out-of-contact.

The use of force-distance curves in order to determine fundamental force-distance dependences is limited. Several fundamental forces act simultaneously and sum up over the tip and sample volume. The measured forces are integrals of several fundamental forces over large volumes of tip and sample. Additional problems such as capillary forces, an unknown tip shape, and piezo creep complicate a more quantitative interpretation of the tip-sample interaction.[1] Due to these limitations, force-distance curves are not used to measure the fundamental forces.

[1] The influence of capillary forces can in principle be estimated by comparing $z_{\mathrm{tip}}(z_{\mathrm{sample}})$ plots in air and water. If the cantilever is fully immersed in water, capillary forces can be excluded.

13.6 Summary

- In static AFM, the tip-sample force is measured via the deflection of the cantilever Δz.
- In the constant force mode of static AFM, a certain force setpoint is kept constant by feedback during scanning of the surface. The resulting topography corresponds to a contour at constant tip-sample force.
- In the repulsive interaction regime, the tip-sample contact consists of many atoms and thus no atomic *resolution* is expected, but atomic *corrugation* can be observed.
- The constant height mode is mostly used to image corrugation induced by long-range interactions such as magnetic or electrostatic forces.
- Frictional forces can be measured via the torsional bending of the cantilever using a quadrant photodiode.
- Force-distance measurements give access to various parameters of the force-distance curve. The working point for subsequent AFM imaging can be chosen using the information from the force-distance curve.