Atomic Force Microscopy

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Atomic Force Microscopy (AFM) is a member of the family of Scanning Probe Microscopy, together with the Scanning Tunneling Microscopy (STM) and the Scanning Near-field Optical Microscopy (SNOM). Unlike them, however, it is also a descendent of a popular instrument from the pre-superresolution era, the Stylus Profilometer (SP). The probe tip in the AFM is analogous to the stylus in the SP, with a few but significant differences. First, the forces probed by the AFM are in the range where the concept of contact becomes hazy: the contact regime can be defined as the situation where the probe is so close to the sample that is repelled by it. At larger distances, the probe and the sample are attracted to each other, for example, by Van der Waals forces. In this case, the operation is said to be in the non-contact regime. Second, the short distance at which the interaction happens in AFM does not allow for the probe to be driven vertically like in the SP. Instead, it is mounted on a cantilever beam. The forces on the surface of the sample are detected through the deflection of the cantilever beam by a suitable method, the most popular consisting of focusing a laser on the back of the cantilever and detecting variations in the direction of the reflection using a four-quadrant photodetector. These differences make the AFM much more versatile than the SP. For example, in the so-called constant-force mode, the force of repulsion is set to a fixed value and an error signal is generated whenever the force deviates from such value. The error signal that is used to correct the distance between probe and sample is used to form an image of the sample. By scanning fast enough, the AFM can work in the so-called constant-distance mode, in which the signal obtained from the deflection of the cantilever is a map of the distribution of the forces throughout the sample. In the dynamic modes of operation, the cantilever is made to oscillate at or close its frequency of resonance. This can involve operation both on the contact and non-contact regimes, that is, the intermittent contact regime. If the cantilever is driven to the sample, the force of interaction changes the amplitude, frequency or phase of the oscillation and the information contained into one or several of these parameters is used to form the image. The sample does not need to be a passive element, as happens in the Piezoresponse Force Microscope (PFM), in which the cantilever is used to provide electrical excitation to a piezoelectric sample. This variation of AFM can collect information about the ferroelectric domains and the topography of a sample, at the same time. In this work, we intend to give a general description of Atomic Force Microscopy, discussing the singularities of the different regimes and modes of operation, as well as of some features and variations of this versatile technique.

1. Introduction

The way in which a stylus profilometer works is very intuitive as can be seen in Figure 1a. A sharp stylus is driven vertically onto a sample until they make contact. The stylus follows the topography of the sample, which is then recorded on a suitable medium. This is comparable to what humans do when rubbing a finger on a surface to feel its shape or texture. In everyday language, to say that two objects are making contact is the same as saying that they are touching each other or that the distance between them is zero.

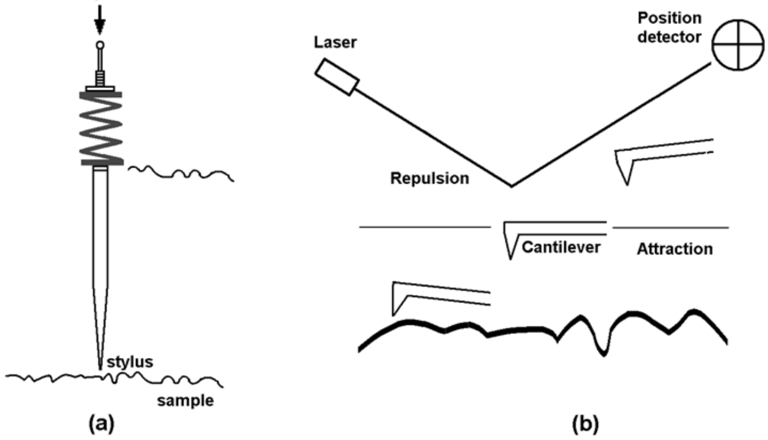

Fig. 1 Imaging by touch (a) Stylus profilometer (b) Atomic Force microscope.

In physical terms, what happens when two bodies approach each other is that, at some point, strong repulsion forces arise that prevent further approach. This phenomenon is the origin of the common sense fact that two objects cannot occupy the same place in space. Figure 2a shows a force-distance curve for an interaction between two carbon atoms. It is remarkable the way in which the repulsion force increases suddenly for distances shorter than 1Å. This repulsion force is associated with Pauli's exclusion principle, which does not allow more than one electron to occupy a quantum state.

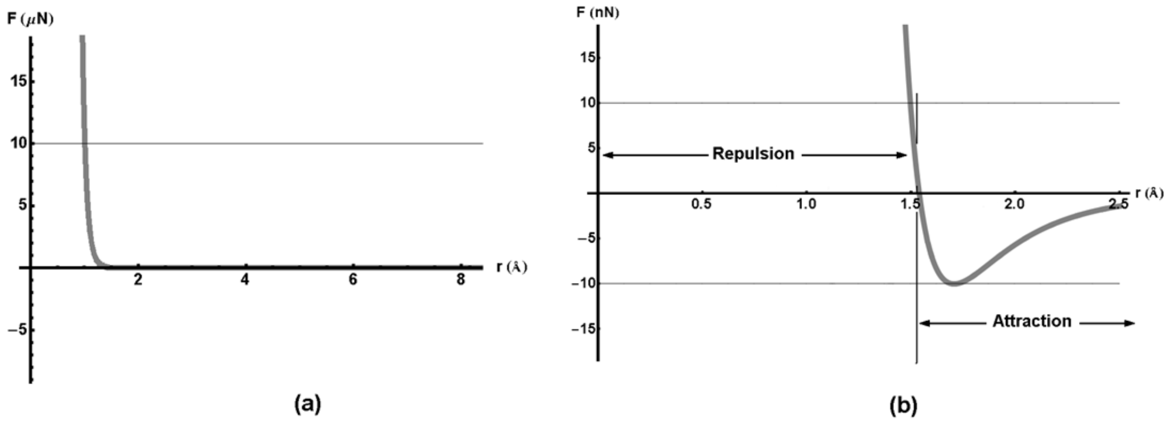

Fig. 2 Repulsion forces in two different ranges (a) Typical of a profilometer (b) Typical of an atomic force microscope.

In a profilometer, the distance at which stylus and sample are held is set by means of the force at which the stylus is driven. Contact is defined as the distance at which the repulsion force is large enough to counteract the driving force. A driving force of a profilometer could be in the order of 10 μN. Too large a force could make the stylus indent the sample whereas too small a force could not be enough for a succesful profiling.

In an atomic force microscope, forces as small as 10 nN can be probed by a tip mounted on a cantilever beam, as can be seen in Figure 1b. On this scale, the force-distance curves look very different. Figure 2b reveals that when two bodies approach each other, the first force they experience is one of attraction. This attraction force increases up to a maximum value and then decreases to be replaced eventually by the repulsion force.

The curves in Figure 2 assumed a Lennard-Jones potential and an attraction force due to the bonding between two carbon atoms. For clean samples, held at low temperatures in a vacuum, this could ideally be the case. In most cases, however, there are several phenomena contributing to the attraction force thus the curves in Figure 2 are far from an exact representation. They have, however, a similar qualitative behavior that is used to exemplify the challenges faced by atomic force microscopy.

In Figure 2b, there is an exact distance at which the force changes from attraction to repulsion, but this is not always used as the definition of contact. In the range of a decreasing attraction force, there is already a repulsive component, even if the net force is not repulsive. If contact is defined as the distance at which a repulsive force enters into play, it can be set as the distance for the maximum attraction force or even as the distance at which the concavity of the force-distance curve is zero [1].

2. The cantilever

The cantilever of an atomic force microscope can be treated as a mass-spring system, as can be seen in Figure 3. The spring obeys Hooke's Law and the undamped resonant frequency is $\omega_0 = \sqrt{k/m}$ where *k* is the elastic constant of the cantilever and *m* is the mass of the tip. The mass of the cantilever beam should be negligible compared to the mass of the tip for this model to be valid.

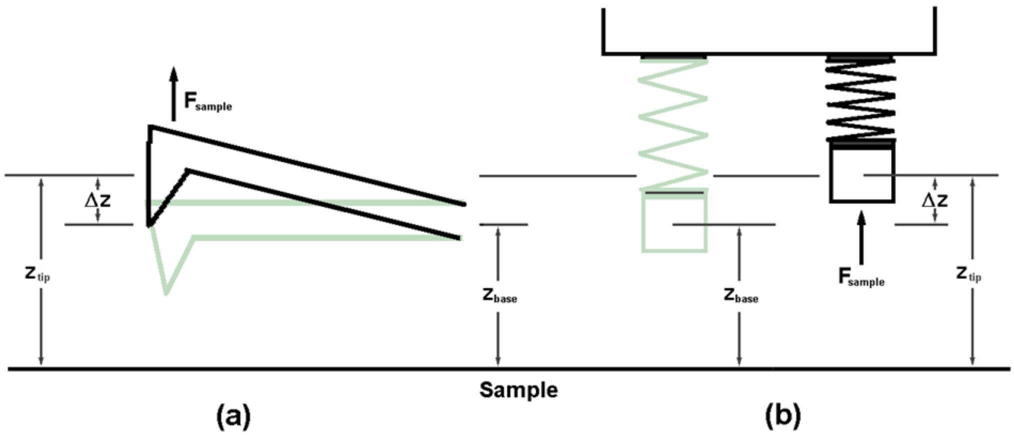

Fig. 3 Modelling the cantilever (a) Cantilever deflection (b) Mass-spring system.

Damping is analyzed through the quality factor $Q = m\omega_0 / \alpha$. In this formula α is the damping coefficient, which when multiplied by the tip speed, gives the friction force in the medium. The resonant frequency for the damped system is shifted to $\omega_0' = \sqrt{1 - 1/2Q^2}$. Upon excitation, the system should reach a steady-state after 2Q cycles [2].

The elastic constant of the cantilever, k, is a very important parameter because it is the way in which force is translated into a measurable deflection. Cantilevers with small k are more flexible and their larger deflections are much easier to measure. There are some cases, however, where stiffer cantilevers have to be used, forcing the use of interferometric techniques for measuring their deflection.

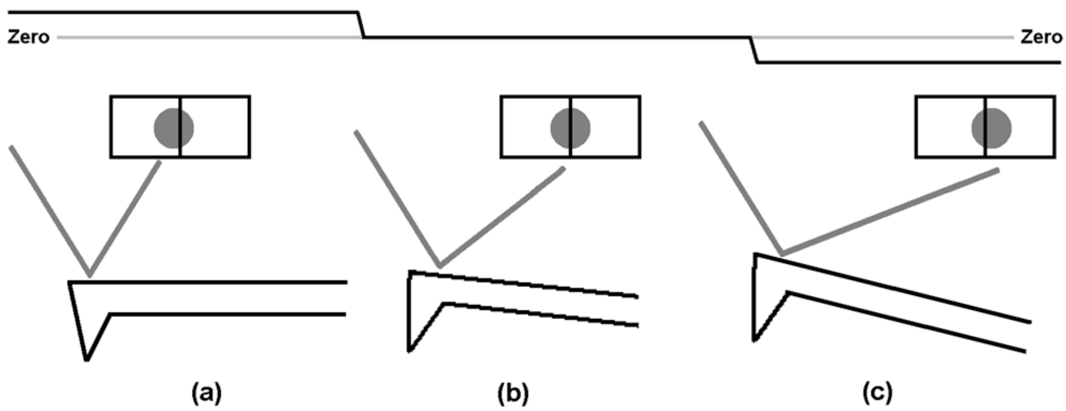

Fig. 4 Measuring deflections through the position of the laser spot (a) Shifted to the left: positive voltage (b) Centered: no voltage (c) Shifted to the right: negative voltage

The most common method for measuring deflections is to focus a laser on the back of the cantilever and detect the small variations in the direction on which it is reflected. This is accomplished by means of a segmented diode detector connected to a differential amplifier, as illustrated in Figure 4. In Figure 4b, the laser spot is split equally on both segments and the output of the differential amplifier is zero. In Figures 4a and 4c, the spot hits more on one segment than on the other, leading to a non-zero signal [3]. It should be noted that a zero voltage in the detector does not imply that the cantilever is not deflected, but that it is deflected by a force at the preset value.

The resonant frequency ω_0 is another important parameter. Since the information is in the deflection of the cantilever, any unintended deflection must be regarded as noise. The cantilever can be excited by low frequency vibrations in the building caused by seismic activity, nearby car traffic or even by human motion inside the laboratory. Because the higher sensitivity is for frequencies close to its resonance, the instrument is protected from these low frequency vibrations by making the resonant frequency of the cantilever as high as possible.

The quality factor Q is proportional to the time that the cantilever will take to reach the equilibrium or a steady-state condition that is needed for a precise measurement. Therefore, a low Q is advantageous in most situations: the operation of an atomic force microscope in air, with Q~1000, is easier than in vacuum conditions with Q~10000. Operation under water, with Q~10, is also possible, opening a wide variety of applications, although a different set of difficulties arises [4].

3. Static operation

In scanning probe microscopy it is very convenient to probe a parameter that varies monotonically with distance. It is obvious from Figures 2a and 2b that the interaction force between probe and sample does not vary monotonically with the distance in the full range, but it does in the range of repulsion. The operation in the range of repulsion is very similar to that of the profilometer and is known as "contact mode". The operation in contact mode is advantageous because it can be static. Static means that there is enough time for the cantilever to bend to an equilibrium state for any scanned spot of the sample.

The force-distance curve of the interaction between probe and sample can be represented by,

$$F_{inter} = f(z) \tag{1}$$

In static equilibrium, this force is counteracted by the restoration force of the cantilever, and can be written as

$$F_{restor} = k\Delta z \tag{2}$$

where Δz is the deflection, given in turn by

$$\Delta z = z_{tip} - z_{base} \qquad 3)$$

where z_{tip} is the distance from the movable end of the cantilever to the sample and z_{base} is the distance from the fixed end of the cantilever to the sample, as can be seen in Figure 3.

If a diamond tip probing the carbon atoms of an organic compound with atomic resolution is assumed, the force-distance curve would be that in Figure 2b. If the elastic constant of the cantilever is set to be k = 10 nN/Å the forces in equations (1) and (2) can be plotted together as in Figure 5. The tip-sample distance z_{tip} in equilibrium lies at the abscissa of the intersection of the line representing the restoration force and the curve representing the interaction force. The distance from the fixed end of the cantilever to the sample, z_{base} is found at the intersection of the line and the x-axis.

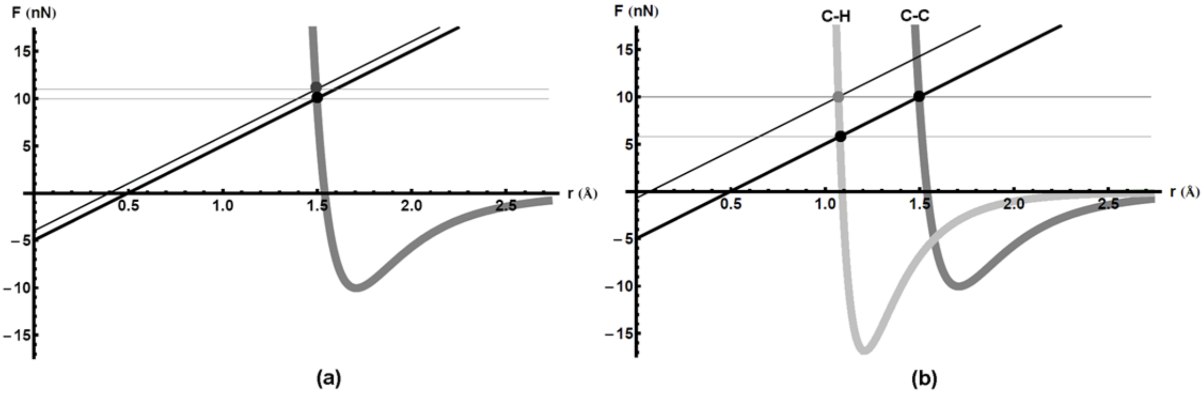

Fig. 5 When scanning the sample, the cantilever can find several sources of variation (a) A height step (b) A change in chemical composition

For an interaction force preset at F_{inter} = 10 nN, the restoration force is represented by the thick line in Figure 5a. The distances at equilibrium can be found to be z_{tip}=1.497 Å and z_{base}=0.497 Å. Thus, the deflection is z = 1 Å, which after being multiplied by the elastic constant gives the required F_{restor} = 10 nN.

Now suppose that while scanning the sample, the probe finds a step of height z_{step}=0.1 Å. The equilibrium is lost when the distances change instantaneously to z_{tip}=1.397 Å and z_{base}=0.397 Å. Although, in the first instant, the restoration force remains at 10 nN, the repulsion force is as large as 70 nN, which starts bending the cantilever upward, reducing the repulsion force and increasing the restoration force at the same time. After the transients are over, a new state of equilibrium is reached, represented by the thin line in Figure 5a. The interaction force is now F_{inter} = 10.97 nN, whereas the distances are z_{tip}=1.494 Å and z_{base}=0.397 Å. The deflection is thus Δz = 1.097 Å, which gives the restoration force of F_{restor} = 10.97 nN required to counteract the interaction force.

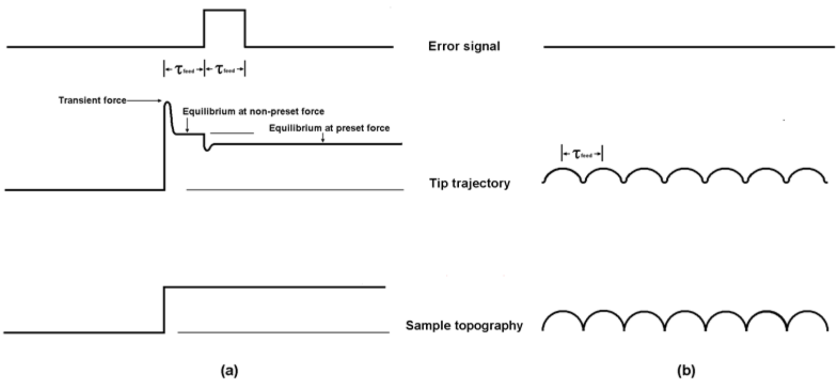

Fig. 6 Constant-force and constant-distance modes of operation (a) The scanning is slow and when a step is found an error signal is generated. (b) The scanning is fast and the cantilever bends in response to variations of the force.

Because this is an equilibrium state, the tip would not have any tendency to bend further. However, as the force is not at the preset value, an error signal will be generated, which will cause the cantilever as a whole to move upward. This will reduce the repulsion and the cantilever will bend downward to reduce the restoration force accordingly, until the preset force is 10 nN and the distances are z_{tip}=1.397 Å and z_{base}=0.397 Å again.

It can be noted that the cantilever bends in response to any imbalance of forces, whereas the position of the cantilever as a whole changes in response to the error signal. It is not convenient for the cantilever as a whole to be moved before a state of equilibrium is reached, because the competing adjustments would cause distortion. The feedback of the error signal is delayed as illustrated in Figure 6a.

4. Constant-force and constant-distance modes of operation.

Summarizing, the step causes a transient imbalance of forces that makes the cantilever bend until reaching equilibrium at a force that is not at the preset level. The error signal is computed from this new state of equilibrium and fed back to make the cantilever as a whole move upward and return the force to the preset level. This is the so-called constant-force mode of operation. In this very common mode of operation, the error signal, or better, a combination of the error signal, its integral and its derivative is used to form the image of the sample.

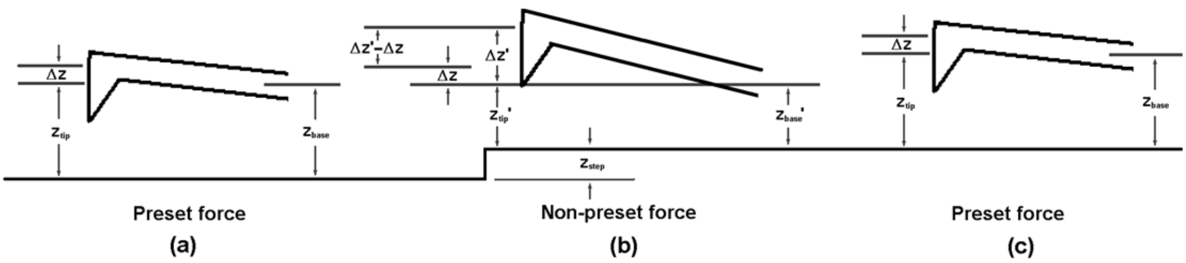

Fig. 7 The cantilever in operation (a) Equilibrium at the preset force (b) New equilibrium at a non-preset force. (c) The equilibrium at the preset force is restored.

For samples of uniform composition the measurement of forces would translate directly into topography. However, this is rarely the case. If the force of F_{inter} = 10 nN is preset while the cantilever is probing carbon atoms, the force of interaction will change suddenly to F_{inter} = 5.80 nN when a hydrogen atom is found, as can be seen in Figure 4b. The restoration force will be transitorily higher, but will be reduced as the cantilever is bent downward until F_{restor} = 5.80 nN

Because in this new equilibrium state the force is not at the preset value, an error signal will be generated, which will cause the cantilever as a whole to move downward until the distances are z_{tip}=1.070 Å and z_{base} =0.07 Å and the force of interaction is 10 nN again. The information left in the error signal cannot be distinguished from that generated when the probe finds a topographic step.

In the constant-force mode of operation, the transients can be minimized by reducing the scanning speed. However, this increases the time required to form an image, which can be impractical for many applications. In any case, a compromise must be made between quality and throughput, depending on the specific imaging needs.

If a mostly flat sample is scanned very fast, the feedback will not be able to adjust for small variations of flatness and the cantilever as a whole will stay at a constant distance from the sample. However, the cantilever will bend in response to these small variations. This is the so-called constant-distance mode of operation, in which the information is taken directly from the deflection of the cantilever, not the error signal. This method is used mostly for imaging individual atoms, although many other conditions as vacuum, cleanliness and vibration isolation must be met for successful imaging.

5. Dynamic operation

Even though the probing of repulsion forces is the easier option for an atomic force microscope, there is also great interest in the study of the attraction forces. However, in this range, force does not vary monotonically with distance and for certain conditions, more than one state of equilibrium can be found. The probe can jump from one state of equilibrium to the other, skipping large portions of the curve that cannot be probed in such circumstances. This phenomenon is known as jump-to-contact [5,6].

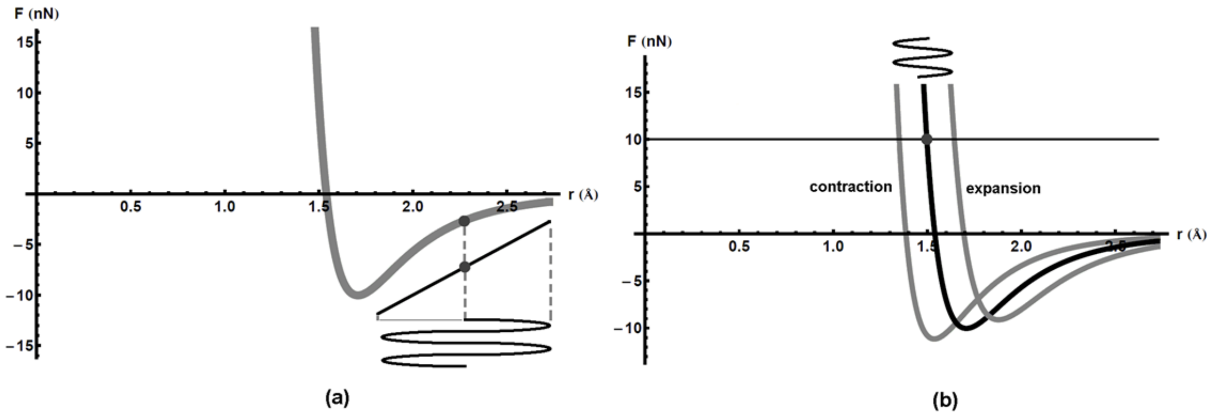

Fig. 8 Oscillations in Atomic Force Microscopy (a) In a dynamical mode of operation, the cantilever is made to vibrate. (b) In Piezoresponse Force Microscopy, a piezoelectric sample is made to vibrate upon a electrical excitation provided through the cantilever.

If the maximum slope of the force-distance curve is smaller than the elastic constant of the cantilever, there will only be one intersection, one state of equilibrium and no jump-to-contact. However, to choose the stiff cantilevers that could comply with this would reduce the sensitivity of the detection system shown in Figure 4, forcing the use of expensive interferometric techniques.

Another option to prevent the jump-to-contact from happening is working out of the static equilibrium condition. The cantilever can be forced to oscillate around an average distance, as seen in Figure 8a. The system will never be in equilibrium since the restoring force will always be larger than the interaction force, although it will reach a steady-state condition after 2Q cycles.

In this approach, known as dynamical operation, three parameters, frequency, amplitude and phase are available to be controlled or monitored. The relationship of these parameters with the surface forces is much more complex than Hooke's law, thus the correct interpretation of data can be an issue. The two most important methods are known as amplitude modulation (AM) and frequency modulation (FM).

In amplitude modulation or tapping mode, the frequency is set close to resonance and the probe is driven to the sample until the amplitude of the oscillation reaches a preset value. An error signal is generated whenever the amplitude changes from this preset value and this error signal together with information about the phase is used to form the image. The information about amplitude and phase is obtained through a lock-in amplifier, using the signal that excites the vibration of the cantilever as a reference.

During one single oscillation of the cantilever, the net force on the tip can vary dramatically, especially if it enters into the contact regime. These variations can be reduced if stiff cantilevers are used in order to keep the amplitude of the vibration small. This approach would make the interpretation of the data easier, but would also reduce the sensitivity of the measurement, as already explained.

Amplitude modulation cannot be used in a vacuum because of the high Q. For frequencies different from resonance, a high Q means that the time taken to reach a steady-state condition is long. Therefore, in a vacuum, the cantilever has to vibrate in resonance, where the steady-state condition is reached instantaneously.

If the cantilever is made to vibrate and the signal coming from the cantilever is fed back to the piezoelectric that drives the vibration, the cantilever will soon be oscillating at its natural frequency of resonance, what is known as self-excitation. Upon approaching the sample, the probe experiences the forces of interaction and the frequency of resonance changes in response. If the amplitude of the vibration is kept constant and the changes on the frequency are used to form the image, this corresponds to the method of frequency modulation [2,7].

6. Piezo response force microscopy

In the dynamical modes of operation described above, the probe is not regarded as a passive element because it is mechanically excited. However, by this same criterion, the sample is still passive, because the forces probed are those that were already there and did not need any excitation to enter into play.

As a matter of fact, the sample can be excited to cause the emergence of additional forces. For example, if an electric field is applied on a sample, this will deform in what is known as the reverse piezoelectric effect. The forces associated with these deformations can then be probed for information.

In a piezoresponce force microscopy (PFM) an alternating voltage is applied on the cantilever, to which a piezoelectric sample will respond by expanding and contracting alternately. These deformations will change the force of interaction between probe and sample as can be seen in Figure 8b. Upon these contractions and expansions of the sample, the free end of the cantilever will bend in order to keep a static equilibrium condition. Thus, the signal in the position detector will have the same frequency as the voltage of excitation.

PFM was developed for the probing of ferroelectric domains in ferroelectric materials. Ferroelectric materials have a strong piezoelectric response, which leads to easily detectable oscillations of the cantilever. The orientation of the ferroelectric domains does not have any effect on the amplitude of the oscillation but has a strong effect on the phase of the oscillation. Thus, the phase is extracted using a lock-in amplifier and used to form a map of the ferroelectric domains [8].

In a PFM, the cantilever is used both as a source of an electrical excitation and as a probing element. It is important to note that no oscillations of the cantilever are excited, but they occurr in response to the forces that the cantilever is probing while scanning the sample. It is not the cantilever that is excited but the sample, and the excitation is electrical not mechanical. Because the force of interaction and the force of restoration are in mechanical equilibrium, PFM must be regarded as performing in a static mode of operation.

7. Conclusion

The AFM is a distinguished member of the scanning probe microscope family. Even though it may be considered as an improved stylus profilometer, a classical instrument, its essential features are based on quantum mechanical concepts. In many ways, the Scanning Tunneling Microscope (STM) is the real precursor of the AFM in the sense that much of the instrumentation and software was borrowed and adapted from it. Due to the limitation of the STM to conductive materials, the AFM ended up being a much more versatile measuring instrument. The possibility of using different kinds of tips (dielectric, conductive, magnetic) made the AFM a very valuable tool in several fields of research and technology.

In this work, the principle of operation of the AFM is explained in a comprehensive but simple way. The most popular modes of operation (constant force, constant distance and dynamic modes) are described exposing the capabilities of the instrument and its ability to adapt to the different measuring situations.

A special reference is made to the Piezoresponse Force Microscope (PFM), where a conductive tip is used to excite a piezoelectric sample. As a result, a map of the polar domains will be produced and valuable information regarding the ferroelectric and piezoelectric properties of the sample will be obtained.

In summary, scanning probe microscopes have become a valuable and indispensable tool in many scientific and technology fields. Their simplicity and relative low cost as compared with other systems with similar capabilities such as scanning and transmission electron microscopes have made them the instruments of choice for micro and nano research and technology. As technology advances, better and more user-friendly microscopes are to be expected.

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