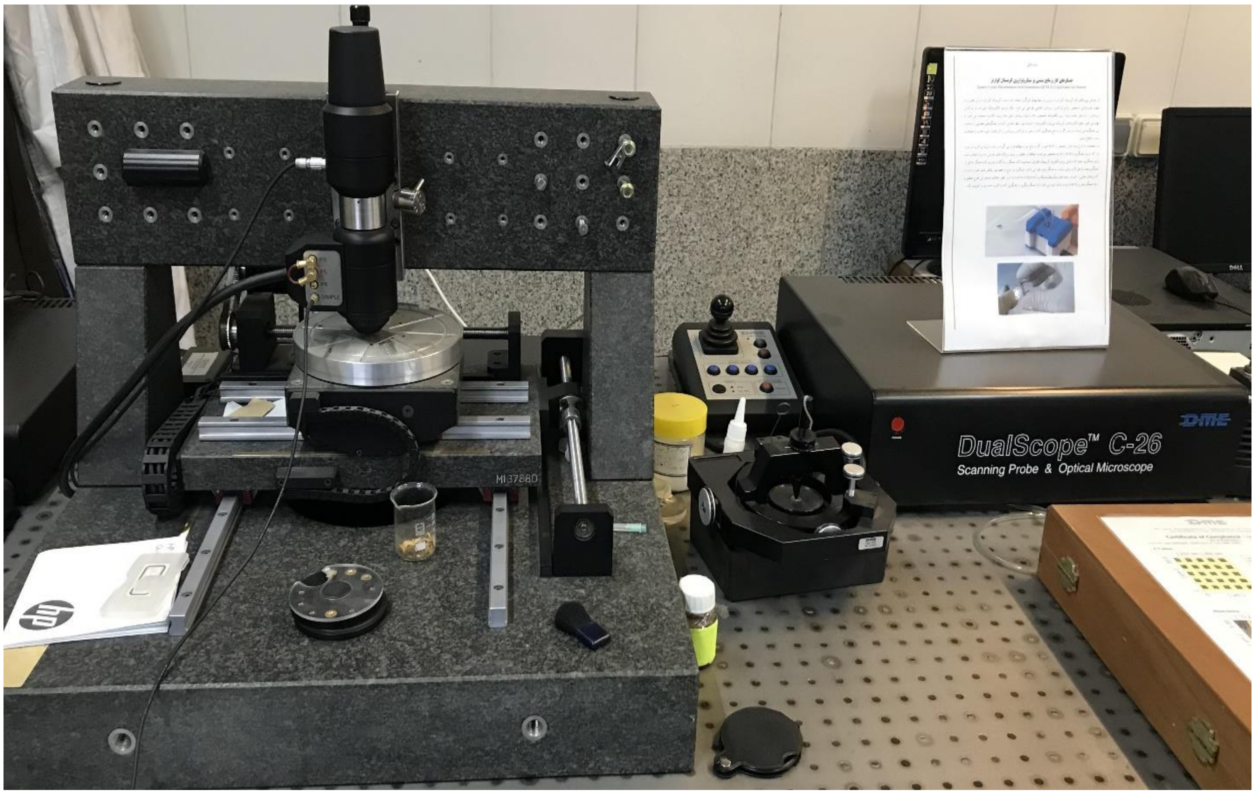
AFM

Atomic Force Microscope



Aim of this document is to give a global overview of the main instrumentation and applications.

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Contents

1. Introduction

Ordinary microscopes cannot be used to observe objects and samples with very small dimensions such as small molecules and atoms; Because these samples have nano dimensions and normal microscopes that work with visible light are not able to show nano dimensions. Therefore, to see nano samples, more accurate and advanced tools should be used to identify and analyze materials.

By using microscopic methods, high-magnification images of the material are obtained so that its details can be studied carefully. The resolution of microscopic images is determined according to the type of beam used. For example, using optical microscopes, the resolution is about 1 micrometer and in the best state 200 nm, while using transmission electron microscopes (TEM), scanning electron microscopes (SEM), Scanning Probe Microscopy (SPM) and Field Ion Microscopy (FIM) with high atomic resolution from about one nanometer to several angstroms are accessible.

We have several types of scanning microscope models, such as scanning probe microscope (which includes AFM, STM scanning tunneling microscope, SNOM/NSOM near-field scanning optical microscope, STED microscope, scanning electron microscope, electrochemical AFM). Although SNOM and STED use visible, infrared, or even terahertz light to illuminate the sample, their resolution is not limited to the refractive limit of light.

In the scanning probe microscope group, the atomic force microscope is one of the most widely used tools for investigating nanomaterials and it is a tool for observing samples with nanometer dimensions and investigating their surface topography [1].

Atomic force microscopy or scanning force microscopy is a model of scanning probe microscopy with very high image resolution, with image resolution shown at fractions of a nanometer, 1000 times better than limiting optical processing. Information is collected by "feeling" or "touching" the surface with a mechanical probe. Piezoelectric elements that facilitate small but accurate and explicit movements with (electronic) command enable accurate scanning.

In atomic force microscopes, weak forces such as van der Waals forces and capillarity between the probe tip and the sample surface are used to form a topographic image of the sample surface. Hence, there is no limit to investigating the sample surface against scanning tunneling microscopes. This microscope is capable of imaging conductive, non-conductive and even biological samples with atomic spatial resolution.

In addition to characterization, AFM microscope can also be used in the field of producing nanostructured materials. Also, this device has the ability to change and move particles and surfaces. This device, with the advantages of high-resolution image preparation, simple operation, not needing for complex sample preparation (such as sample preparation methods in an electron microscope), three-dimensional image preparation, and the possibility of investigating surface topography has entered as an acceptable tool in various fields of science and nanotechnology and has found very wide applications. This device, depending on its model, has the ability to perform tests in ambient and liquid vacuum conditions. AFM has been applied to a wide range of natural sciences, including solid state physics, semiconductor science and technology, molecular engineering, polymer chemistry and physics, molecular biology, cell biology, and medicine [5]. Uses in solid state physics include (a) identification atoms on the surface, (b) evaluating the interactions between a particular atom and its neighboring atoms and (c) studying variations in physical properties resulting from changes in atomic arrangement by atomic manipulation. In molecular biology, AFM can be used to study the structure and properties of mechanism or assembly of proteins to be used. For example, atomic force microscopy is used to image microtubules and measure their stiffness. In cell biology, AFM can be used to distinguish cancerous cells from non-cancerous ones based on cell stiffness and also to evaluate the interactions between a particular cell and neighboring cells in a competitive system or cell membrane shape.

Today, different commercial devices with similar basics and different working modes have been offered, which differ from each other in accuracy and image quality. In this category, while introducing the atomic force microscope and how it works, different working modes and the advantages and disadvantages of each are investigated.

1.1. AFM and SEM difference

The main difference between atomic force microscopy and competing technologies such as light microscopy and electron microscopy is that AFM does not use lenses or beams. As a result, to the distances clarity related to deviation and refraction and providing space for guiding the beam (by correcting the suction) are not limited, and it is not necessary to stain the samples.

As a first point, while the sample preparation and scanning processes in SEM can destroy the natural structure of the sample, atomic force microscope (AFM) does not require any sample reconstruction. While the most important applications of SEM in the investigation of nanofiber structures include the rapid investigation of the shape, orientation, diameter, and uniformity of the fibers, three-dimensional imaging with AFM makes it possible to determine the degree of surface roughness, the degree of roughness along the length of the fiber, and the determination of the thickness of the produced fabric. In addition, by observing some technical considerations AFM can perform as well as SEM in estimating the average diameter.

1.2. Advantages and disadvantages of atomic force microscopy

Advantages

Resolution of images within the range a few nanometer

Making a 3D profile of the surface

Not needing for sample preparation

Ability to work in air and liquid environment

Atomic manipulation

Disadvantage

Down velocity

2. History

Based on the previous designs for the design and construction of scanning tunneling microscope (STM) by Gerd Binnig, which he had done in collaboration with Heinrich Rohrer, in the Zurich IBM research laboratory, he presented AFM in 1986, with the collaboration of Kelvin Quayt and Christoph Gerber from Stanford University. His goal of this work was to measure very small forces (less than 1μN) between the tip of the AFM needle and the surface of the sample [1, 2, 3, 4, 5, 6]. The commercial production of these products was started with the STM microscope in 1987 and the AFM microscope in 1989. Following the invention of STM and then

AFM, many efforts were made to study the morphology and structure of surfaces and their interfaces, and in a short period of time, many other identification tools with applicable similar principles, were made under the general title of scanning probe microscopes and presented to the world of science.

3. The application range of the Atomic Force Microscope

While the scanning tunneling microscope can only be used to study surfaces that have some degree of electrical conductivity, atomic force microscopes can be used to study any type of engineering surface; Therefore, it can be used to study all kinds of conductive, semi-conductive and non-conductive materials. Today, AFM is a favorite surface researcher for topographic measurements and calculation of vertical forces at the micro to nano scale. Also, this characterization device can be used to study scratches and abrasions, as well as measure elastic and plastic mechanical properties (such as the hardness of the object against the sinking object and the modulus of elasticity) [4, 5, 7].

AFM has been used in many studies to write, manipulate and move individual xenon atoms, molecules, silicon and polymer surfaces. In addition, this microscope has been used for various types of nanolithography and the production of nanostructures and nanomachining [6, 7, 8, 9, 10]. Atomic force microscopes designed to measure vertical and lateral forces are called lateral force microscopes, or friction force microscopes [11, 9, 8]. A group of FFMs have the ability to measure lateral forces in two orthogonal directions [12]. Many researchers have modified and improved the designs of AFM and FFM, and now these improved systems are used to measure adhesion, friction and bond forces in solid and liquid surfaces at the nano and micro scale [13, 14].

4. Atomic force scanning microscope device system

The AFM atomic force scanning microscope analyzes the surface of the sample with a sharp needle, 2 microns long and usually less than 10 nm in tip diameter. The needle is located at the free end of a pincer (cantilever) with a length of about 100 to 450 microns [10].

One of the important points in scanning probe microscopes is to scan the sample surface with high resolution. Piezoelectric materials provide this possibility. These materials change length by applying voltage and proportional to its direction and amount. In this way and by applying the cantilever along with the needle to the piezo scanner, it is possible to scan the surface of the sample with a resolution of 0.1 angstrom. In primary SPM microscopes, three rods perpendicular to each other constituted this scanner, and exerting voltage to each of these three rods caused its length variation, resulting in needle deflection in the X, Y, and Z directions. In new microscopes, scanning is accomplished by a hollow cylinder of piezo material. The internal surface of the cylinder is covered with a thin layer of an electrically conductive metal (such as nickel) and four metal strips are placed on the outer surface of this piezo cylinder. Exerting voltage with the same direction and value to all four external electrodes (compared to the internal electrode) causes expansion and as a result lowering of the cylinder and the attached needle to it and or contract and go up. Applying voltage with opposite signs to different electrodes (for example, X and -X) causes the cylinder to flexion, which actually includes movement in the X, Y plane and also movement in the Z direction. With the software control of voltage application, the needle can be move as desired in all three directions. The electronic system of the device provides the possibility of controlling this movement with an accuracy of 0.1 angstrom.

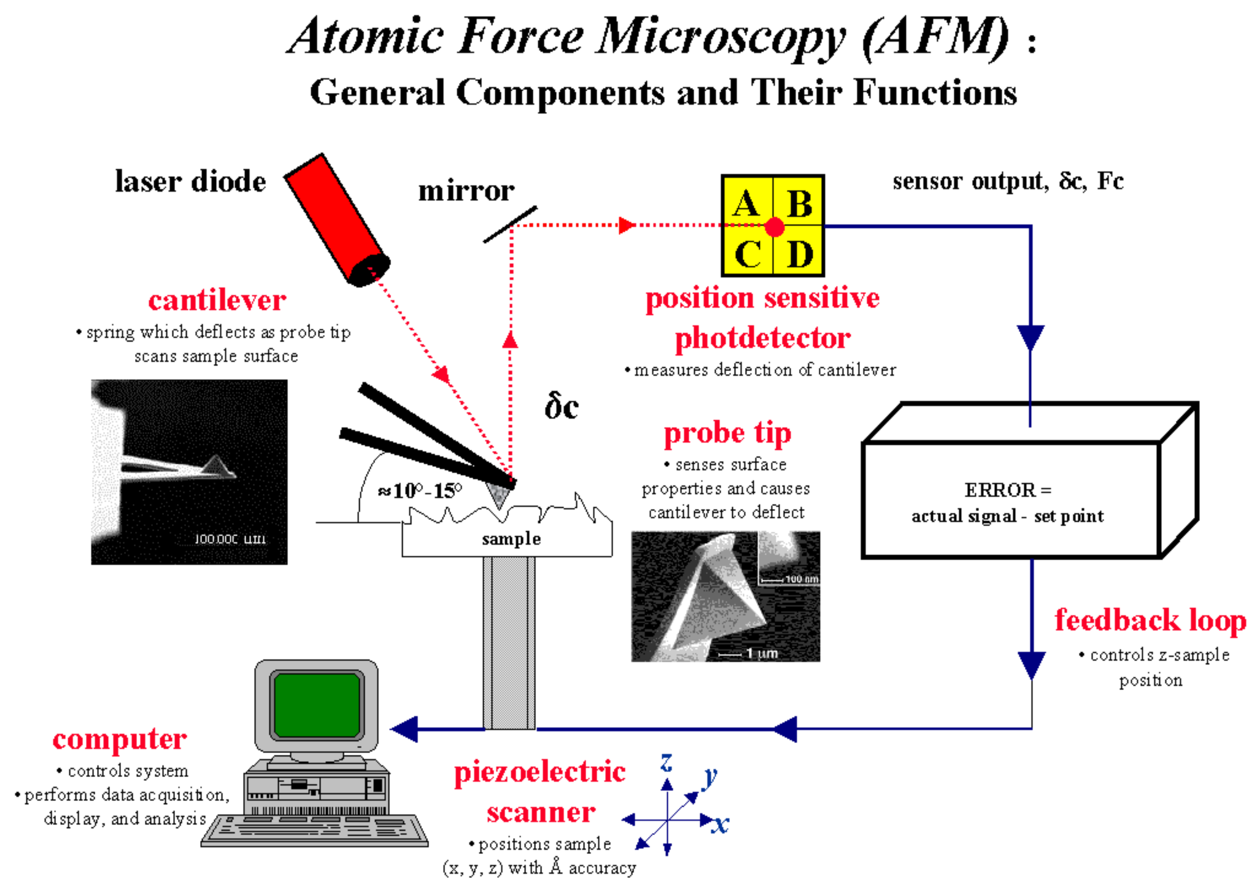

Fig. 4.-1. General components of atomic force microscope and their function

The forces between the needle and the surface of the sample cause bending or deflection of the cantilever, and a detector measures in the systems where the sample performs the scanning movement, the amount of deflection of the cantilever while the needle scans the surface of the sample. The cantilever deflection can be used as the input of a feedback circuit that the piezoelectric probe in confrontation the surface topography of the sample in such a way moves up and down in the z-direction that the cantilever deflection remains constant. Measuring the cantilever deviations allows the computer to produce a topographical image of the surface [11].

4.1. Scanner

The piezo scanner, the cantilever and needle assembly, the laser detection system sensitive to the cantilever position, and in some cases a pre-amplifier, make up the scanner part of the device. The scanner is installed by a special metal alloy tripod or minimum thermal expansion and contraction on a stand or x,y table. The sample is placed on the table and its surface is scanned by a needle. This base is equipped with energy absorbing tires. The base of the scanner is equipped with a CCD camera that can be directly used as a microscope with a magnification of about three hundred times.

4.2. Cantilever

In most AFMs available today, the position of the cantilever is determined using optical methods. The most common ones are shown in Figure 2-4.

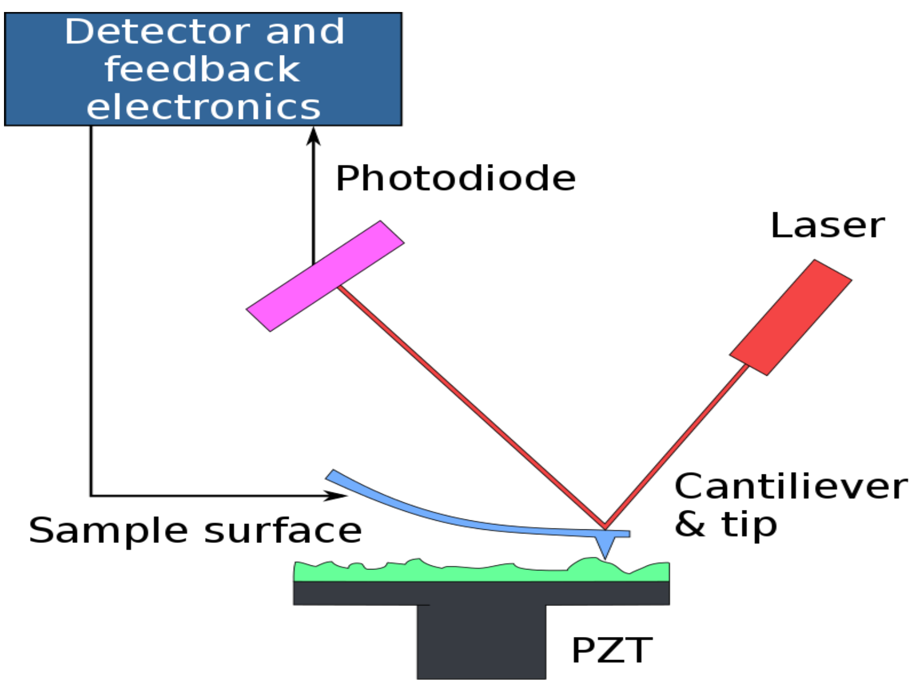

Fig. 4.-2. Components of how-to detection the position of the cantilever with the common method in the atomic force microscope

A laser beam is reflected from the back of the cantilever to a position-sensitive photodetector

The cantilever is usually made of silicon or silicon nitride with a radius of curvature of the tip on the nanometer scale. When the tip is brought near the surface of the test sample, the forces between the tip and the sample lead to its deflection (according to Hooke's law) [6].

By bending the cantilever, the location of the laser beam on the detector changes and the PSPD (Position Sensitive photo Diode Detection) can measure displacement as small as 10 angstroms (1 nm). The ratio of the distance between the cantilever and the detector to the length of the cantilever acts as a mechanical amplifier. As a result, the system can measure the vertical movement of less than angstrom of the cantilever tip. Another method to detect the deviation of the detector is based on optical interference. Diamond, Si_3N_4, Si, W and Ir can be mentioned among the commonly used materials in the construction of cantilevers.

4.3. Control circuit

The signals obtained due to the movement of the needle, with or without initial amplification, are entered into the controller part that contains electronic circuits or down noise, and after amplification and initial processing, they are sent to the computer to form an image. The controller controls the scanner by sending electrical signals. The software installed on the computer processes the output signals from the controller and converts them into images. simultaneously, the software provides the possibility of processing and 3D measurements on the obtained image.

5. Types of forces available in the scan operation

Atomic force microscopes are confronted with forces such as short-range forces, electrostatic forces, capillarity, etc. during operation. For example, the two forces that are present during the static AFM operation in addition to the van der Waals repulsion force are mentioned below:

5.1. Applied force by the cantilever

The force exerted by the cantilever is like the force of a compressed spring. The size and sign (attraction or repulsion) of the cantilever force depends on the cantilever deflection and its spring constant.

5.2. capillarity force

Capillarity force is usually applied by a thin layer of water (which may be caused by the humidity of the environment) [13]. Capillarity force occurs when a layer of water forms around the needle. In this case, a strong attraction force of about 10^{-8} newton appears, which keeps the needle in contact with the surface. The magnitude of the capillarity force depends on the distance between the needle and the sample. As long as the needle is in contact with the sample, the capillarity force is constant. It is also assumed that the water layer is almost homogeneous. As a result, the variable force in static AFM must be compensated by van der Waals repulsion force. The size of the total force applied to the sample varies from 10^{-8}N (in a condition where the water almost pulls the needle towards the sample and the cantilever pushes it away from the sample) to a more common range of 10^{-6} to 10^{-7}N [3].

6. Working modes of atomic force scanning microscope

When working with an atomic force microscope, various forces contribute to the deflection of the AFM cantilever. Different forces mean short-range atomic forces between the atoms of the tip of the needle and the atoms on the surface of the sample. Among these forces, we can mention covalent forces or van der Waals forces of attraction and repulsion type. The dependence of the Vandals force on the distance between the needle and the sample is shown in Figure 1-6.

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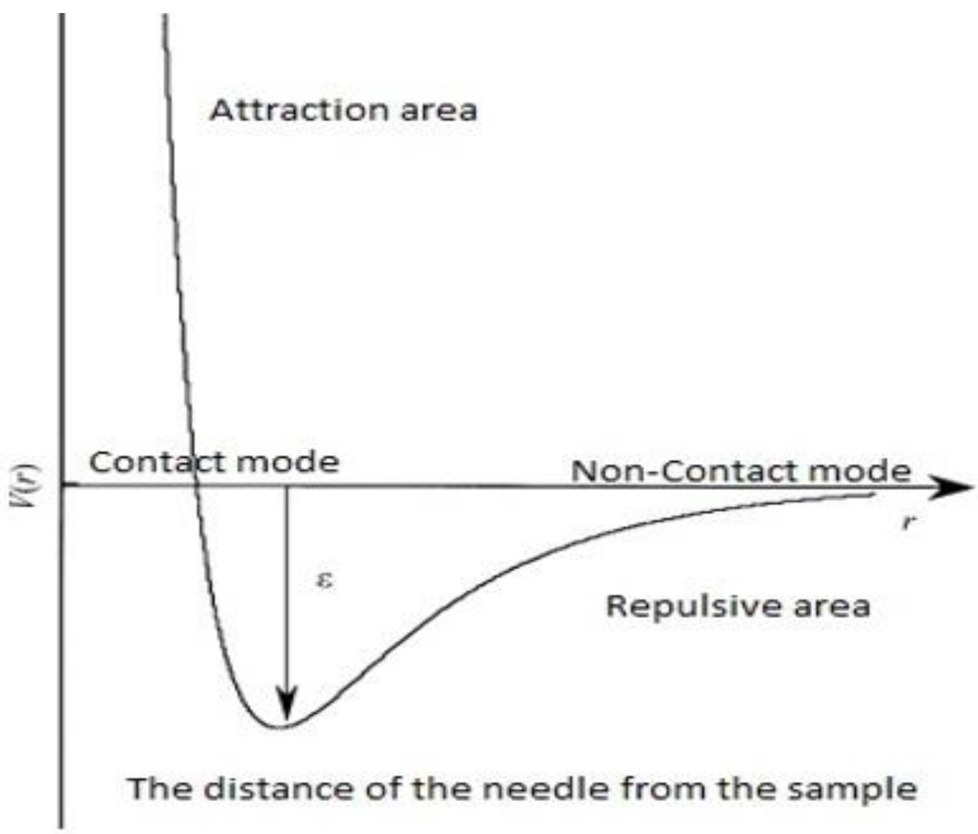

Fig. 6.-1. Potential energy diagram of probe and sample

6.1. Functional modes

In Figure 1-6, two states corresponding to two areas are marked:

1- Static mode (DC-AFM) or repulsive mode
2- dynamic mode (AC-AFM) or attraction mode

6.1.1. Static or contact mode

In the static state, the tip of the probe is lied at a distance of a few angstroms from the surface and is almost in contact with the surface. In this case, the force between the atom of the tip of the probe and the surface of the sample is repulsive, and the tip act in the region of van der Waals repulsive forces between tip atoms with the surface and without causing vibration on the cantilever. The force between the other atoms of the probe and the surface of the sample is still attraction. In the static mode, the depressions and elevations on the surface of the sample lead to bending of the cantilever and the device measures the deflection of the cantilever with a feedback mechanism and keeps it at a fixed point. The bending of the cantilever causes the reflection of the laser beam to shift on the cantilever, and this variation in the reflection angle is sent to the detector. When the tip is scanning the surface, imaging is done by recording the applied voltage to the moving piezoelectric. The contact mode is suitable for hard surfaces with thin tips, ultra-sharp and hard tips, and it is not suitable for samples that have a soft surface and causes damage to the surface of the sample. The use of this mode has a very suitable resolution than other modes.

Fig. 6.-2. Contact or static mode image

6.1.1.1. Fixed height mode

In the state that the height of the piezo scanner is fixed during scanning, the cantilever deflection variations can be directly used to generate topographic information. In this way, it is often used to create atomic-scale images of surfaces that are atomically flat. Here the deflections of the cantilever and therefore the variations in the applied force are small. Fixed height mode is required to capture simultaneous images of varying surfaces, where high scanning speed is necessary.

6.1.1.2. Fixed force mode

The cantilever deflection can be used as the input of a feedback circuit that the piezoelectric probe in confrontation the surface topography of the sample in such a way moves up and down in the z-direction that the cantilever deflection remains constant. In this state, the image is generated from the movement of the piezo scanner. By keeping the deflection of the cantilever constant, the total force applied to the sample will be constant. In the constant force mode, the scanning velocity is limited by the response time of the feedback circuit, but the total force applied by the needle on the sample is controlled. For many applications, the constant force mode is preferred.

6.1.2. Dynamic mode

In the dynamic mode, the probe oscillates with a specific frequency (100-400 Hz) and amplitude (a few tenths of Angstroms). In the dynamic mode, the cantilever is placed at a distance of several tens to several hundreds of angstroms from the surface of the sample, and in this state, the interatomic force between the cantilever and the sample (mainly due to long-range van der Waals interactions) is the at attraction force.

6.1.2.1 Non-contact dynamic mode

In the non-contact mode of the atomic force microscope, the tip acts in the region of van der Waals forces of attraction with the surface and by creating vibration on the cantilever. In this case, the cantilever oscillates near a natural resonance frequency. Then the sample approaches to reduce the cantilever amplitude to the determined value. In this way, the tip-sample interaction causes a sharp decrease in amplitude, when this distance reaches nanometer dimensions, the tip scans the surface of the sample. In this case, the feedback mechanism measures the amplitude of the oscillation and keeps it constant. The cantilever frequency variation is measured by a piezoelectric piece connected to the cantilever and used to make an image. This mode is more suitable for air and liquid environments. Since less force is applied to the sample, as a result, this method involves less destruction for soft and thin samples. Although it is more difficult to produce an atomic image with an atomic force microscope than with a scanning tunneling microscope, the non-contact dynamic mode can produce atomic resolution in a very high vacuum.

Fig. 6.-3. Non-contact or dynamic mode image

6.1.3. Tapping Mode

This mode is similar to the non-contact mode, with the difference that in the alternate contact mode, the tip of the vibrating cantilever gently hits the sample and the oscillation range is much larger than the non-contact mode. In this mode, imaging is done using the cantilever vibration amplitude. In this way, the cantilever strikes the surface of the sample with a certain frequency, and as a result, if there is the depressions and elevations, the amplitude of the cantilever's oscillations modifies, and this change is recorded by a piezoelectric piece connected to the cantilever.

Figure 4-6 shows a schematic force-distance curve for an atomic force microscope. At a far distance from the sample, the cantilever is not attracted by the interatomic force and it is in free equilibrium state. But when the cantilever approaches the surface of the sample, the gravitational forces attract the cantilever towards the sample. When the tip is in contact with the surface, repulsive forces The forces prevail and move the cantilever away. Gray boxes indicate the amplitude in contact and non-contact modes. The white box indicates the typical range of alternating contact.

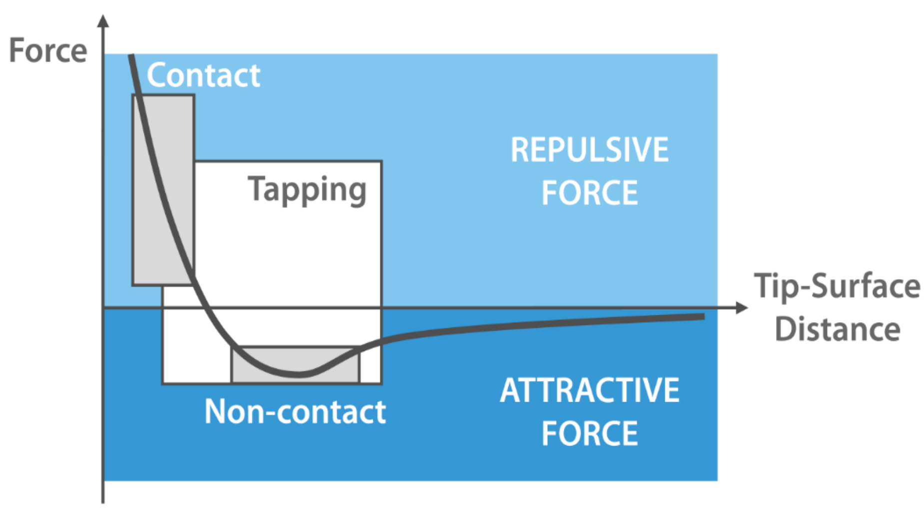

Fig. 6.-4. Atomic force microscope force-distance curve

7. Advantages and disadvantages of static and dynamic modes

The advantages of dynamic atomic force microscopes are that the topography of the sample can be measured without contact or with very small contact between the needle and the sample. The total force between the needle and the sample in dynamic mode is very low (usually around 10^{-12}N). This low force is advantageous for studying soft or elastic samples. Also, samples such as silicon wafers are not contaminated through needle contact. On the other hand, Because the force between the needle and the sample is small in the dynamic mode, it is more difficult to measure it than the several times larger force in the static mode.

In addition, the cantilever used for dynamic AFMs should be stiffer than the cantilever of static AFMs, Because the soft cantilever can be stretched to the side of the sample surface and be in contact with it. On the other hand, dynamic mode is preferred over static mode for measuring soft samples. Low force and stiffness of the cantilevers, in dynamic mode, are both factors that reduce the dynamic AFM signal. In the dynamic mode, there is no problem of needle or sample loss, which is sometimes observed after many scans by the static mode.

For solid samples, static and dynamic AFM images may look the identical. But if, for example, several layers of water have condensed on the surface of a solid sample, the images may be completely different. An atomic force microscope operating in static mode can penetrate this layer and image the subsurface, while in dynamic AFM mode, it images the liquid surface. Table 7.1. shows the strengths and weaknesses of AFM in different working modes.

7.1. Static(contact) mode properties

- Static contact mode is the simplest mode to receive the main information of indentation and protuberance of solid surfaces.
- The force between the needle tip atoms and the sample atoms is repulsive.
- High velocity
- Suitable for hard surfaces
- Ability to check friction
- Measuring the amount of elasticity/softness
- The possibility of damaging soft samples

7.2. Dynamic(non-contact) mode properties

- The force between the needle tip atoms and the sample atoms is attraction.
- Long life of the needle
- Low resolution
- The surface must be completely clean.
- Very high vacuum

Table 7.1. Strengths and weaknesses of work modes

Properties	Static(contact) mode	Dynamic(non-contact)	Tapping Mode
strengths	High scanning speed and achieving atomic resolution Easier scanning of rough samples with maximum variation in vertical topography	No destruction in soft samples due to the application of low force to the surface of the sample (both lateral and normal forces have the lowest value) Atomic resolution in Very high vacuum environment	Elimination of side forces in most cases Higher lateral resolution in most samples Less forces (resulting in less damage to the sample or probe).
weaknesses	The possibility of image distortion by lateral forces The application of large vertical forces in the interaction between the sample and the needle affected by the capillary forces resulting from the existence of a fluid layer Reducing the spatial resolution and destroying the soft surfaces of the sample due to the combination of other forces	Lower lateral resolution and lower scanning speed (to avoid contact with the fluid layer) Limitation in the type of sample (it is usually used in highly hydrophobic samples with a minimal fluid layer) and the thickness of the Superficial absorbed layers and the impossibility of effective measurement.	Scanning speed lower than contact mode

8. Summary and conclusion of atomic force scanning microscope working conditions

In atomic force microscopy, the force between the probe needle and the sample surface, which causes the cantilever to bend, is measured by the detector. In addition to the fact that these microscopes can be used for various types of nanolithography and the production of nanostructures and nanomachining, they are also used to study mechanical properties, wear or scratches, etc. These microscopes work with two working modes: static (contact) and dynamic (non-contact). In static mode, the cantilever is located at a short distance from the sample surface, and when the surface of the sample is scanned by the needle, the static force causes the cantilever to bend. In this case, the force between the cantilever and the sample is the repulsive force. Static mode works with two working modes: constant height and constant force. In dynamic mode, the resonance frequency of the cantilever can be used as a measure of force change (or needle-to-sample distance change). In this case, the atomic force between the cantilever and the sample is of the type of attraction. In this case, there is no damage due to the lack of contact with soft samples, but the scanning speed is lower than in the contact mode.

AFM has three dominant capabilities: force measurement, topographic imaging, and manipulation.

- In force measurement, AFMs can be used to measure the forces between the probe and the sample as a function of mutual separation. It can be applied to force spectroscopy for the purpose of measuring the mechanical properties of the sample, such as the Young's modulus of the sample, which is a measure of stiffness.
- In imaging, the reaction of the probe to the imposed forces on the sample can be used to form a high-resolution three-dimensional image of the sample surface. This image is obtained by raster scanning the position of the sample relative to the tip and then recording the height of the probe in a constant probe-sample interaction.
- In manipulation, the forces between the tip and the sample can be used to change the properties of the sample in controlled ways. Examples of this atomic manipulation include: local stimulation of cells and scanning probe engraved.

At the same time as the topographic images are obtained, other characteristics of the sample can be measured locally and shown in the form of an image, usually similar to high-resolution topographic images. Examples of these properties are mechanical properties such as hardness and adhesion strength and electrical properties such as conductivity or surface potential. In fact, most of the extended SPM techniques are AFMs that use this mode.

8.1. Advantages and disadvantages of atomic force microscopy

- Advantages
  - High speed
  - Simplicity of sample preparation
  - Accurate height information
  - Ability to work in air, vacuum and liquids (vise versa electron microscopes)
  - Ability to study living biological systems

- Limitations
  - Limited vertical study range
  - Limited Magnification range
  - Dependence of the obtained information on the type of microscope tip
  - The possibility of damaging the microscope tip or sample

9. Application field of AFM device and sample conditions of AFM test in Center for Nanoscience and Nanotechnology- Institute for Convergence Science and Technology (ICST) - Sharif University of Technology

Typical values and conditions for AFM testing in common modes are summarized in Table 9.1.:

Table 9.1. AFM test values and conditions

Sample type	Limitation of use
Bulk samples	For solid samples, the largest size: 1 cm × 1 cm × 1 cm
Powder samples	In tenths of grams... with a suitable solvent (ethanol, etc.) for the synthesis of a suitable dispersed solution
Very thin thickness	Thin film samples, non-conductive samples

Introduction of the cantilever, probe and piezoelectric type(genus), scanner measurement accuracy and laser light radiation direction for AFM testing in common modes are summarized in Table 9.2.:

Table 9.2. The material of the scanner components, the measurement accuracy of the scanner and laser light radiation direction of the AFM device

laser light radiation direction	measurement accuracy of the scanner with more accuracy	measurement accuracy of the scanner with less accuracy	The material of the cantilever, probe and piezoelectric
45 degrees suitable for measuring lat eral force in two directions	The maximum window dimensions are 50mic×50 mic The minimum window dimensions are 100 nm×100 nm	The maximum window dimensions are 200 mic×200 The minimum window dimensions are 1 mic×1 mic	Cantilever and probe: silicon, silicon nitride (anti-wear) Piezoelectric: lead zirconate titanate (PZT)

Nominal accuracy of AFM	AFM diagnostic accuracy
horizontal in nanometer size vertical in tenths of nanometers size	It depends on the material of the sample and the interaction of attraction and repulsion forces

AFM measurements can be performed with a conducting SPM probe which can also be used as AFM probe in DC or AC mode. When such a probe is used, it is especially important to secure an electrical connection, not only between the SPM probe and SPM probe holder, but also between sample surface and the external bias generator. It should be checked that the probe signal goes to the external current meter. Conducting SPM probes are fixed in the probe holder with conductive glue (silver,...). If you want to ascertain the electrical connection you can carefully measure the resistance on the probe with a multi measurement instrument to verify the electrical connection before performing further electrical connections.

For performing the measurement do the following steps,

1. Mount a cantilever which has been glued with conducting glue. It may be possible that a special cantilever coating is needed to measure the desired sample.

2. Use a wire to electrically connect the sample to the external bias generator as described

When the cantilever has been correctly mounted, the next steps with a DS 95 scanner are to choose the scan mode, perform a laser adjustment and find the resonance frequency of the cantilever if the AFM is operated in the AC mode. For a DS 45 scanner, there is only one scan mode and the laser adjustment has been made manually.

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