Photochemical Material Analysis
(광화학소재분석기술)

:Surface and Thin Film Analysis
(표면 및 박막분석)

4. Scanning Probe Microscopy

Atomic Force Microscopy

Scanning Probe Microscopy

Introduction

- Atomic force microscopy is an amazing technique that allows us to see and measure surface structure with unprecedented resolution and accuracy

- Very small images only 5 nm in size, showing only 40–50 individual atoms, can be collected to measure the crystallographic structure of materials, or images of 100 micrometres or larger can be measured, showing the shapes of dozens of living cells at the same time

- Since its invention in the 1980s, AFM has come to be used in all fields of science, such as chemistry, biology, physics, materials science, nanotechnology, astronomy, medicine, and more

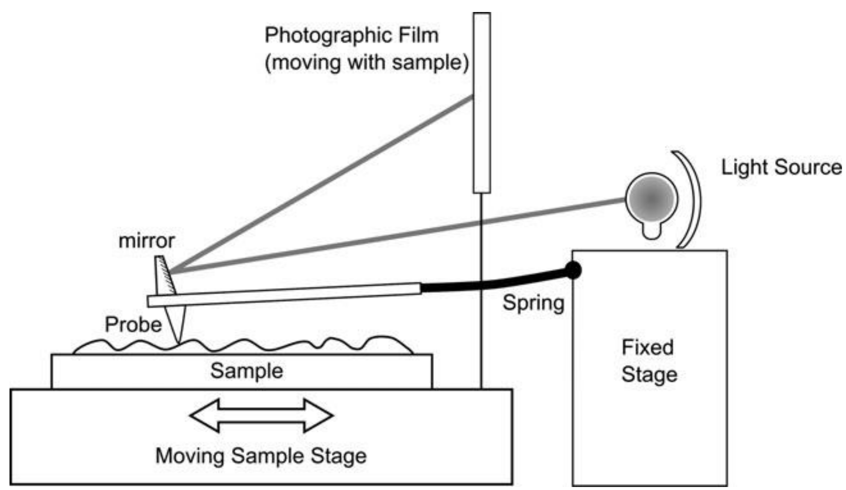
- An AFM physically 'feels' the sample's surface with a sharp probe, building up a map of the height of the sample's surface. This is very different from an imaging microscope, which measures a two-dimensional projection of a sample's surface.

Scanning Probe Microscopy

Introduction: Background to AFM

- The AFM works by scanning a probe over the sample surface, building up a map of the height or topography of the surface as it goes along

- The predecessor of the AFM was the **stylus profiler**, which used a sharp tip on the end of a small bar, to which was dragged along the sample surface, and built up a map, or more often a linear plot, of sample height



Optical lever design used for one of the early models of a surface profiler in the 1920s. This profiler had a vertical resolution of approximately 25 nm.

Scanning Probe Microscopy

Introduction: Background to AFM

- A magnified profile of the surface was generated by recording the motion of the stylus on photographic paper. This type of 'microscope' generated profile 'images' with a magnification of greater than 1000 ×

- A common problem with **stylus profilers** was the possible bending of the probe from collisions with surface features

- Fundamental problems with this sort of instrument persist, notably that the probe touches the surface in an uncontrolled way, which can lead to probe damage in the case of a hard sample, and sample damage in the case of a soft sample.

Scanning Probe Microscopy

Introduction: Background to AFM

Non-contact type of stylus profiler

- In 1971 Russell Young: called the topografiner
- Young used the fact that the electron field emission current between a sharp metal probe and a surface is very dependent on the probe sample distance for electrically conductive samples

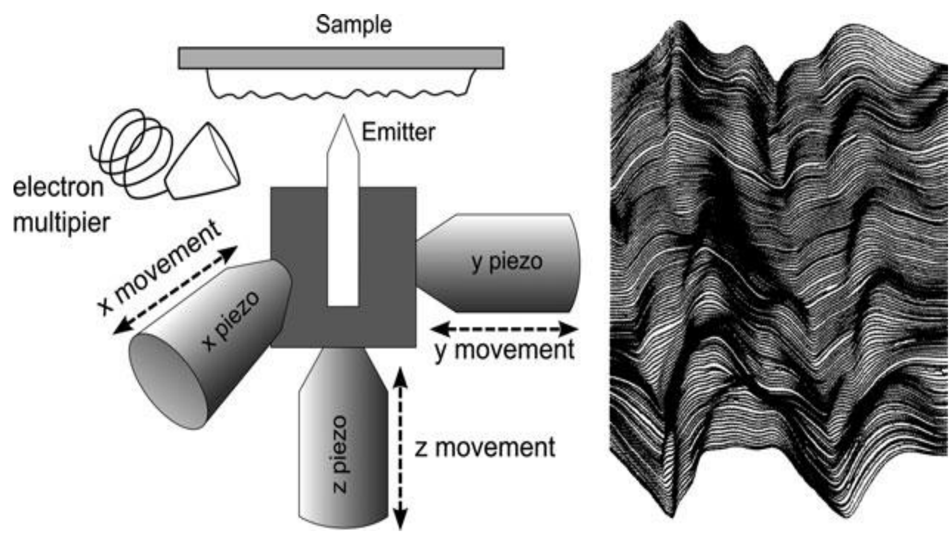


Fig. 1.2. A schematic diagram of Young's topografiner (left), and one of the first images collected with the instrument (right). Reprinted with permission from [12].

Introduction: Background to AFM

- The probe was mounted directly on a piezoelectric ceramic element which was used to move the probe in a vertical direction (z) above the surface

- An electronic feedback circuit monitoring the electron emission was then used to drive the z-axis piezoelectric element and thus keep the probe-sample distance at a fixed value. Then, with the x and y piezoelectric ceramics, the probe was used to scan the surface in the horizontal (X-Y) dimensions.

- By monitoring the *X-Y* and *Z* position of the probe, a 3-D image of the surface was constructed. The resolution of Young's topografiner was limited by the instrument's vibrations.

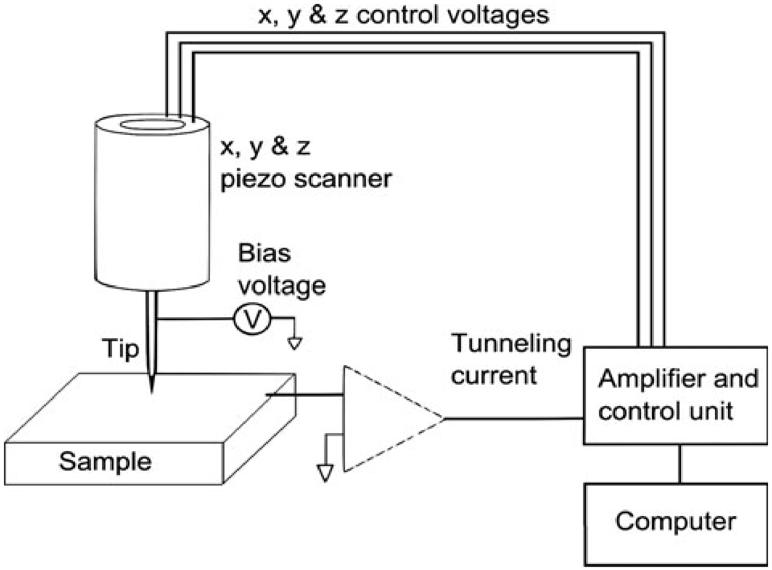
Scanning Probe Microscopy

Introduction: Background to AFM

Scanning tunnelling microscope (STM)

- In 1981: IBM, were able to improve the vibration isolation of an instrument similar to the topografiner such that they were able to monitor electron tunnelling instead of field emission between the tip and the sample

-> the first scanning tunnelling microscope (STM)



Simplified schematic of a scanning tunnelling microscope (STM).

Scanning Probe Microscopy

Introduction: Background to AFM

Scanning tunnelling microscope (STM)

- The STM works by monitoring the tunnelling current and using the signal, via a feedback loop, to keep the STM tip (a sharp metal wire) very close to the sample surface while it is scanned over the surface in the X and Y axes in a raster pattern

- Like the topografiner, the movement of the tip over the surface in x, y and z is controlled with three piezoelectric elements

- The distance the z piezo has to move up and down to maintain the tunneling current at the same value is equivalent to the sample height, so the computer can build up a map of sample height as the tip scans over the surface

- Despite the amazing results obtained with STM, the limitation to conducting samples led the inventors to immediately think about **a new instrument that would be able to image insulating samples**

Scanning Probe Microscopy

Introduction: Background to AFM

Atomic Force Microscopy (AFM)

- In 1986 Binnig, Quate and Gerber published a paper entitled 'Atomic Force Microscope'

- They described how they replaced the wire of a tunnelling probe from the STM with a lever made by carefully gluing a tiny diamond onto the end of a spring made of a thin strip of gold

  -> the cantilever of the first AFM

- The AFM caused a revolution. Suddenly, with a relatively cheap and simple instrument, extremely high-resolution images of nearly any sample were possible

- Oscillating modes have further increased the range of samples that AFMs can scan, and reduced the chance of sample damage as well

Scanning Probe Microscopy

Introduction: Background to AFM

Atomic Force Microscopy (AFM)

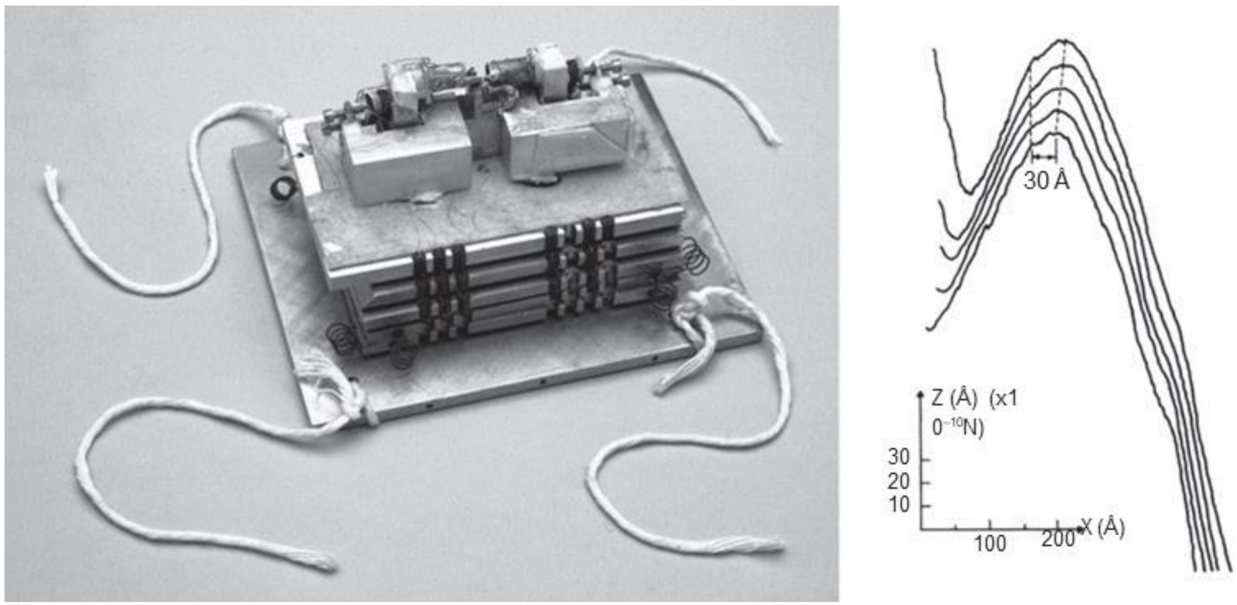


Fig. 1.5. The first AFM instrument built by Binnig, Quate and Gerber in the Science Museum, London (image copyright Science Museum/SSPL), and the first AFM image – reprinted with permission from[19]. Copyright 1986 by the American Physical Society.

Introduction: Background to AFM

Atomic Force Microscopy (AFM)

- One of the great advantages of the AFM is the ability to magnify in the X, Y and Z axes
- One of the limiting characteristics of the AFM is that it is not practical to make measurements on areas greater than about 100 μm
- This is because the AFM requires mechanically scanning the probe over a surface, and scanning such large areas would generally mean scanning very slowly.

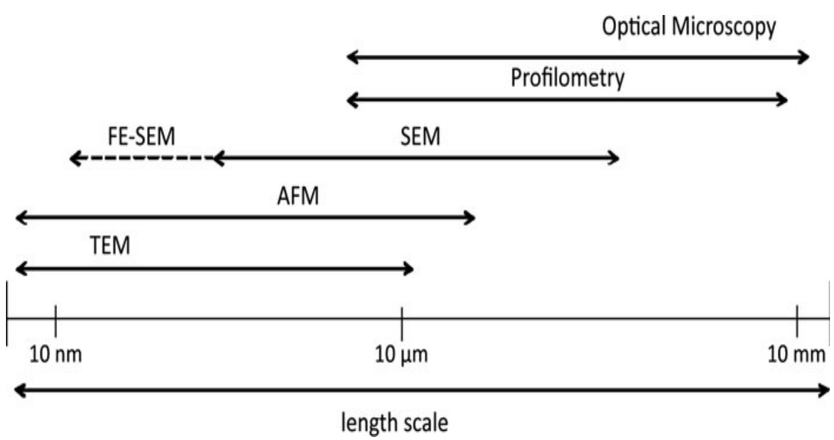


Fig. 1.6. Comparison of length-scales of various microscopes.

Scanning Probe Microscopy

Introduction: Background to AFM

Atomic Force Microscopy (AFM) vs. SEM, TEM

Table 1.1. Comparison of AFM with SEM and TEM.

	AFM	SEM	TEM
Sample preparation	little or none	from little to a lot	from little to a lot
Resolution Relativ	0.1 nm	5 nm	0.1 nm
e cost	low	medium vacuum(SE	high va
Sample environment	any	M) or gas	cuum
		(environmental SEM)	
Depth of field	poor Conduct	good	poor cond
Sample type	ive or	conductive	uctive
	insulating		
Time for image Maxim	2–5 minutes	0.1–1 minute	0.1–1 minute
um field of view Maxi	100 μm	1 mm	100 nm
mum sample size Meas	unlimited	30 mm	2 mm
urements	3 dimensional	2 dimensional	2 dimensional

Scanning Probe Microscopy

Introduction: Background to AFM

Atomic Force Microscopy (AFM) vs. SEM, TEM

- In general, it is easier to learn to use an AFM than an electron microscope because there is minimal sample preparation required with an AFM, and nearly any sample can be measured

- With an AFM, if the probe is good, a good image is able to be measured

- AFM has the advantage of being able to image the sample with no prior treatment, in an ambient atmosphere

  -> This makes scanning quicker, and can also mean fewer artefacts are introduced by the vacuum drying, or the coating procedure

- On the other hand, AFM image recording is usually slower than an SEM, so if a large number of features on one sample are required, AFM may be considerably slower than SEM for the same sample.

AFM Instrumentation

Basic concepts in AFM instrumentation

Piezoelectric transducers

- Piezoelectric materials are electromechanical transducers that convert electrical potential into mechanical motion
- When a potential is applied across two opposite sides of the piezoelectric device, it changes geometry
- The magnitude of the dimensional change depends on the material, the geometry of the device, and the magnitude of the applied voltage

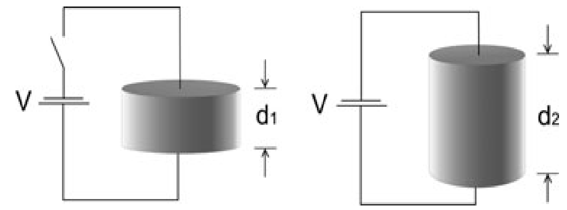


Fig. 2.2. A piezoelectric disk will expand radially ($d_2 > d_1$) when a voltage potential is applied to the top and bottom electrodes. The disk will change shape such that volume is preserved.

AFM Instrumentation

- Typically, the expansion coefficient for a single piezoelectric device is on the order of 0.1 nm per applied volt.

  -> Thus, if the voltage used to excite the piezomaterial is 2 volts, then the material will expand approximately 0.2 nm, or approximately the diameter of a single atom.

- It is the ability to accurately control such tiny movement that makes piezoelectric materials so useful for AFM

  -> piezoelectric materials are used for controlling the motion of the probe as it is scanned across the sample surface

Scanning Probe Microscopy

AFM Instrumentation

Force transducers

- The force between an AFM probe and a surface is measured with a force transducer

- Force transducers may be constructed that measure forces as low as 10 piconewtons between a probe and a surface. Typically, the force transducer in an AFM is a cantilever with integrated tip (the probe), and an optical lever

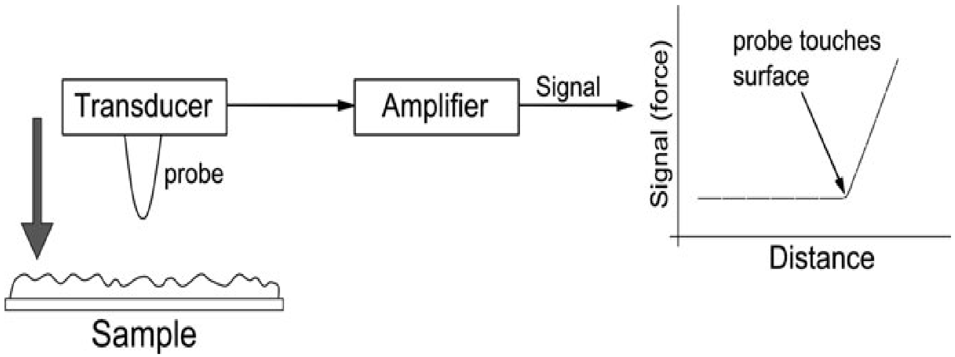


Fig. 2.3. Scheme of force transducer operation. The function of the transducer is to measure the force between the AFM probe tip and the sample surface.

Scanning Probe Microscopy

AFM Instrumentation

Feedback control

- Feedback control is used to maintain a set force between the probe and the sample

  -> AFM is more sensitive than a stylus profiler that simply drags a tip over the sample surface.

- The control electronics take the signal from the force transducers, and use it to drive the piezoelectrics so as to maintain the probe–sample distance, and thus the interaction force at a set level.

  -> Thus, if the probe registers an increase in force (for instance, while scanning the tip encounters a particle on the surface), the feedback control causes the piezoelectrics to move the probe away from the surface

  -> If the force transducer registers a decrease in force, the probe is moved towards the surface

Scanning Probe Microscopy

AFM Instrumentation

Feedback control

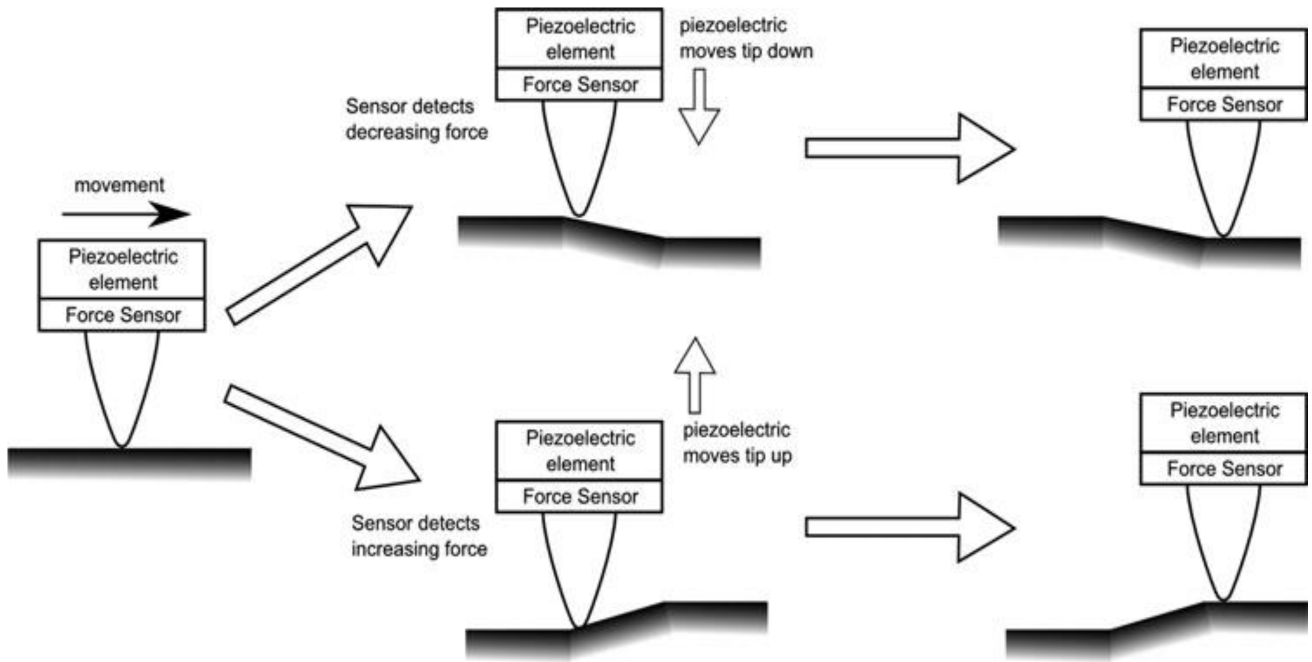


Fig. 2.4. Schematic of feedback control; when the force sensor senses a change in sample height, the piezoelectric moves to maintain the same tip–sample force.

Scanning Probe Microscopy

AFM Instrumentation

AFM block diagram and requirements

- The force transducer measures the force between the probe and surface; → the feedback controller keeps the force constant by controlling the expansion of the *z* piezoelectric transducer.

- Maintaining the tip–sample force at a set value effectively also maintains the tip–sample distance fixed.

- Then, the *x–y* piezoelectric elements are used to scan the probe across the surface in a raster-like pattern.

- The amount the *z* piezoelectric moves up and down to maintain the tip–sample distance fixed is assumed to be equal to the sample topography.

- In this way, by monitoring the voltage applied to the z piezo, a map of the surface shape (a height image) is measured.

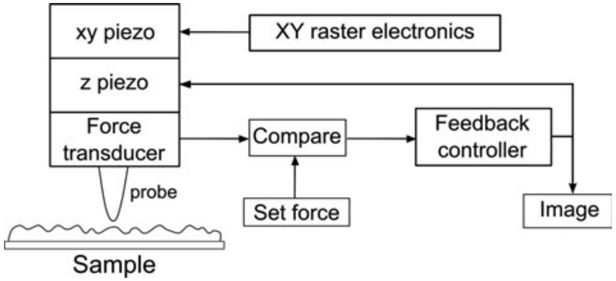


Fig. 2.5. Block diagram of AFM operation.

Scanning Probe Microscopy

AFM Instrumentation

Engineering Challenges for successful atomic force microscope

- A very sharp probe must be constructed so that high-resolution images are measured.
- To get the probe within the scanning range of the surface, a macroscopic translation mechanism must be constructed.
- The force transducer must have a force resolution of 1 nN or less so that the probe is not broken while scanning.
- A feedback controller that permits rapid control so that the probe can follow the topography on the surface must be created.
- An X-Y-Z piezoelectric scanner that has linear and calibrated motion must be used.
- A structure that is very rigid must be constructed so that the probe does not vibrate relative to the surface.
- A high-speed computer that can display the images in real time as they are collected must be used.
- A stage that allows rapid exchange of the probe used for scanning must be created.

AFM Instrumentation

AFM stage

The AFM stage is the heart of the instrument: **probe and sample holders**

There is a coarse approach mechanism, the *Z* motor, which can move the AFM scanner towards the sample. There is also an *X*–*Y* positioning stage which is not required but is useful for positioning the feature for imaging under the probe.
To help with this, there is usually an optical microscope for viewing the probe and surface.

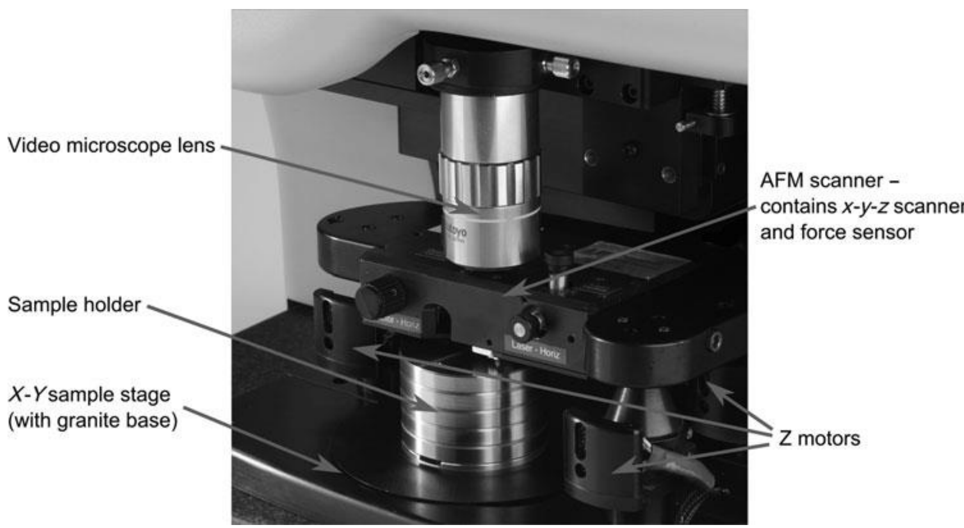


Fig. 2.6. Photo of an AFM stage, with components highlighted.

Scanning Probe Microscopy

AFM Instrumentation

AFM stage

– The design of all AFM instruments can be divided into two different configurations

-> the sample is scanned and the force sensor is held in one place (Lift)
-> the sample is held fixed and the probe is scanned (right)

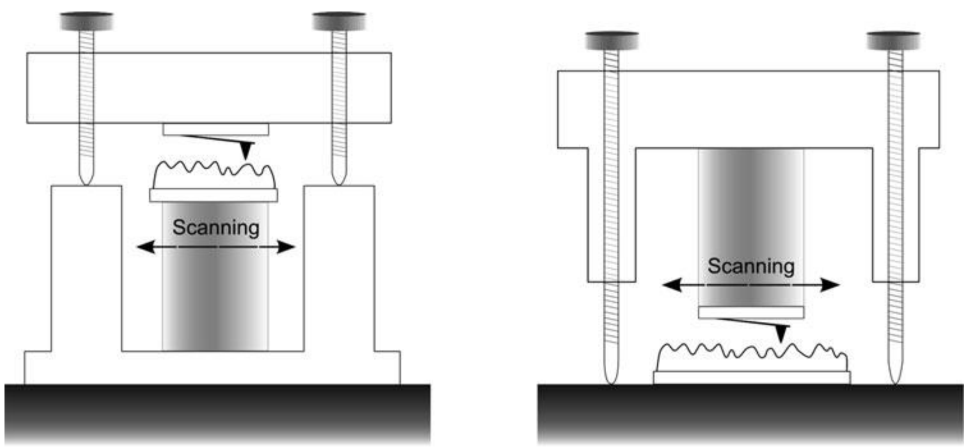


Fig. 2.7. The difference between **sample-scanning (left)** and **probe-scanning (right)** microscopes. In a sample scanning AFM the sample is mounted on an *x*-*y*-*z* scanner and the force sensor remains fixed. In the probe scanning AFM the sample remains fixed and the probe is scanned. The advantage of a probe scanning AFM is that it can scan larger samples.

AFM Instrumentation

AFM stage

- The design of all AFM instruments can be divided into two different configurations

  -> the sample is scanned and the force sensor is held in one place (Lift)

  - The mass of the sample is included in the feedback loop, reducing the size of sample that may be probed, as well as <u>practical limits on the sample's dimensions</u>

  -> the sample is held fixed and the probe is scanned (right)

  - The advantage of the probe scanning (also known as tip-scanning) microscope is that it can be used on any size of sample
  - Because there is nothing underneath the scanning probe except the sample, it is simple to add accessories to this type of microscope
  - the construction of a probe scanning microscope is much more difficult, as the whole tip-optical-lever assembly must be moved while scanning, and <u>care must be taken not to introduce further vibrations</u> from the scanning mechanism into the probe.

Scanning Probe Microscopy

AFM Instrumentation

X-Y-Z scanner

- The scanners used for moving the probe relative to the sample in an AFM are constructed from piezoelectric materials.

  -> This is because such piezoelectric materials are readily available, easily fabricated in desirable shapes, and cost effective

Piezoelectric scanners

- The most common types of piezoelectric materials in use for AFM scanners are constructed from amorphous lead barium titanate, $PdBaTiO_3$ or lead zirconate titanate, $Pb[Zr_xTi_{1-x}]O_3$, $0 < x < 1$ (usually abbreviated as PZT).

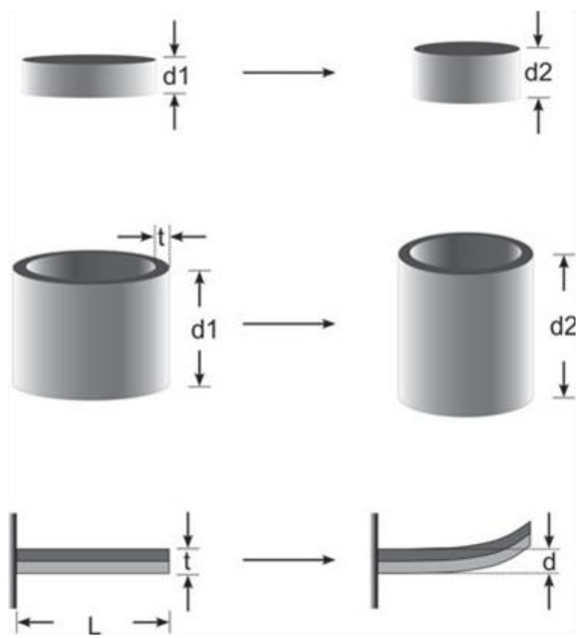
Scanning Probe Microscopy

Atomic Force Microscopy

AFM Instrumentation

Piezoelectric scanners

- Piezoelectric materials can be fabricated in several shapes such that they have more or less motion



$$d_2 - d_1 a V \quad (2.1)$$

where V = voltage applied

$$d_2 - d_1 a \frac{d_1}{t} \times V \quad (2.2)$$

where V = voltage applied

t = tube wall thickness

$$d a \frac{L^2}{t} \times V \quad (2.3)$$

where V = voltage applied

t = bimorph thickness

L = bimorph length

Fig. 2.8. Typical geometries for piezoelectric elements used in AFM. From top: piezoelectric disk, tube and bimorph scanners.

Scanning Probe Microscopy

AFM Instrumentation

Piezoelectric scanners

- Ideally, the piezoelectric ceramics would expand and contract in direct proportion to the driving voltage

-> Unfortunately, this is not the case, and all piezoelectric materials show non-linear behavior. They show two primary non-ideal behaviors, hysteresis and creep.

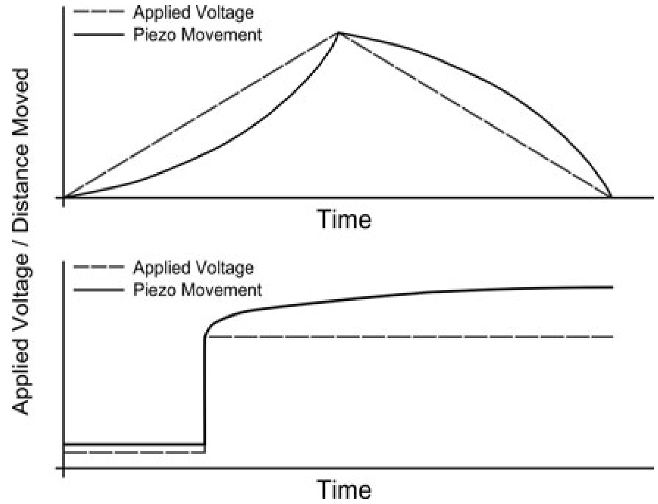


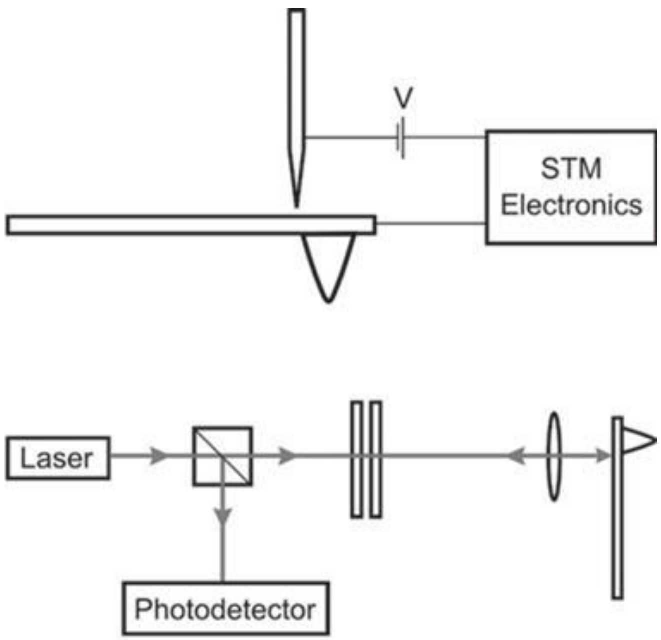
Fig. 2.9. Examples of non-linear behaviour in piezoelectric scanners. Top: hysteresis; when a voltage ramp is applied to the piezo, the response is non-linear. Bottom: creep; after an impulse applied to the piezo, the movement continues in the same direction.

Scanning Probe Microscopy

AFM Instrumentation

Force sensors

– The force sensor in an AFM must be able to measure very low forces. This is because, for a very sharp probe to be used, a low applied force is required so that the pressure (force/area) can be low enough so that the probe is not broken



Scanning Tunnelling Microscope:
In the original AFM built in 1985 a scanning tunnelling microscope tip was use to measure the motion of a cantilever [19]. Although this technique was viable, implementation and operation were very difficult.

Interferometer:
A Michelson interferometer can be adapted to measure the deflection of a cantilever in an AFM [37]. Although very sensitive, the interferometer was not successful because of fringe hopping. That is, the probe could jump between interference fringes while scanning.

Fig. 2.14. Different force sensors employed in AFM designs.

Scanning Probe Microscopy

Atomic Force Microscopy

AFM Instrumentation

Force sensors

Crystal oscillator: A piezoelectric crystal such as quartz can be used to measure the force between a probe and a surface [38]. If the probe mounted on the crystal is vibrated and positioned close to a surface, the interaction of the probe and surface will cause a change in the vibration. This change is proportional to force.

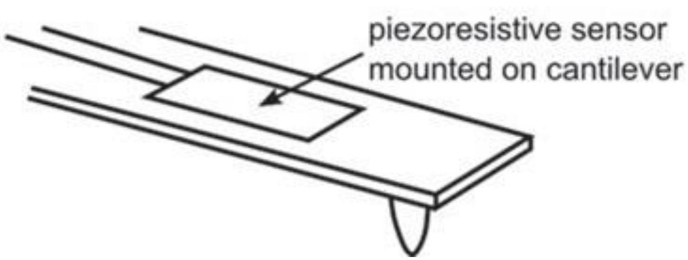


Fig. 2.14. Different force sensors employed in AFM designs.

Piezo-resistive cantilevers:
A cantilever can be fabricated that has a small piezo-resistive element in it that changes resistanceif the cantilever bends [39, 40].
This type of sensor is viable, but very difficult to manufacture in appropriate quantities.

-> With the advent of microfabricated cantilevers the optical lever AFM became the most widely used design for the force sensor in an AFM, and today, nearly all AFMs employ optical lever force sensors.

AFM Instrumentation

Force sensors – optical lever sensor

- A laser beam is reflected by the back side of a reflective cantilever onto a four-segment photodetector.

  -> If a probe, mounted on the front side of the cantilever, interacts with the surface the reflected light path will change.

- The force is then measured by monitoring the change in light detected by the four quadrants of the photodetector.

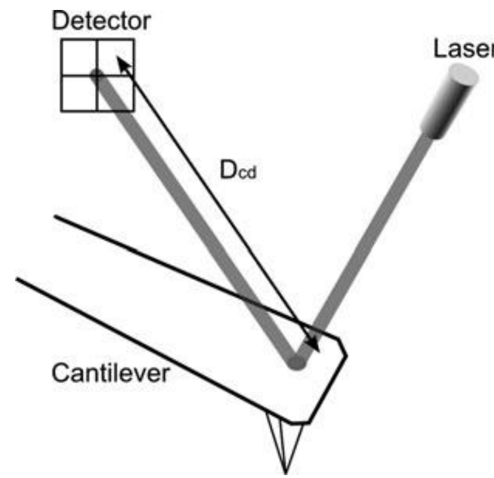


Fig. 2.15. Schematic diagram of the optical lever sensor. In an optical lever, as the end of the cantilever bends the position of the laser spot on the detector changes. As the cantilever – detector distance D_{cd} is large, a small movement of the cantilever causes a large change in the laser spot position at the detector.

Scanning Probe Microscopy

AFM Instrumentation

Force sensors – optical lever sensor

Integrating optical lever force sensors and scanners

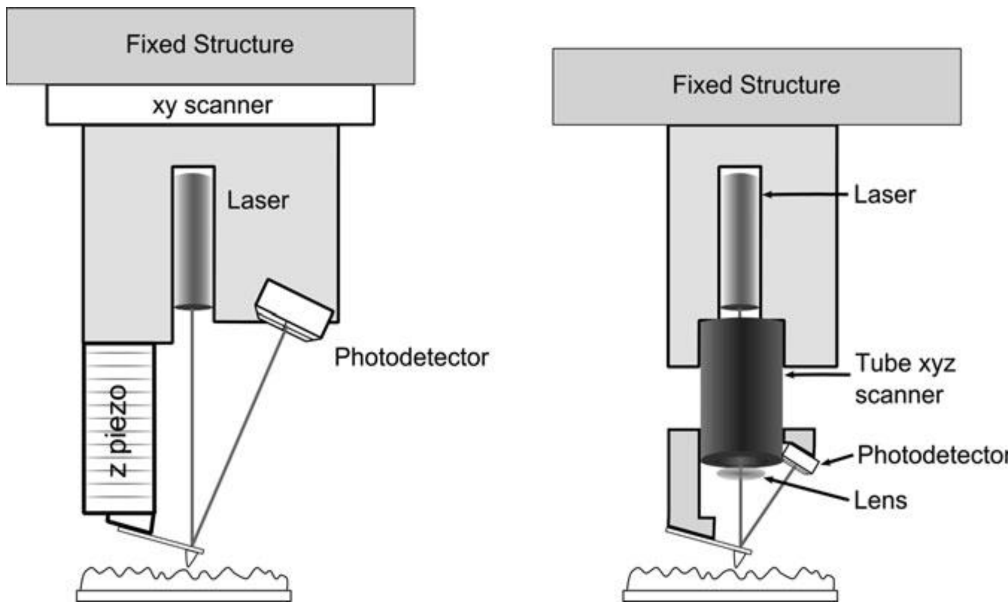


Fig. 2.16. Designs for tip-scanning AFMS with optical lever sensors. Left: the laser is scanned with the cantilever. Right: the laser is fixed and the cantilever is scanned, a lens keeps the laser light focused on the cantilever.

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