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Scanning Probe & Atomic Force Microscopy

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Welcome

MyScope was developed by Microscopy Australia to provide an online learning environment for those who want to learn about microscopy. The platform provides insights into the fundamental science behind different microscopes, explores what can and cannot be measured by different systems and provides a realistic operating experience on high end microscopes.

We sincerely hope you find our website: www.myscope.training an enjoyable environment. In there you can explore the microscopy space and leave ready to undertake your own exciting experiments.

Module Contents

Introduction

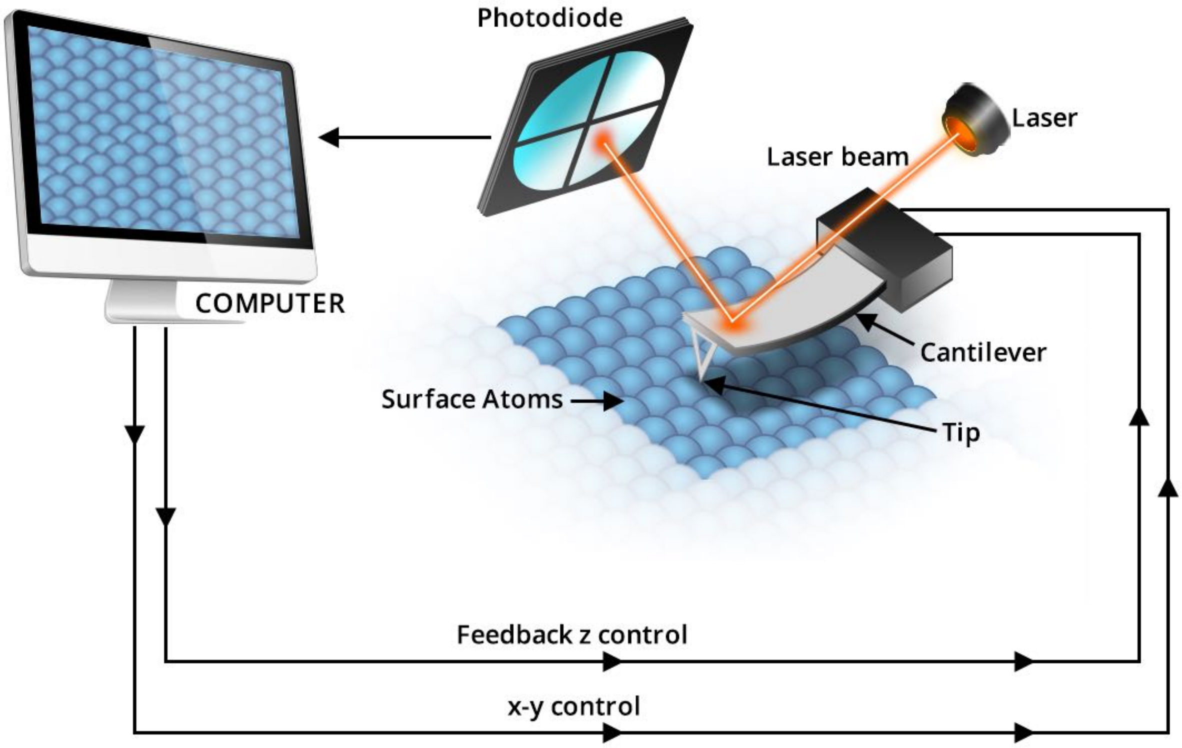
For the majority of people, the most well known types of microscopes are optical or electron microscopes. Optical microscopes focus a beam of light through a series of glass or quartz lenses for magnification of up to x1000. Electron microscopes create magnified images of samples by focusing an electron beam using magnetic fields produced by electromagnetic lenses composed of wire coils. The electron microscope improved the magnification of images to x100,000. However, both methods generate only two-dimensional images. A new technique was needed for the scientific community to provide accurate information in three dimensions.

This new technique had its genesis in 1981 when G. Binnig and H. Rohrer, from IBM research laboratories, invented a new type of microscope called a scanning tunnelling microscope (STM). For this invention they received the Nobel Prize in 1986. The most impressive feature of this new type of microscope is the extremely high spatial resolution of the order of 0.01 nm that can be achieved. This allows scientists to image, and even to manipulate, individual atoms or molecules of materials. The main difference between this technique and optical and electron microscopes is that there is no need for lenses, light or electron sources. The physical foundation for the STM is known as the tunnelling effect, a quantum mechanical property. The tunnelling effect can be produced by simply applying a voltage between a sharp metallic tip and the investigated surface, both separated by a vacuum barrier. If this vacuum barrier is approximately a few atomic diameters thick, electrons are able to tunnel through it, and a current will flow. This tunnelling current depends exponentially on the barrier distance or height. Therefore, by scanning the tip over the surface at either a constant current or height, the record of the vertical tip motion will reflect the surface topography of the sample.

The success of STM gave birth to a large family of instruments generally referred to as Scanning Probe Microscopes (SPM). Each member of this family uses a different type of interaction or force between the probing tip and the sample. The most widely implemented ones are the STM and the Atomic Force Microscope (AFM). The SPM family works on a principle similar to a record player. A sharp tip (e.g. silicon or silicon nitride in AFM, diamond in a record player) is scanned across the surface (the sample, or the record). The interaction between the tip and the surface is measured and converted into an electrical signal which is processed into interpretable results (three-dimensional image of sample topography, or sound from stereo speakers). However, unlike the record player, the sensing tip of an SPM is raster scanned across the sample (similar in nature to how a television image is produced). In addition to topographic imaging, many modern AFMs have the capability to image via a number of different mechanisms including frictional force, phase contrast, amplitude, adhesion and elasticity. Interactions producing electrostatic, magnetic, and chemical forces can also be mapped as long as the tip is capable of sensing these forces. For example, for magnetic force imaging the tip may be coated with iron or nickel.

AFM - Background information

The Atomic Force Microscope (AFM) operates by scanning an AFM probe across a sample surface. The AFM probe consists of a sharp tip at the end of a flexible cantilever that protrudes from a holder plate also called a holder chip.The tip is typically pyramidal or conical in shape and is four to five µm in height with a diameter at the apex of 10 to 20 nm, and is positioned at the end of a cantilever which is typically 100 to 200 µm long. The AFM probe is usually made from silicon or silicon nitride, with cantilever spring constants ranging from 0.05 to 50 N/m depending on the AFM mode of operation being employed. Either the probe or sample is mounted on a piezoelectric scanner which can move in the x,y, and z directions, and is used to raster scan the probe across the sample surface to acquire an image in 3 dimensions.



A schematic illustration of an atomic force microscope connected to a computer.

How does an AFM operate?

The AFM functions by scanning the sharp tip over a surface much the same way as in the olden day record player needle did to produce music. The tip is held at the end of a cantilever shaped like a diving board (figure_1). As the tip is repelled by or attracted to the surface by the intermolecular interactions (forces) between the atoms of the tip and the surface, the cantilever is deflected. The magnitude of deflection is monitored by a detection system, commonly a laser beam that reflects off the end of the cantilever at an oblique angle and onto a photodiode (figure_1). A feedback mechanism from the detector system to adjust the piezoelectric scanner is used to maintain either a constant force or a constant deflection (height) between the tip and sample surface, depending on the AFM set-up.

Imaging: Interactions

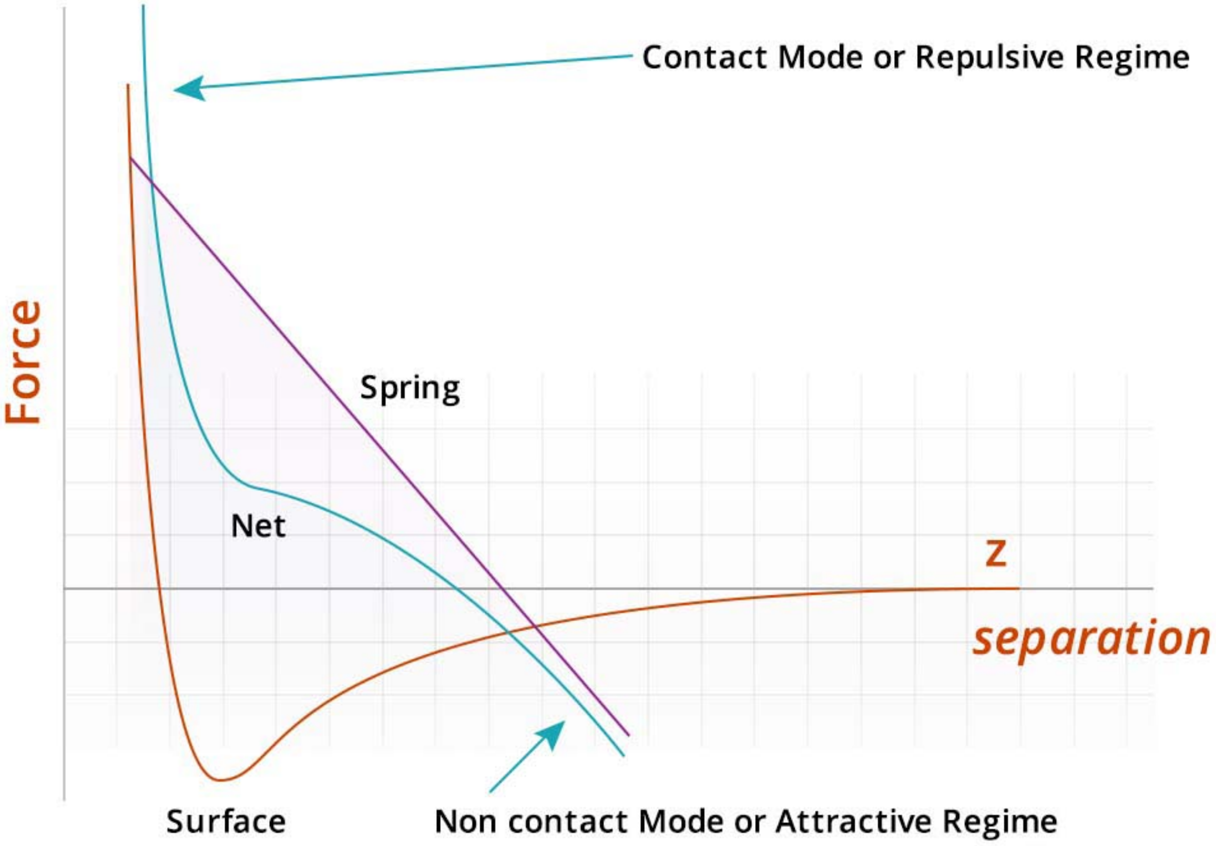
Interactions between the probe and sample

At very close tip-sample distances (a few angstroms), a strong repulsive force appears between the tip and the sample surface due to the overlap of atomic orbitals. The repulsive force increases as the distance between the tip and sample surface decreases.

As the probe tip scans the surface at this close distance, a feedback system to the piezoelectric scanner raises and lowers the sample to keep a constant repulsive force between the tip and the sample surface. A plot of this upward and downward motion (z), as a function of the tip x - y position on the sample surface, provides a high-resolution image of the surface topography. This mode of operation is called "contact mode". When the repulsive force is in place, the tip and sample are considered to be in 'contact'. High resolution is possible because very small changes in separation (z) (see figure_below), lead to large changes in force which is the parameter being monitored. However, for some samples such as biological specimens or soft material such as some polymers, 'contact' of the surface with the tip can damage the sample.

To overcome this problem, an alternative set-up is that the tip vibrates rapidly up and down and only 'taps' or comes very close to the sample surface while at the bottom of its oscillation. This mode, referred to as **"tapping mode"**, "semicontact mode", or "dynamic mode", is the most common mode of operation as it prevents sample damage. However, now changes in separation (z) lead to smaller changes in force (see figure_below), and hence the resolution of images from tapping mode is not as high as images from contact mode.

"Non-contact" mode AFM uses longer range forces which are attractive. These attractive forces occur at tip-sample surface distances of > 10 nm.



Various forces curves at play in AFM. The force curve for a spring (the cantilever) is a straight line (purple). The force curve for interactions between two atoms (brown) is the normal Lennard-Jones type curve. The net force curve (blue) shows the changes in forces measured by AFM.*
** An explanation of the Lennard-Jones curve: over short distance (z separation) the forces are repulsive due to overlapping electron orbits (e.g. Pauli repulsion); whereas at longer distances, the forces are attractive e.g. van der Waals force or dispersion force).*

What can you measure?

Measuring forces in the tip-sample space

Force spectroscopy

In addition to imaging the surface of samples, another major application of AFM is force spectroscopy. Force spectroscopy involves the direct measurement of forces between the tip and the sample surface as a function of the distance between the two. The result is called a force-distance curve.

The deflection of the cantilever is monitored as it is moved towards and away from the surface, and the deflection plotted as a function of piezoelectric displacement gives the force-distance curve. The horizontal axis in such a curve is typically the vertical distance moved by the sample stage. The vertical axis is the deflection of the cantilever as the tip is moved towards the sample surface, contacts and pushes against the surface and then away from the surface. The vertical axis can then be converted to force by multiplying the deflection of the cantilever by the spring constant of the cantilever (Hooke's law).

An example of a force distance curve is displayed in the figure_below. The blue curve is the approach cycle and the orange curve is the retract cycle. The different sections of the curve are defined as follows.

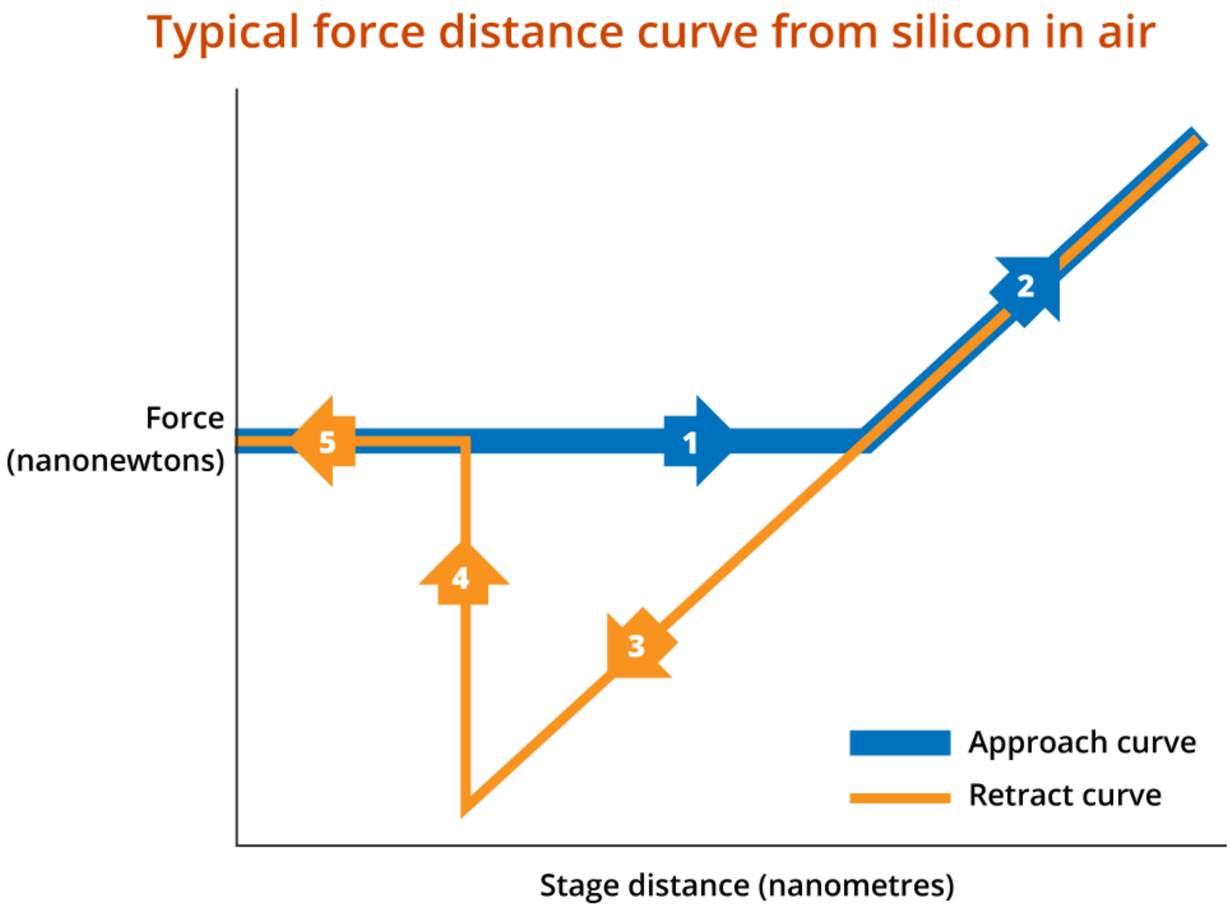
1. The probe is moving towards the sample surface and does not experience any force and therefore no change in deflection. Therefore the curve is flat. When the tip contacts the surface there is often observed a small snap-on section.

2. The probe is then pushed against the surface. This is known as the compliance regime and can be used to determine the mechanical properties of surfaces.

3. The approach cycle is completed and the retract cycle begins.

4. The snap-off section of the retract curve. This can yield information on tip-sample adhesion and has been used to determine bond strengths.

5. The tip has completely disengaged from the surface and the probe is now moving away from the surface. It now experiences no surface forces and therefore the deflection of the cantilever is zero and the curve is flat.



A typical force distance curve acquired on silicon in air

Calibration

In order to determine the force experienced by an AFM probe important calibrations must be performed. The first is converting the photodiode output from mV or nA (depending on the instrument manufacturer) to nm. This is usually done by performing force-distance curves against a surface much stiffer than the cantilever. The slope of the force curve in the compliance regime will then give the conversion factor or sensitivity. This is also sometimes referred to as the inverse optical lever sensitivity or INVOLS. Surfaces that can be used for such a calibration are typically mica, glass or silicon. For very stiff cantilevers sapphire is a good choice. The calibration surfaces should also be flat and clean and an average of at least 10 force curves on at least 3 separate locations on the sample surface should be acquired.

The next calibration that must be performed is determining the AFM cantilever spring constant. A large number of techniques now exist to calibrate this value. Methods are based on:

- **Beam theory**: These require knowledge of the cantilever dimensions and material properties.

- **Resonance methods**: Require knowledge of the resonance frequency of the cantilever and/or the thermal noise response of the cantilever.

Loading the cantilever with a known force

The appropriate method needs to be chosen and will depend on the type of cantilever used. All AFM users endeavouring to calibrate AFM cantilevers must make sure they use all appropriate corrections as many techniques require corrections due to, for example, the tip-offset distance, cantilever angle to the surface, or tip-height.

Uses

The forces that can be measured include atomic bonding, van der Waals forces, single molecule stretching and rupture forces. Force spectroscopy can be used to measure the dispersion force due to a polymer adsorbed on the surface of a substrate, or in biophysics, the mechanical properties of biological materials.

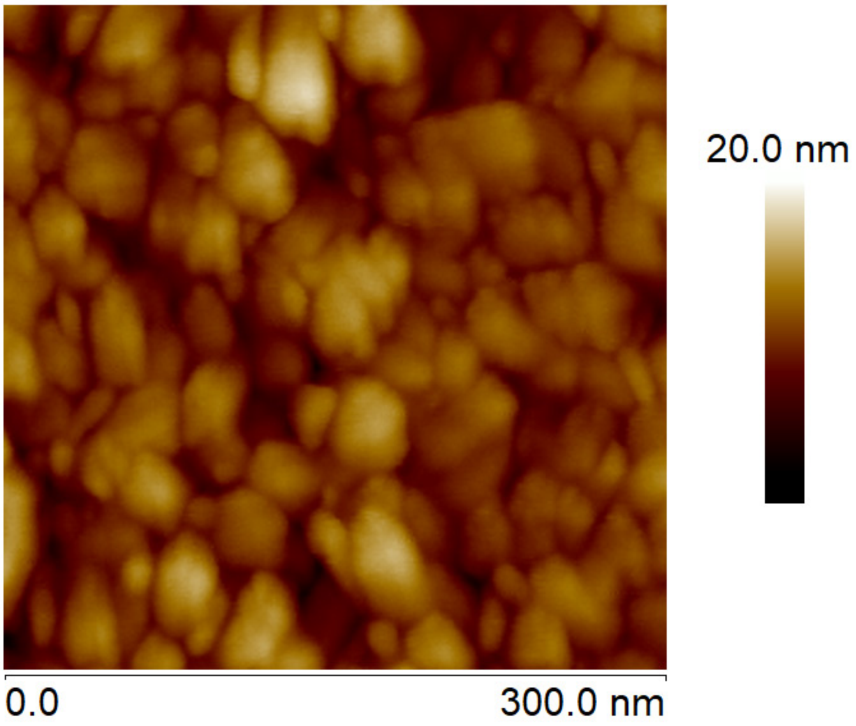
Imaging modes - Intro

AFM can operate in three modes according to how the AFM probe moves:

- Contact mode is also called static mode, and is where the probe does not oscillate and is always in very close contact with the sample surface.
- Tapping mode is a dynamic mode also called intermittent contact or semi-contact mode, and is where the probe oscillates such that the tip only comes into very close contact with the sample at the bottom of its oscillation.
- Non-contact is also a dynamic mode where the probe oscillates above the sample without the tip coming into very close contact with the surface.

Contact mode

Contact mode/ constant force AFM operates by scanning the tip across the sample surface while monitoring the change in cantilever deflection with a split photodiode detector. A feedback loop maintains a constant deflection between the cantilever and the sample by vertically (z) moving the scanner at each (x,y) data point. The vertical movement is used to build an image. By maintaining a constant cantilever deflection, the force between the tip and the sample remains constant. Spring constants usually range from 0.01 to 1.0 N/m, resulting in forces ranging from nN to μN in an ambient atmosphere. Operation can take place in ambient and liquid environments.

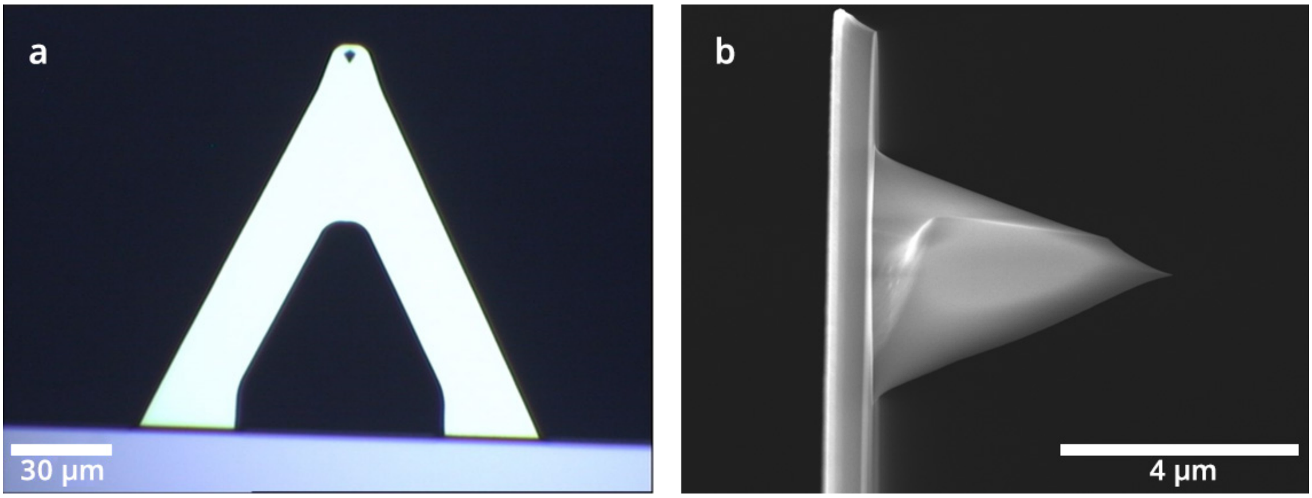


Contact mode/ constant height AFM operates by keeping the height (z) of the scanner constant, and using the cantilever deflection, as monitored by the split photodiode detector, to directly generate the topographical image. Contact mode/ constant height mode is most useful for atomic-scale images of atomically flat surfaces.

Contact mode is useful for obtaining 3D topographical information on nanostructures and surfaces. A disadvantage of this mode is that, because the tip is in constant 'contact' with the sample surface, large lateral forces can be exerted on the sample as the tip is raster scanned over the surface resulting in damaged samples and deformed images.

Tip choice for contact mode in air

Since the tip is in essence in contact with the surface, the spring constant of the cantilever is not as stiff as for tapping mode operation in air. Spring constants tend to be <1 N/m, and the cantilever geometry is generally V-shaped to minimize cantilever twisting and buckling. Resonant frequencies for the cantilevers of contact mode probes are usually between 10 to 80 kHz. In the figure_below, (a) Is an optical image of a typical contact mode probe while (b) is an SEM image of a typical contact mode probe showing the tip in more detail. The probes are usually made of silicon nitride and have tip diameters of approximately 20 to 40 nm.

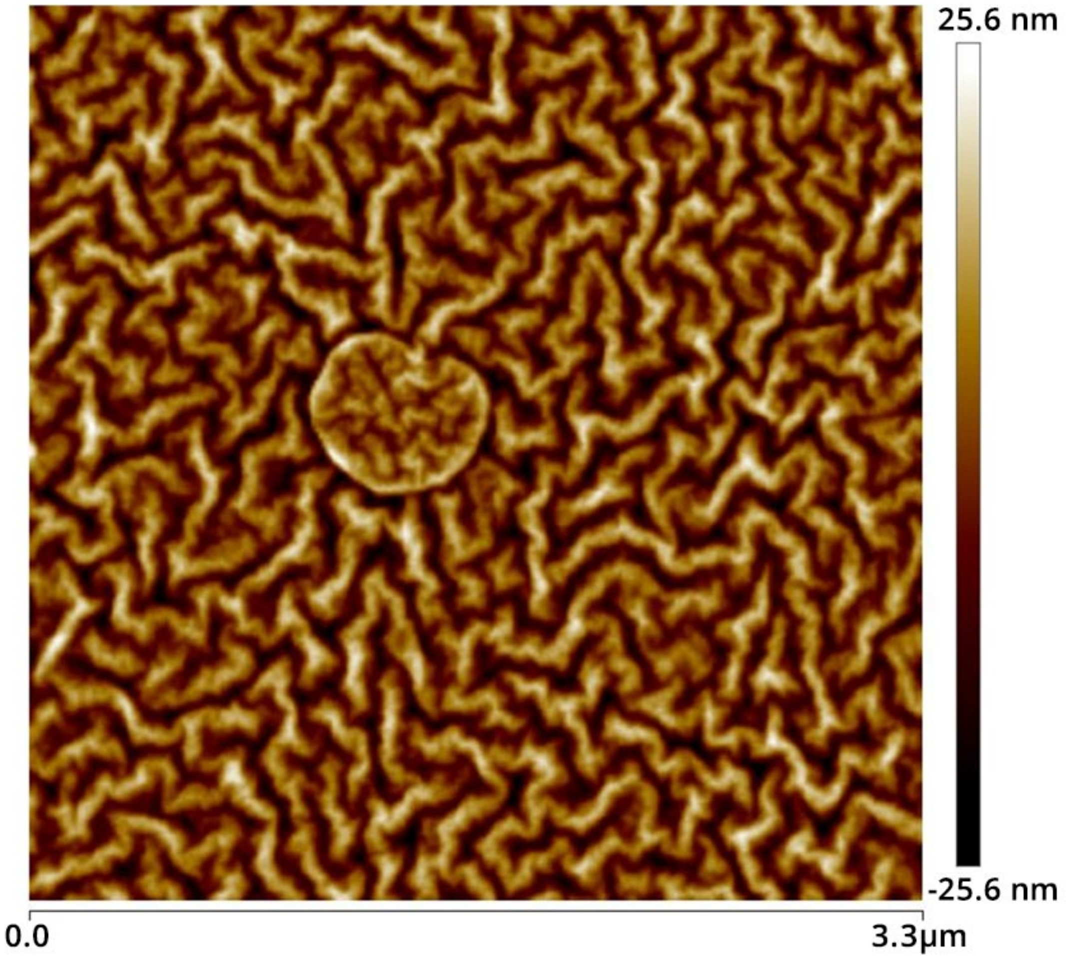


(a) Optical image of tapping mode cantilever. (b) SEM image showing the tip in more detail

Tapping mode

Tapping Mode AFM operates by scanning a tip attached to the end of an oscillating cantilever across the sample surface. The cantilever is oscillated at or near its resonance frequency with an amplitude ranging from 20 nm to 100 nm. The frequency of oscillation can be at or on either side of the resonant frequency of the cantilever. The tip lightly “taps” on the sample surface during scanning, contacting the surface at the bottom of each oscillation. Changes in the vibration frequency of the cantilever vibrating near its resonance frequency are measured. The feedback loop maintains a constant oscillation amplitude, and therefore, a constant tip-sample interaction is maintained during imaging.

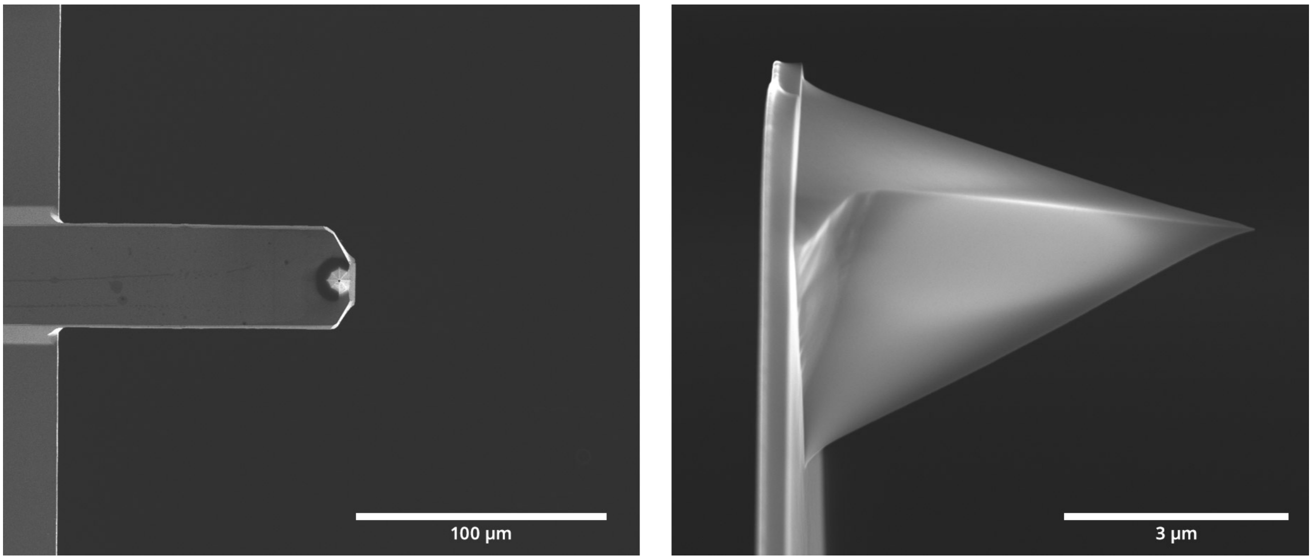
Like contact mode, tapping mode can be operated in ambient and liquid environments. Tapping mode has a number of advantages compared with contact mode, including greatly reduced lateral and normal forces being applied to the sample surface. Therefore, tapping mode is preferred to image samples with structures that are weakly bound to the surface or soft samples. The figure_below is an example of a tapping mode image in air of carbon nanotubes on a silicon surface.



Tapping mode AFM image of Cr nanoparticles embedded in the surface of a compliant epoxy substrate. The surface stress induces local curvature on the nanoscale. Image courtesy of Jitesh Hora and Drew Evans, University of South Australia.

Tip choice for tapping mode in air

The tip taps the surface and must not adhere or stick to the surface due to capillary forces. The cantilevers are therefore designed to have spring constants, on average, between, 2 N/m and 50 N/m. Resonant frequencies for tapping mode in air cantilevers are usually between 50 to 400 kHz. In the figure_below, left is an optical image of a typical tapping mode probe while in the right is an SEM image of a tapping mode probe showing the tip in more detail. The probes are usually made of silicon and have tip diameters of approximately 20 μm or less.



(a) Optical image of tapping mode cantilever. (b) SEM image showing the tip in more detail.

Non-contact mode

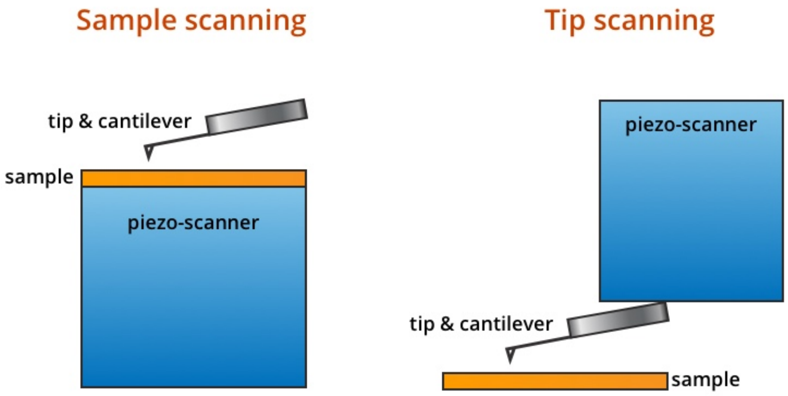
As the name implies, the (oscillating) tip is set further from the sample surface where it is surrounded by attractive forces, and the tip does not contact the surface at any stage.

In this mode, magnetic, electrostatic, van der Waals, and other longer range forces can be measured. Topography can be measured with some difficulty. The choice of constant force versus constant height experiments is still possible.

Specimen - Specimen choice

Specimen choice and preparation can be a limiting factor with any type of AFM.
The AFM piezoelectric scanners are usually limited to a maximum vertical movement of approximately five µm with a maximum x - y range of 100 x 100 µm. Therefore any surface with more than five µm of roughness will be extremely difficult to image with most AFMs.
For sample scanning systems sample size is also typically restricted to a maximum area of 1 x 1 cm and with a maximum sample thickness of approximately 3 mm.

For tip scanning systems sample size can be much larger and thicker.



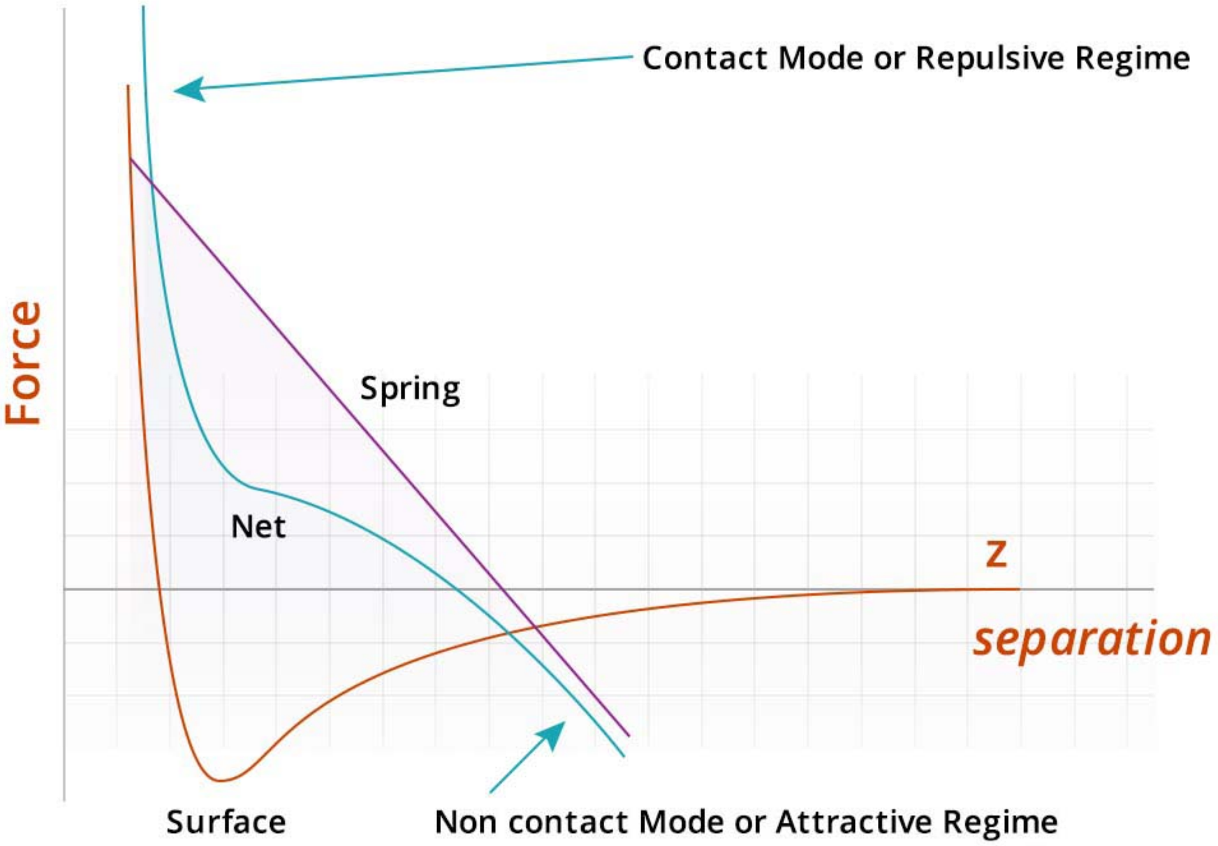
Sample and tip scanning set-ups for AFM. Tip scanning allows for a much larger and thicker sample.

The choice of mode for AFM should be based on the characteristics of the sample surface of interest and the hardness and stickiness of the sample.

- Contact and tapping modes
- Non-contact mode

Contact and tapping modes

This is done in the repulsive regime of the surface tip interaction.
Most topography is done in this mode. In the repulsive regime, small changes in distance give large changes in force and hence cantilever position. This means that the user gets the greatest sensitivity to measure the topography of the sample.



Various forces curves at play in AFM. The force curves for a spring (the cantilever is a straight line) while the force curve for interactions of atoms is the normal Lennard-Jones type interaction. The net force curve shows the changes in forces measured in AFM.

Constant force or constant height experiments can be done in these modes.

Contact mode is most useful for hard surfaces. However, the tip is in 'contact' with the surface at all times so it may get contaminated by loose material on the surface, and tips may become worn or damaged leading to tip artefacts in the resultant images. Soft materials and biological specimens may be damaged by imaging in contact mode. Also, if the sample is covered by an adsorbed liquid layer over the surface of the rigid sample, the liquid layer may not be imaged because in contact mode the tip remains so close to the surface it will penetrate the liquid layer and image the underlying surface.

Tapping mode lessens potential damage to both the specimen and the tip compared to contact mode. Tapping mode is most useful for biological specimens and samples with poor surface adhesion e.g. carbon nanotubules. Tapping mode can also image supported, thin liquid layers over rigid specimens.

Non-contact mode

Non-contact mode prevents tip and sample degradation from contact between the two, and is therefore good for soft samples such as biological specimens and thin organic films. If a very thin fluid layer is covering a rigid sample, in non-contact mode the tip may be oscillating above the fluid. Therefore the liquid layer and the underlying surface can be imaged simultaneously.

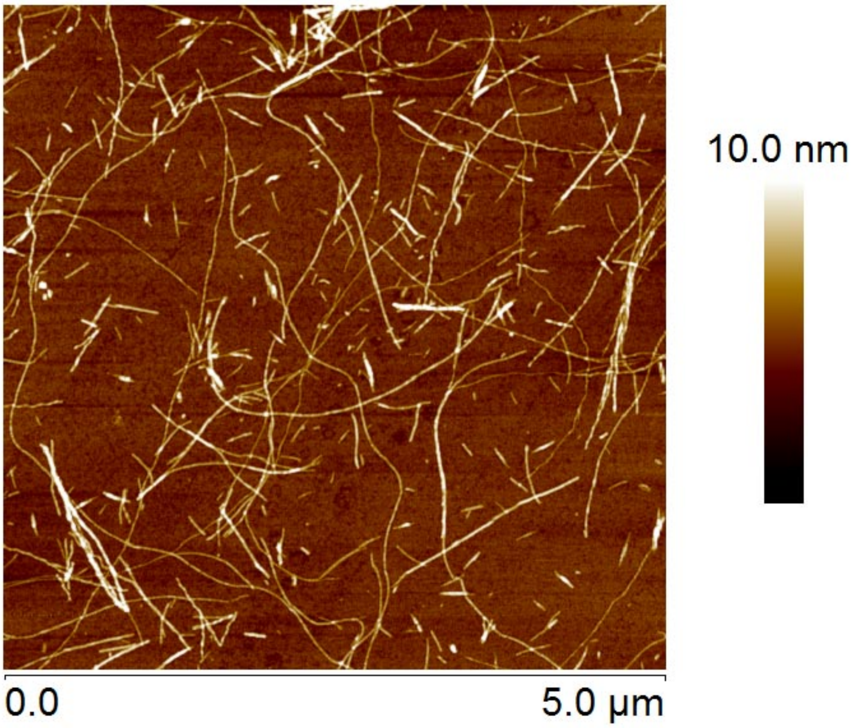
Under ambient conditons though, often a thin layer of water inherently forms on the tip and the sample surface. In non-contact mode, a 'bridge' of liquid may form between the tip and the sample surface due to capillary action, and this causes the tip to in effect 'contact' the surface. Therefore, accurate imaging using non-contact mode under ambient conditions is difficult. Non-contact mode AFM works best under ultra-high vacuum conditions and then atomic scale resolution is possible.

In non-contact mode the attractive forces are weak (see figure_in previous page), and the technique is sensitive to external vibration.

Data display

How is the Data Displayed?

Most Scanning Probe experiments produce data as a function of the sample's x and y position. This data can range from tunneling current, to force between sample and tip, to height to reach a certain force etc. In the vast majority of cases, the data is displayed as a false colour image. Typically high values of the parameters being measured are assigned lighter colours while low values are given darker colours. An example is shown in the following:

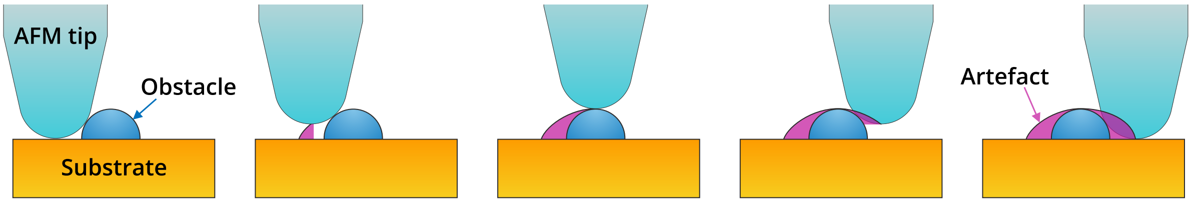


This is a 3D image in the x, y and z planes. The z plane is shown as a graded colour from dark (lowest) to light (highest).

Artefacts - Image artefacts

Tip convolution

Tip convolution is due to the radius of curvature of the tip being similar to or larger than the width of the feature being imaged. As the tip moves across the feature, the sides of the tip come into contact with the feature sooner than the apex of the tip, and the detector and feedback system responds accordingly. The effect is to broaden the x, y dimensions of the feature being imaged.



An AFM artefact (tip convolution) arises from using a tip with a similar or higher radius of curvature with respect to the feature which is to be visualized.

Tip convolution is an inevitable consequence of AFM imaging, but manufacturers of AFM probes are continually making tips with smaller diameters and higher aspect ratios. For the smallest diameter probes, silicon is currently the material of choice. Manufacturers claim to be able to produce tips with diameters less than 10 nm while the smallest silicon nitride tips are usually on the order of 20 - 30 nm in diameter.

The disadvantage of smaller diameter probes is that the pressure applied to the surface and the tip increases for a given imaging force. This can potentially result in damage to the surface or wear of the probe.

Carbon nanotubes promise to be able to produce the ultimate AFM tips. Their diameters can be as small 1 - 2 nm and their aspect ratio (length/width) can be measured in the thousands. This allows such probes to access parts of a sample ordinary AFM tips cannot. Carbon nanotubes are also incredibly strong materials that do not wear like silicon or silicon nitride. The disadvantage is attaching carbon nanotubes to AFM probes. A number of methods are available but as yet no manufacturer is mass producing carbon nanotube probes at a wafer scale.

Thermal drift

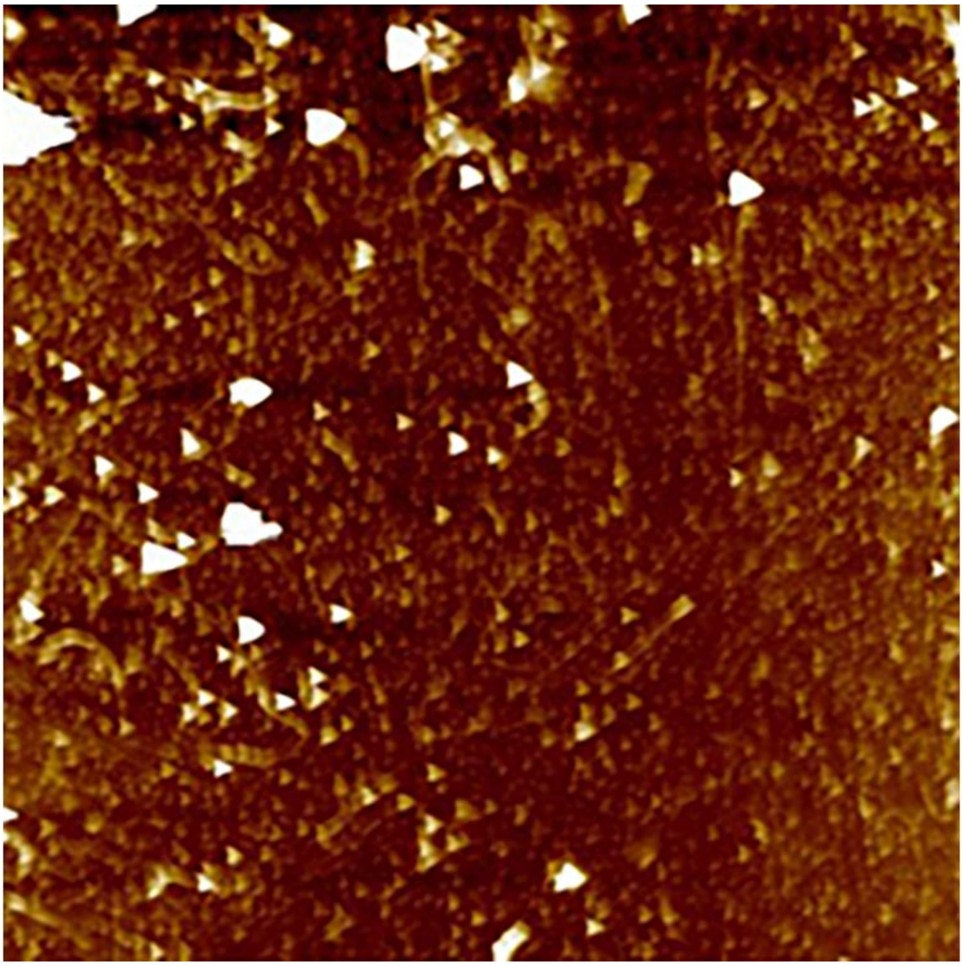
Thermal drift, as the name suggests, is a distortion of the image due to changes in the temperature of the probe-sample environment. AFM is sensitive to heat from external sources, for example lights, and internal self-heating components. Thermal expansion may result in the sample becoming 'loose' on the stage and moving as the sample is scanned. Drift results in image distortions including features appearing smaller or longer in one direction than they actually are.

Tip artefacts

Artefacts result from the tip either becoming contaminated by material on the sample surface or from wear due to scanning. The resultant image can possess a number of tip artefacts and it is important for AFM users to recognize these artefacts.

Worn tip

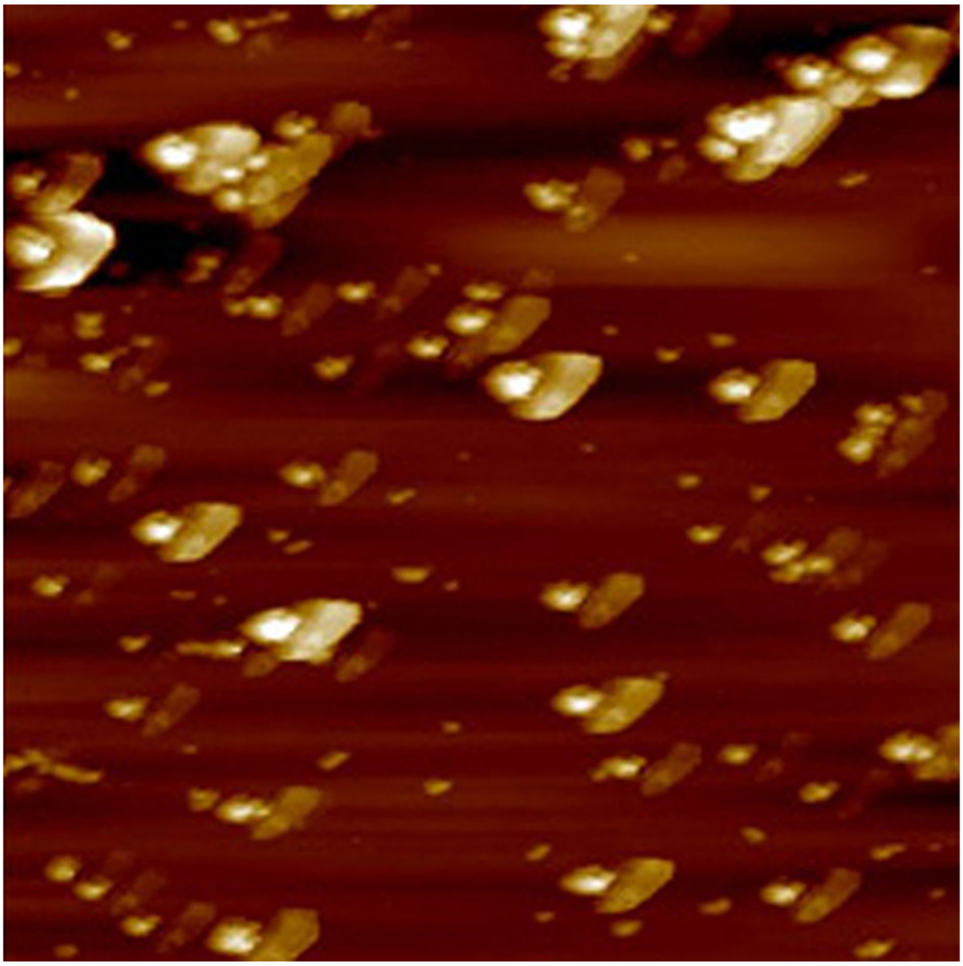
The tip may be worn down due to excessive imaging or using excessive force. A worn tip reduces overall image quality, and may introduce odd shapes to the image.



An AFM image showing oddly-shaped artifatcs due to a worn tip.

Tip contamination

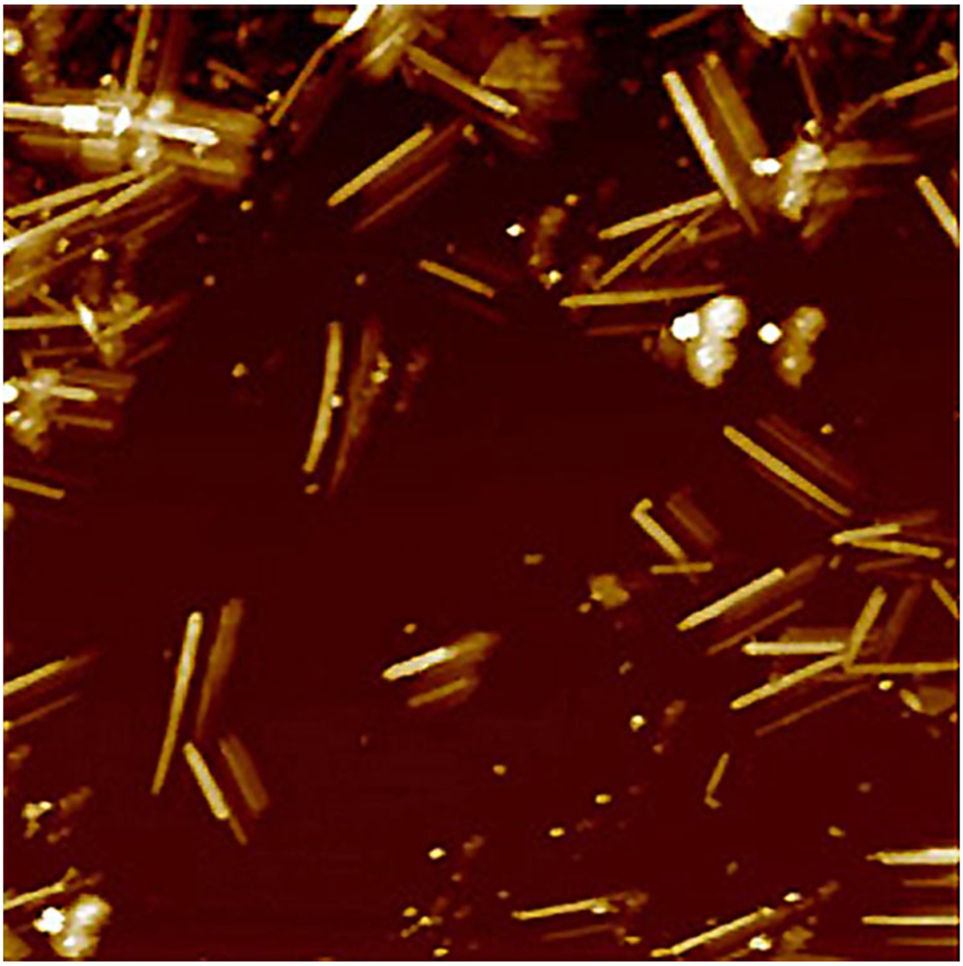
Tip contamination is due to the imaging tip 'picking up' material from the sample surface. This is typically more of a problem on soft biological tissues. Tip contamination can introduce strange, sometimes repeating, shapes to an image.



An AFM image showing repeating blob-shaped artefacts due to tip contamination.

Double tip

If the tip is damaged or picks up contamination, a tip with two or more sharp points may form. This will show on the image as a doubling of surface features.



A tip with a double point gives AFM images showing double images of surface features.

Debris on the surface is a good indicator of tip conditions since these are often spherical in shape - images of these, with a good tip, should therefore also be nearly spherical. Changing the scan direction should have no effect on the image artefact - to verify that these features are due to the tip, the user may physically rotate the sample relative to the tip and see if the artefacts have also rotated or not.

Scanner artefacts

Piezoelectric elements in scanners are used to position the probe tip relative to the sample surface at the nanometer scale with great accuracy. However, the behaviour of the piezoelectric elements does not always meet the theoretical ideal, that is, a linear increase in movement along with an increase/ decrease in applied voltage. The movement of these piezoelectric devices is subject to deviations from the ideal including non-linearity, hysteresis and creep.

Scanner sensitivity is the movement (nm/µm) per voltage (V).

Because of differences in the material properties and dimensions of each piezoelectric element, each scanner responds slightly differently to an applied voltage.

Nonlinearity

Nonlinearity means that the sensitivity of the scanner is not a linear function of the (amount of) applied voltage. Typically, sensitivity is greater at larger applied voltages than at near zero applied volts.

Nonlinearity of the x - y plane displays as surface features appearing stretched or contracted at the top of the image, and then appearing to correct nearer to the middle and bottom of the image. Some linear features may appear curved. This artefact will typically not reduce when the same region is scanned multiple times. Although obvious in images of linear structures like calibation grids, this artefact may go unnoticed in images of samples lacking evenly spaced features.

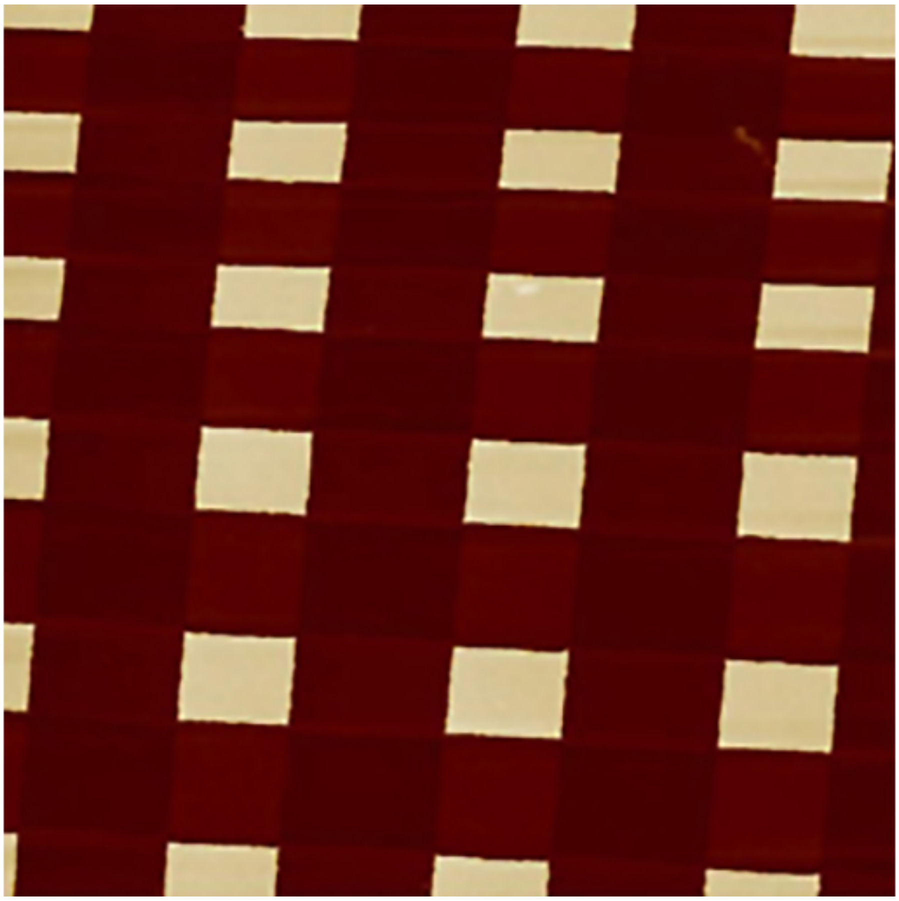


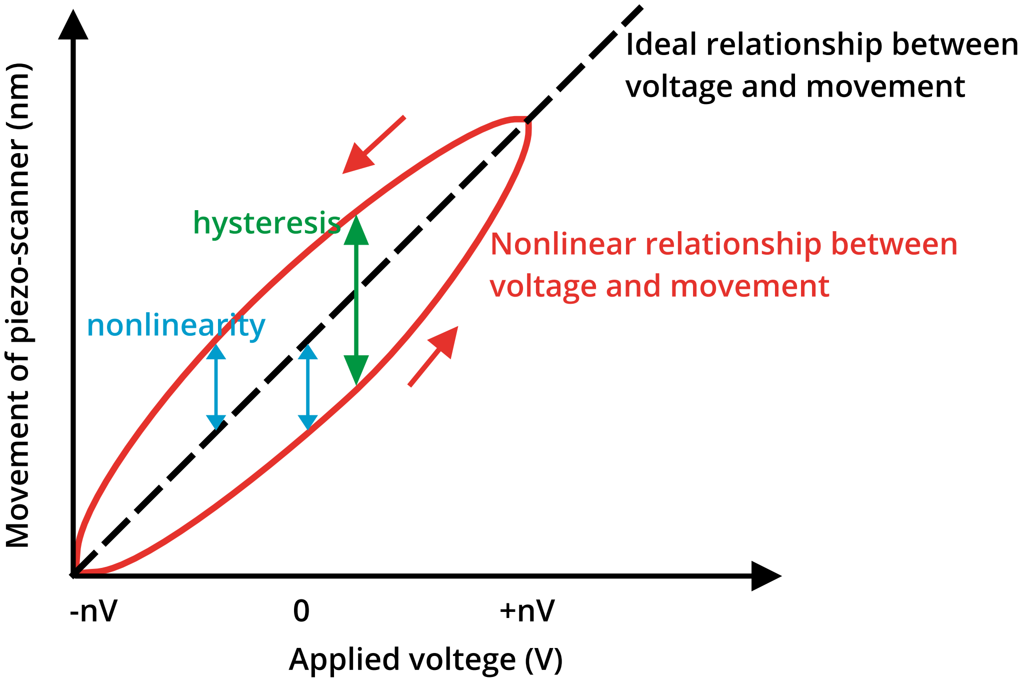
Image of a calibration grid showing non-linearity in the x - y plane.

Hysteresis

Hysteresis occurs in piezoelectric elements in the scanners when the response traces a different path depending on the direction of a voltage change. The magnitude of the effect depends on many variables including the starting voltage, the size of the voltage change, the rate of voltage change, and the scan angle.

In AFM, rastering across the sample surface (driven by voltage changes to the piezoelectric elements) means the scanner will move non-uniformly in the x - y plane if hysteresis is present. This non-uniformity has several effects, including differences in scanner sensitivity at the beginning and end of a scan. If the scanner moves further than it should vertically, in the z plane, tall features will appear to have a very 'sharp' edge in the image.

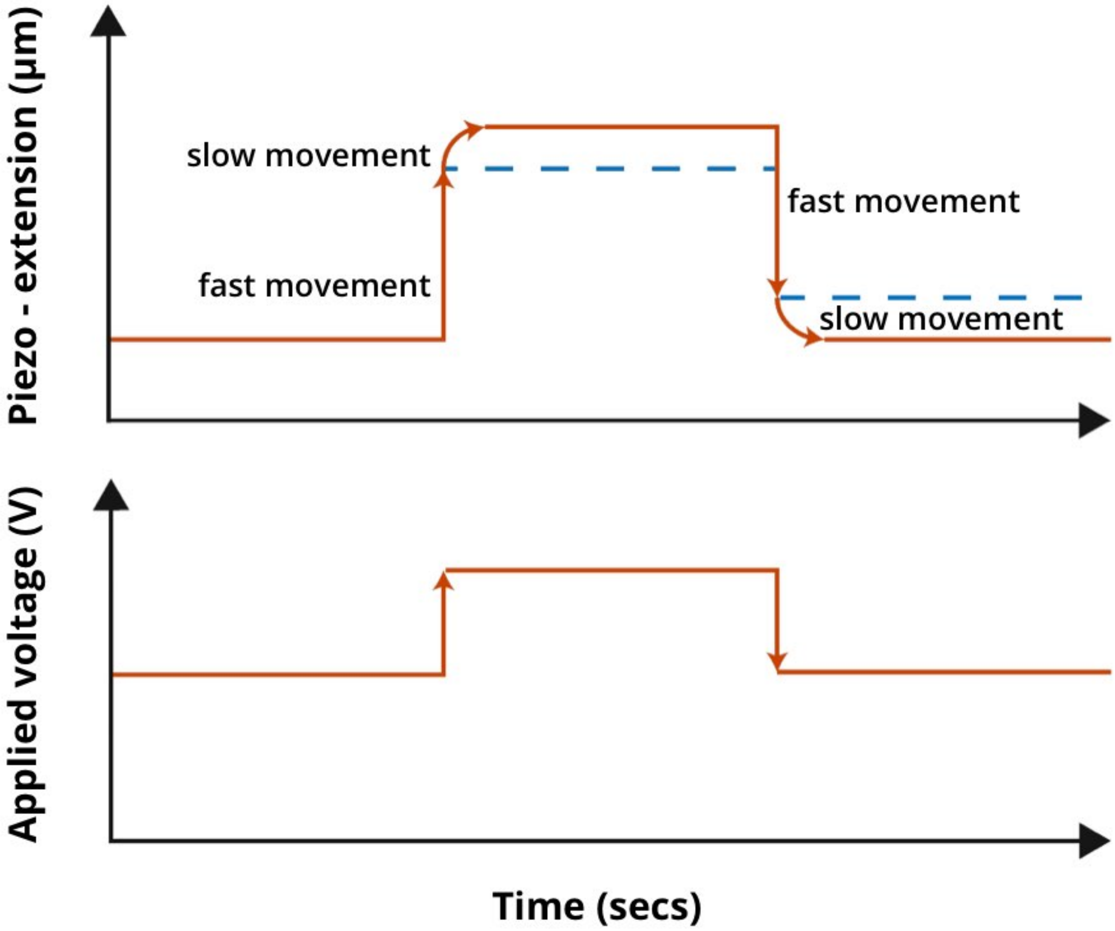
Hysteresis typically occurs when changing the scan size or the centre offset of the image, and typically only affects the first 10 - 20 lines of an image. Hysteresis can be reduced or eliminated by re-starting the scan, and/or scanning more slowly.



The dashed line represents an ideal linear relationship between applied voltage and the movement (extension) of the piezoelectric elements in a scanner. The red lines represent the actual nonlinear relationship that usually exists in piezo-scanners. This non-ideal relationship leads to the scanner artefacts of nonlinearity where scanner sensitivity depends on the size of the voltage, and hysteresis where scanner sensitivity depends on the direction of voltage change.

Scanner creep

Creep describes the continued motion of the scanner after a rapid change in voltage, for example, when moving the scanning position. The piezo scanner does not respond all at once; it moves the majority of the distance quickly (< 1 ms), then the last part of the movement is slower. The slow movement is called creep. If large voltage changes occur during scanning, then the slower movement causes a distortion of the image. Creep may result in surface features appearing smaller or longer in one direction than they actually are. Creep accounts for the initial lateral distortion apparent after zooming or moving to a new scan area, and which settles out after several scan lines have been traced.

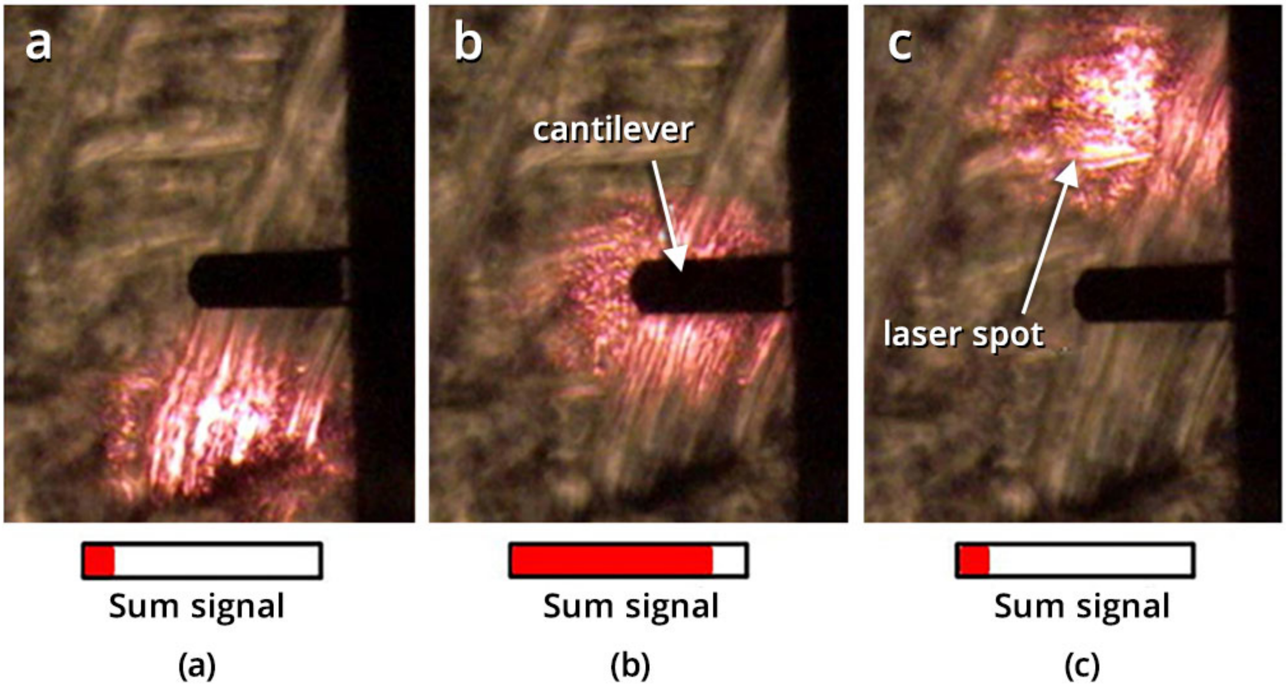


The continued slow movement of the piezoelectric scanner after a rapid change in applied voltage results in creep. Creep is measured as the ratio of the slow dimensional change to the fast dimensional change.

To correct for scanner artefacts, some AFM machines employ a sensor to detect actual movement of the scanner and compensation adjustments are made to the scanner drive signal in real time; this is called a 'closed loop' AFM.

AFM in practice - Laser alignment - Laser alignment on cantilever

Aligning the laser onto the cantilever is one of the most critical parts of the AFM set-up. The laser alignment will help determine the signal to noise ratio, the sensitivity of the cantilever deflection or amplitude and ultimately the quality of any data acquired using the AFM. If not performed properly then a number of artefacts can result including, for example, excessive force being applied to the surface when scanning and optical interference fringes from reflective surfaces can be detected. Typically, adjustment screws are used to move the laser in the X and Y directions and onto the back of the AFM cantilever. The back of AFM cantilevers are often metal coated (gold or aluminium) in order to improve reflectivity. Determining when the laser is aligned on the lever is done either using an optical microscope to visualise the laser spot and cantilever or the laser spot is observed on a piece of white paper and the operator looks for diffraction patterns on the paper when the laser hits the back of the cantilever. As the laser is moved on and off the cantilever the sum signal from the photodiode is also monitored. When the laser signal is maximised and positioned as close to the end of the cantilever as possible, without loss of sum signal, then the cantilever can be considered to be laser aligned. The figure_below shows optical images of the laser alignment procedure. In a) the laser is off the cantilever and the sum laser signal detected by the photodiode is low. b) shows the laser spot located on the back of the cantilever and the sum signal is now high and the cantilever is in the correct position for operation. In c) the laser is moved off the cantilever and the sum signal is now low again.



Optical images of the laser alignment procedure. (a) Laser is off the cantilever and the sum laser signal detected by the photodiode is low. (b) The laser spot located on the back of the cantilever and the sum signal is now high and the cantilever is in the correct position for operation. (c) The laser is moved off the cantilever and the sum signal is now low again.

Cantilever tuning

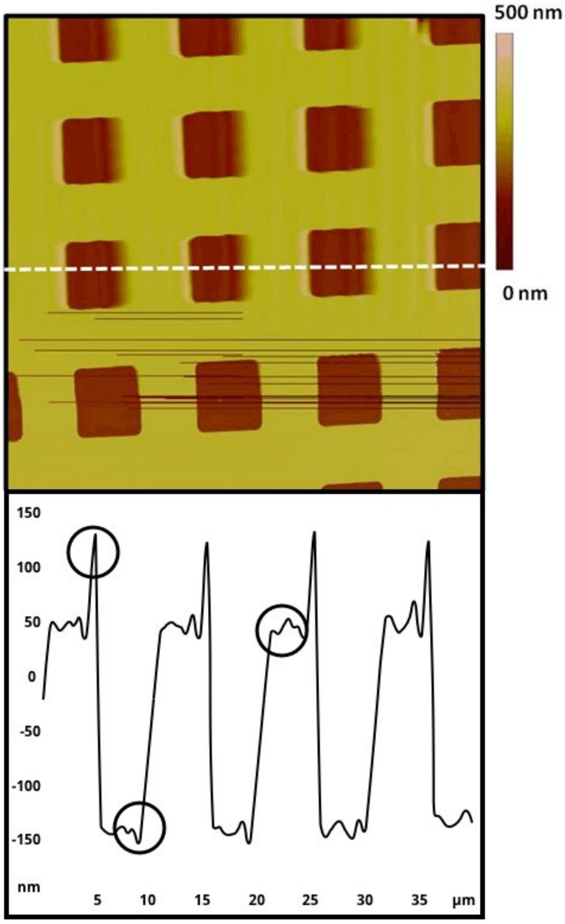
Only for tapping and non-contact modes

Once the laser is aligned onto the cantilever and the photodiode is centred, then the cantilever can be tuned. This means that the resonant frequency of the cantilever can be determined. This is almost always an automated process in the AFM software, and all the operator has to input is the frequency range to scan over and the target amplitude of the cantilever. In most tapping mode tip holders there is a small piezo-ceramic element that is located where the AFM chip is held securely in the tip holder. This piezo can be driven by a signal and made to vibrate. This then vibrates the chip which then vibrates the cantilever. The resonant frequency of the cantilever can then be found by scanning through a range of frequencies and monitoring the amplitude of oscillation of the cantilever on the photodiode. When the peak with the highest amplitude is detected within the frequency range scanned then the AFM computer will assume this is the resonant peak for the cantilever. It is therefore important to scan though the correct frequency range as harmonics may be detected if the incorrect frequency range is used.

Manufacturers for AFM probes will supply them with nominal resonant frequencies or a range of frequencies which are a good guide to use. A number of parameters are able to be monitored and/or adjusted but the main ones are start frequency, end frequency and target amplitude.

Image quality optimisation - Scan rate

This parameter controls the speed at which the tip moves over the sample surface.
Scanning too fast will mean the tip will not track or trace the surface effectively and result in scan artefacts such as those depicted in the figure_below. The image is of a calibration grid with square holes with a distance of 10 µm between the edges and a depth of 180 nm. At the top is the height image while the bottom is a representative cross section of the surface.



Height image of a calibration grid with the scan rate set too high. The area scanned is 40 x 40 µm. At the top is a cross section, or trace, acquired where the dotted line is located. The circled sections on the bottom are regions on the trace that are distorted due to scan rate.

Scanning too slow and causing an increase in temperature can also be a problem as thermal drift can distort the shape of features on the surface.

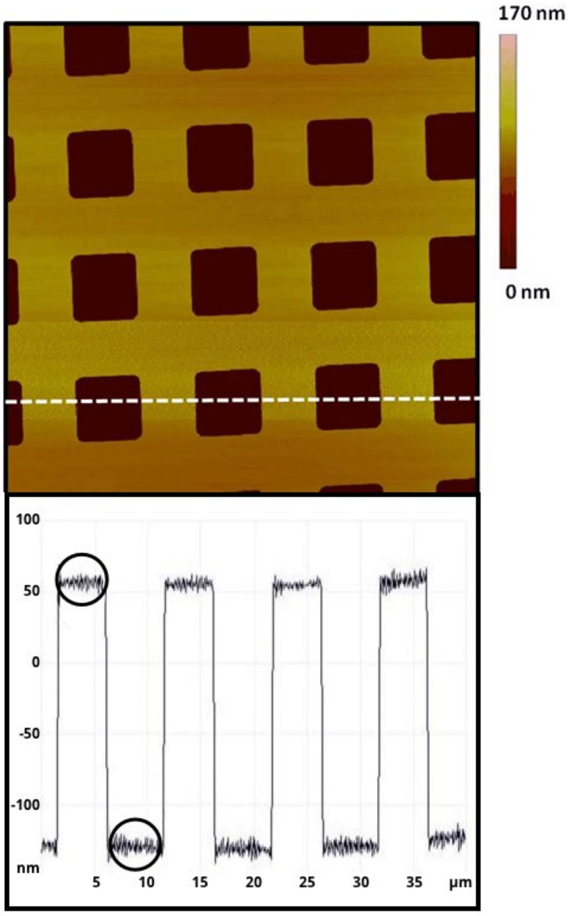
Typical scan rates for most samples is between 0.5 to 2 Hz. The scan rate is closely connected to the tip velocity by the size of the area scanned. For scan areas larger than 10 µm the scan rate will typically be < 1 Hz, but for areas smaller than 10 µm it may be possible to scan faster than 1 Hz and still produce good quality images. This can again be system dependant as some AFMs will be able to scan on average faster than others and still produce images of the same quality.

Gains

There are two main feedback gains.
The first and easily the most sensitive is the **Integral gain**. Increasing this value increases the amount of the input signal (from the photodiode) which is fed back into the output signal (the z piezo). The higher the values, the faster the AFM will react to changes in the topography in the sample. Thus, the higher you can have the integral gain, the better image quality can be achieved. The problem is that if the integral gain is set too high, feedback oscillations will result (see figure). The aim is to set them as high as possible while avoiding oscillations in the image.

The **Proportional gain** is not nearly as sensitive as the integral gain and a general rule is that once the integral gain is optimised then adjust the proportional gain to the same value.

It is difficult to give exact gain values since each AFM system will have different scaling factors for these parameters, but another general rule is that for relatively rough samples the gains should be higher than for smooth samples.

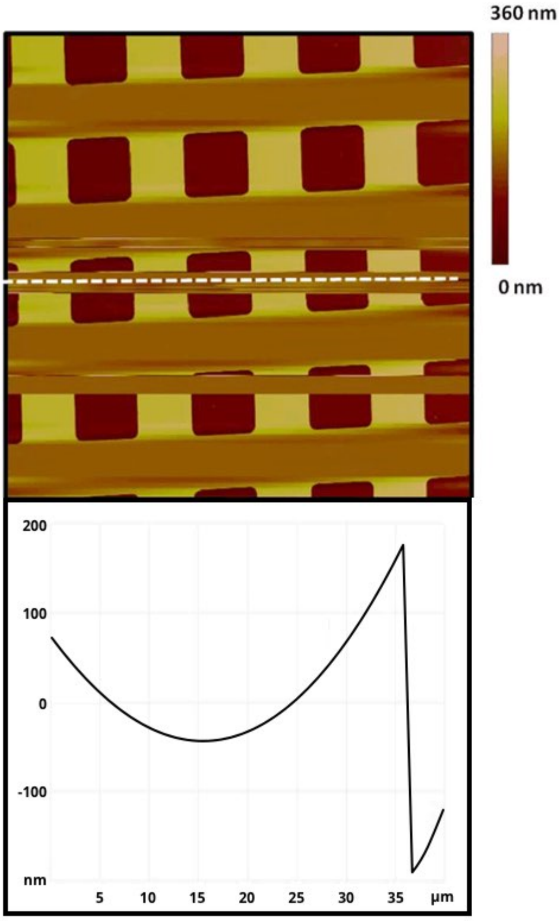


Height image of calibration grid with the integral gain set too high. The area scanned is 40 x 40 µm. The diagram at the bottom is a trace acquired where the dotted line is located on the image at the top. The circled sections on the diagram are regions on the trace that are distorted and noisy due to the integral gain set too high.

Set-point - Contact mode

In contact mode the set-point refers to the deflection set-point of the cantilever, and is the cantilever deflection (usually given in Volts or nanoAmps) at which the AFM will scan, and is determined by the operator. Since the force the cantilever exerts on the sample is directly proportional to the deflection of the cantilever via Hooke's Law, it is possible for the operator to control the amount of force being applied to the sample surface while imaging.

Increasing the set-point will result in increasing the imaging force while reducing the set-point will result in reduced imaging forces. This can be very important for a number of reasons. If the set-point is too low, then the tip may not be able to track the surface properly and could come out of feedback if not enough force is applied. This is demonstrated in the figure_below. However, if too much force is applied to the surface then the tip could damage the sample surface, or in time damage to the tip itself can result in image artefacts e.g. double tipping.



Height image of a calibration grid with the deflection set-point too low. The area scanned is 40 x 40 µm. The bottom is a trace acquired where the dotted line is located on the top. The trace does not represent the true surface topography.

When changing these parameters it is important to remember that they can all affect each other. Therefore it is good practice to optimise each one and then repeat. For example

1. Optimise set-point
2. Optimise gains
3. Optimise scan rate

Then repeat until maximum image quality is achieved.

Tapping mode

In this mode of operation, set-point refers to the amplitude of oscillation of the cantilever at which the tip taps the sample surface. In this case lowering the amplitude set-point will increase the tapping force exerted on the sample, and increasing the amplitude set-point will reduce the tapping force. Similar constraints apply to this parameter for tapping mode as for contact mode. If the set-point is too high, then the tapping force is reduced to such a level that the tip may not track the surface properly and move in and out of feedback. This will result in poor image tracking, similar to that displayed in figure_13. If the set-point is too low, and the tapping force is too high, then the tip may become damaged and artefacts will result. In general, the amplitude set-point will be 50 - 90 % of the cantilever free amplitude.

When changing these parameters it is important to remember that they can all affect each other. Therefore it is good practice to optimise each one and then repeat. For example

1. Optimise set-point

2. Optimise gains

3. Optimise scan rate

Then repeat until maximum image quality is achieved.

Feedback

The following a is general discussion of feedback in the context of collecting images in SPM. Feedback in SPM is an example of control. Other examples include thermostats or the cruise control in your car.

A process control loop regulates some dynamic (changing in time) variable in a process.

Basically the way this works is that we input some set point and then the “controller” compares the value to the set point and tries to adjust it back to the set point. There are lots of ways to make the comparisons and lots of ways to make the adjustment. Only adjust one parameter.

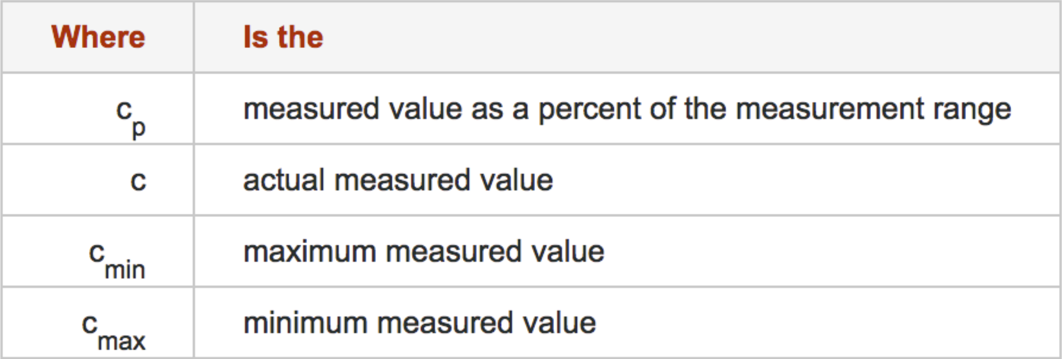
To measure the 'error' we can simply measure the actual value (b) and the compare it to the set point (r)

$$e = r - b$$

where e is the error.

More often the error is expressed as a percent of the possible measurement range (the span):

$$c_p = \frac{c - c_{min}}{c_{max} - c_{min}} \times 100$$



Where	Is the
c_p	measured value as a percent of the measurement range
c	actual measured value
c_{min}	maximum measured value
c_{max}	minimum measured value

SO to get the error expressed as a percent of the possible measurement range (the span):

$$e_p = \frac{r - b}{b_{max} - b_{min}} \times 100$$

Where	Is the
e_p	error as a percent of the span
r	set point
b	actual measured value
b_{max}	maximum measured value
b_{min}	minimum measured value

OR: More often the error is expressed as a percent of the possible control range which might be less than the full measurement range (the span):

$$p = \frac{u - u_{min}}{u_{max} - u_{min}} \times 100$$

Where	Is the
p	controller output as % of full scale
u	value of the output
u_{min}	maximum value of controlling parameter
u_{max}	minimum value of controlling parameter

Control signal - Control modes

Reverse and Direct Action:

- Direct: Increase the control signal when the variable increases.
- Reverse: Decrease the control signal when the variable increases (heat).

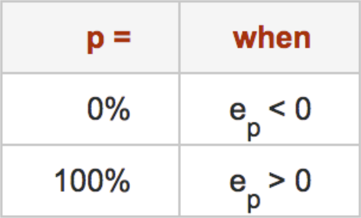
Two Common Modes:

- Discontinuous
- Continuous. In this mode the output of the controller changes smoothly in response to the error or rate of change of the error. The most common controllers these days are combinations of the continuous controller modes.

Discontinuous

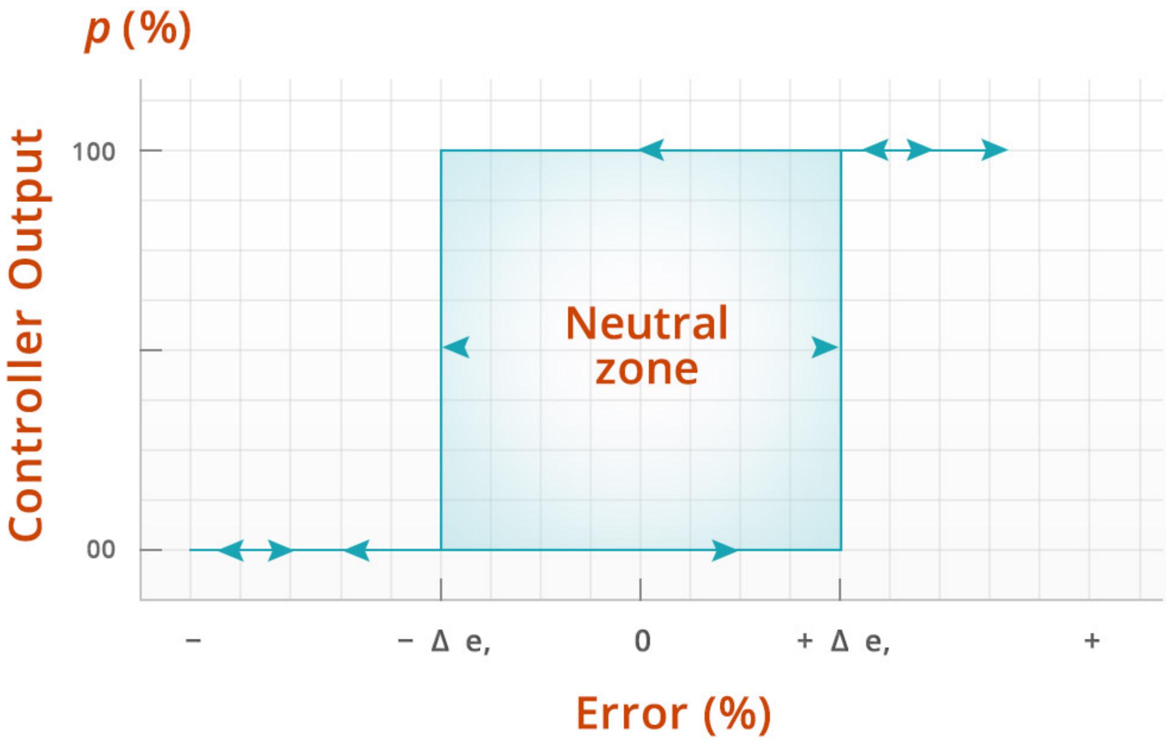
Two Position Mode

It is basically ON/OFF

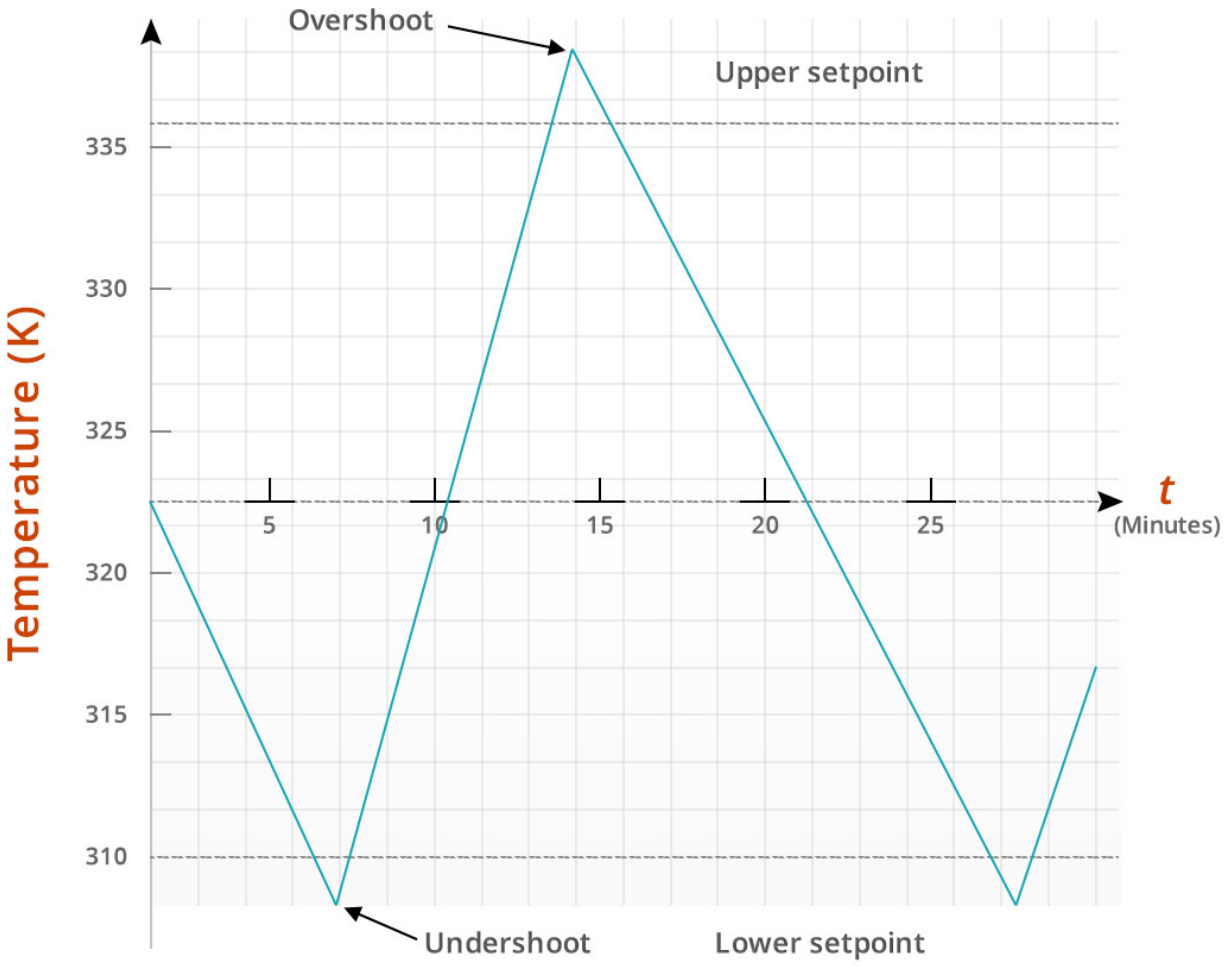


p =	when
0%	$e_p < 0$
100%	$e_p > 0$

There is often a neutral zone where no change to control output is made:



As an example: for a hot water tank; T (temperature) drops by 2 degrees per minute when OFF, heats up at 4 degrees per minute when ON, with a control lag of 0.5 min and a neutral zone between the upper and lower set points which are equal to +/- 4% of the set point of 323 K.

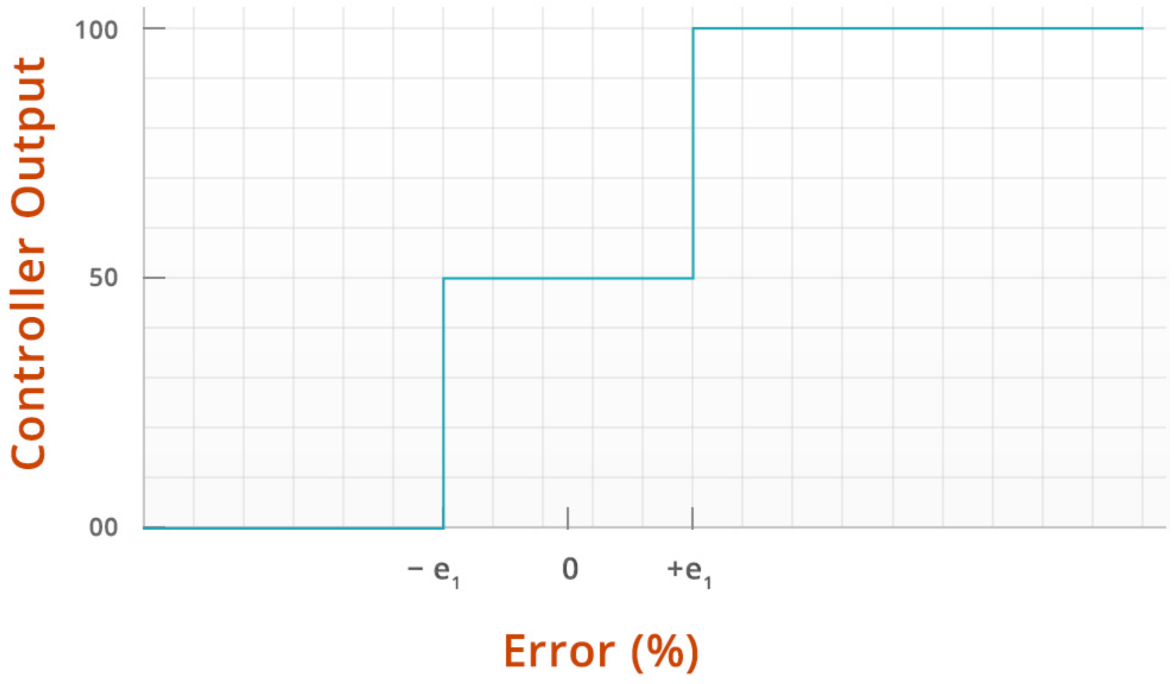


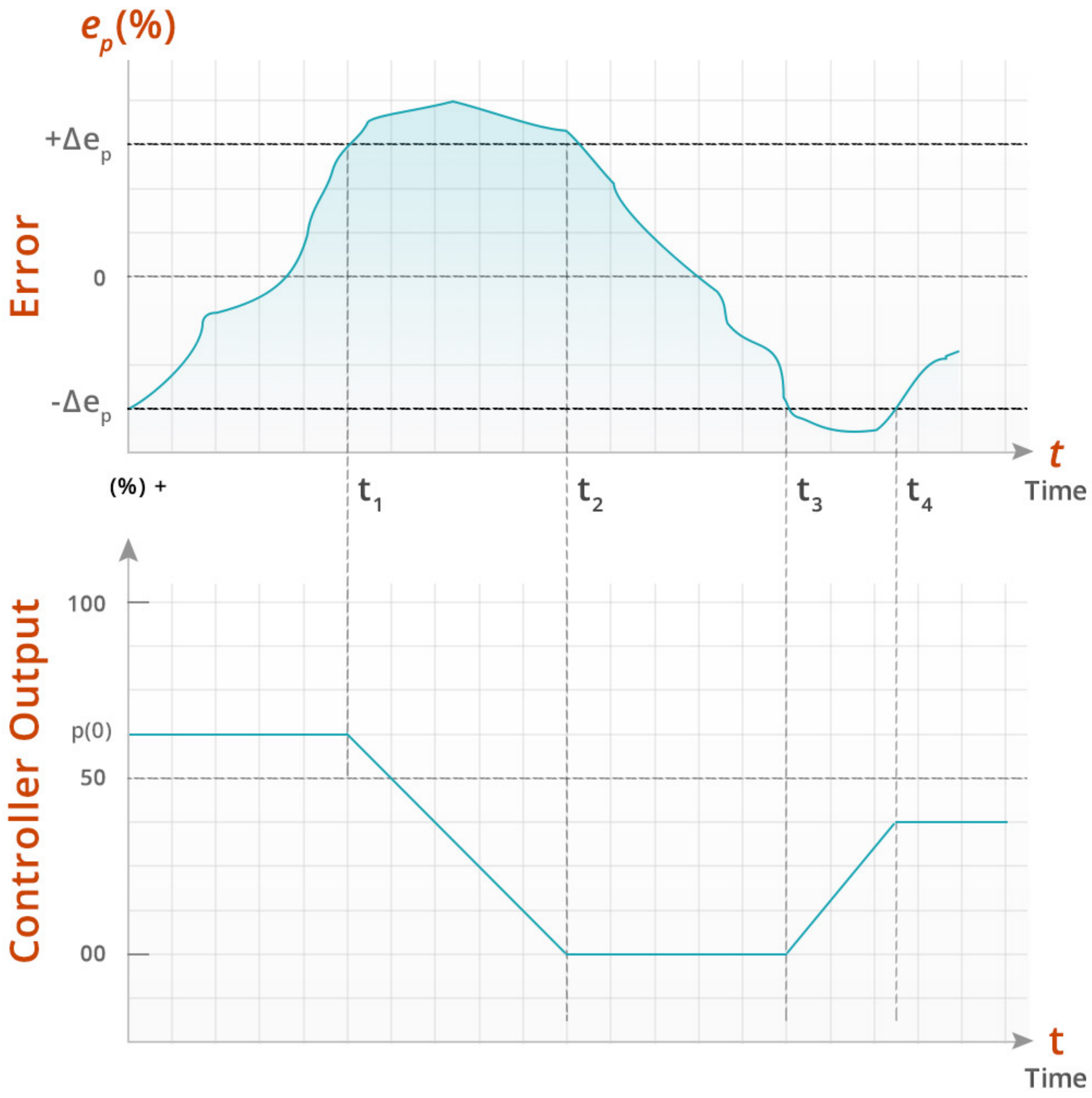
Multiposition Position Mode

$$p = p_i \; e_p > i = 1,2, \; , n$$

For three positions:

p =	when
0%	$e_p < -e_1$
50%	$-e_1 < e_p < e_1$
100%	$e_p > e_1$


Controller Output
100
50
00
− e_1
0
+e_1
Error (%)



Floating control

In this case, output of the controller is determined both by the error and an input which changes the output at a defined rate

$$\frac{dp}{dt} = \pm K_F \qquad \left|e_p\right| > \Delta e_p$$

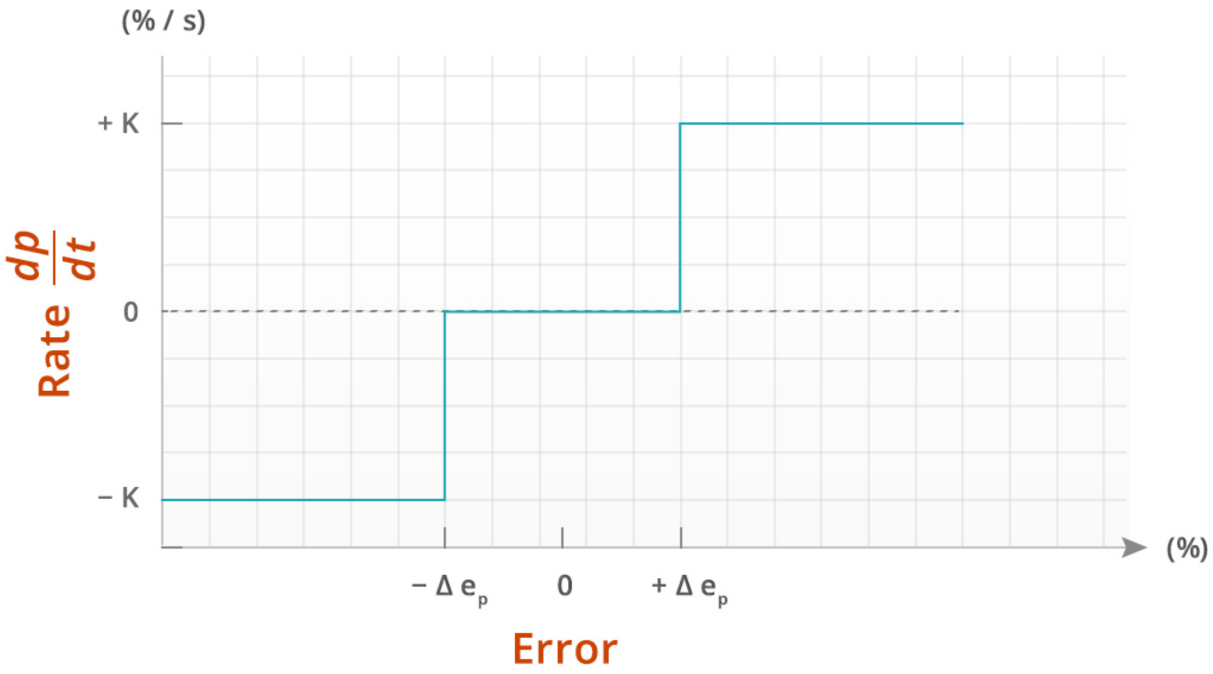
Where

Rate of change of the controller output with	Time
K_F	rate constant (% s^{-1})
De_p	half the neutral zone

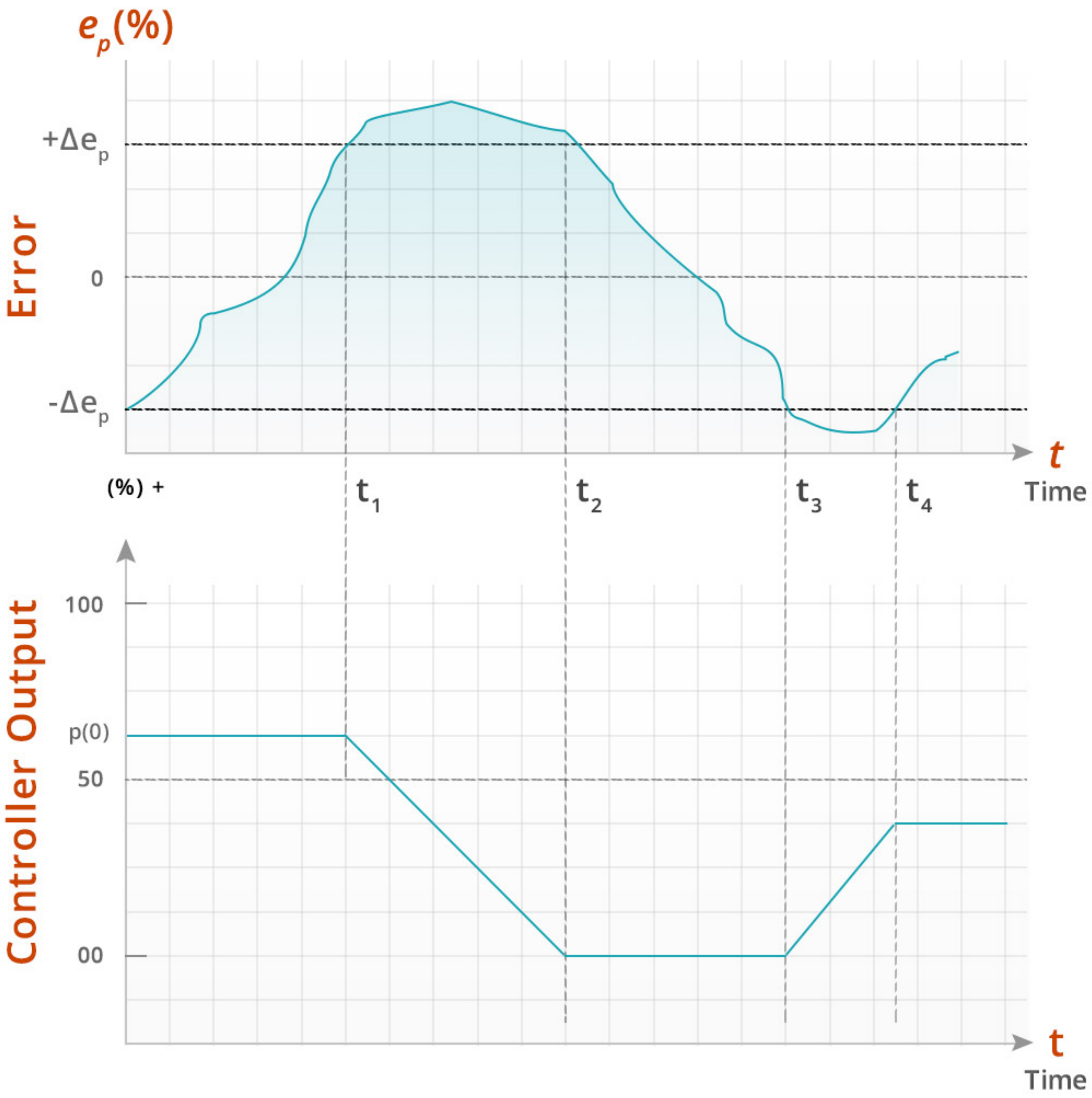
So

$$p = \pm K_F t + p(0) \qquad \left|e_p\right| > \Delta e_p$$

where p(0) is the controller output at t = 0.



(a)

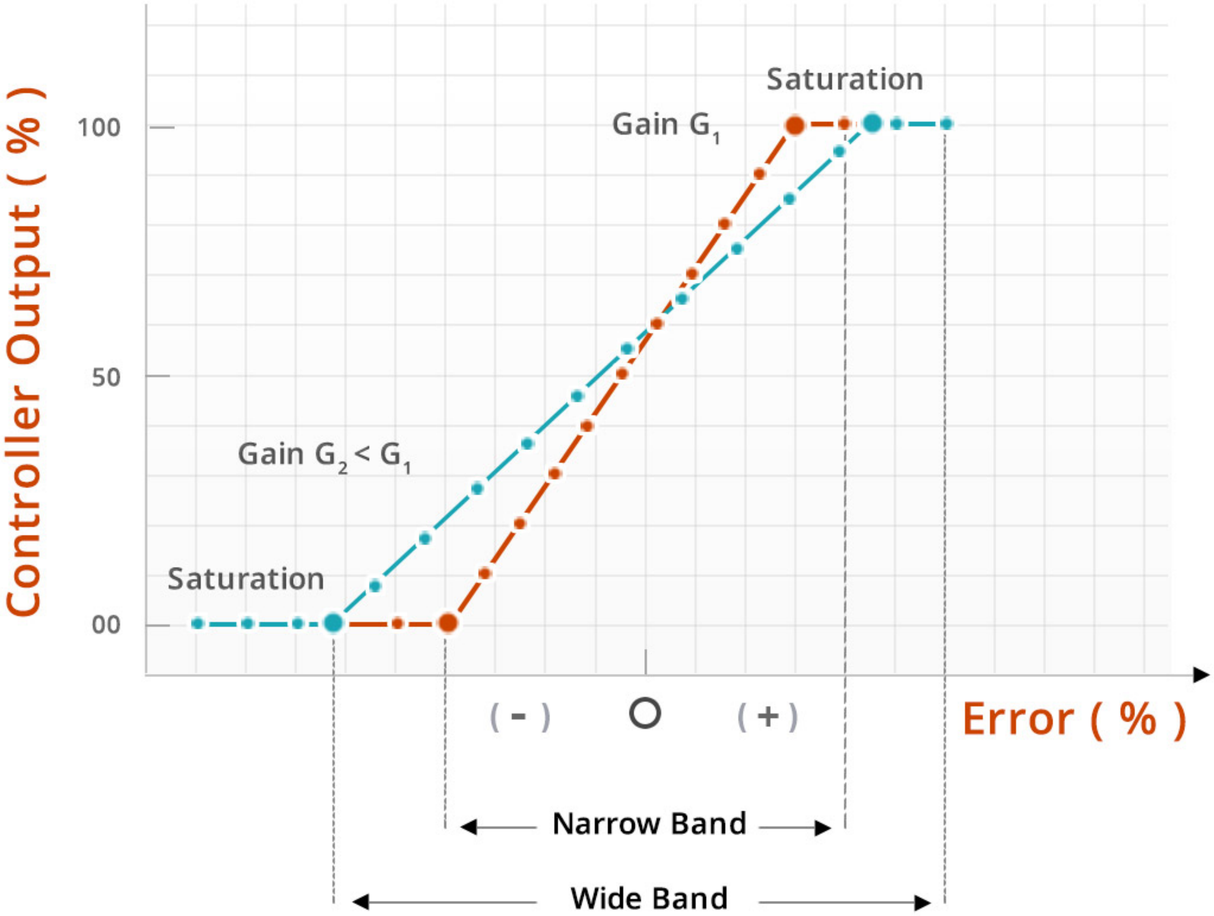

e_p(%)
Error
$+\Delta e_p$
0
$-\Delta e_p$
t
Time
(%) +
t_1
t_2
t_3
t_4
Controller Output
100
p(0)
50
00
t
Time

Continuous - Proportional control (P)

In this mode a smooth, linear relationship exists between the controller output and the error. In an equation:

$$p = K_p e_p + p_0$$

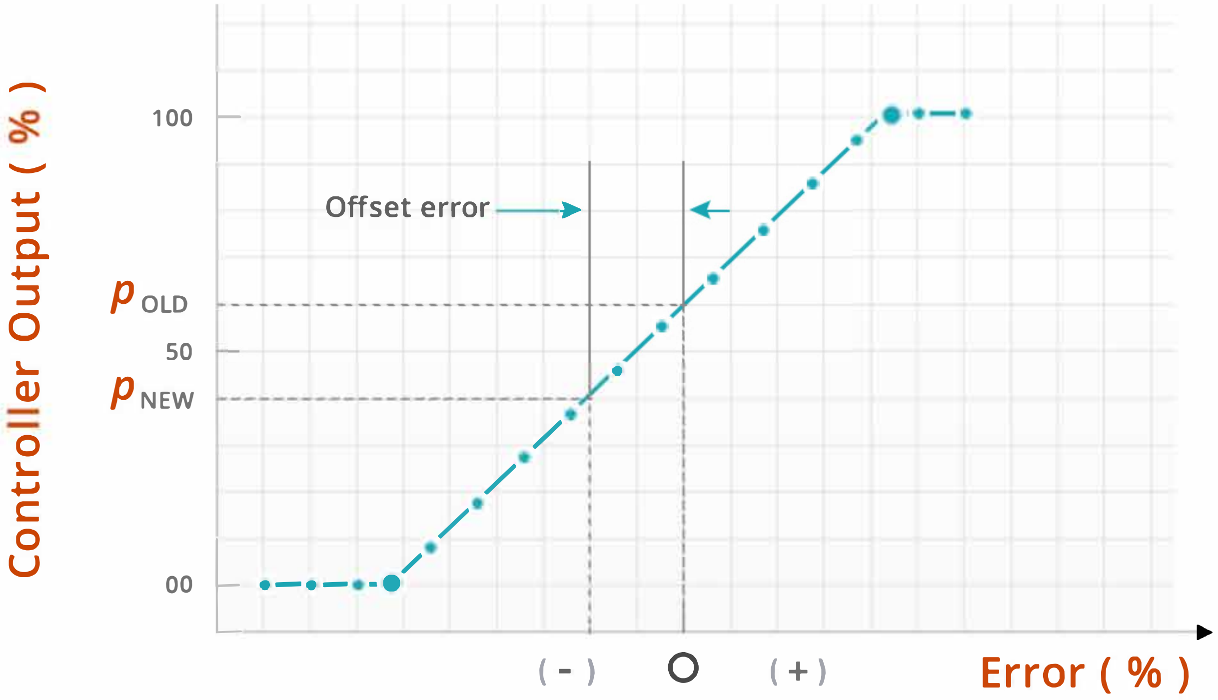
Where	Is the
K_p	proportional gain between error and controller output (% per %)
p_0	controller output with no error (%)



Proportion Band – only works for certain errors depending on the gain used..

Offset

If the conditions change, then the system can never get back to zero error conditions.

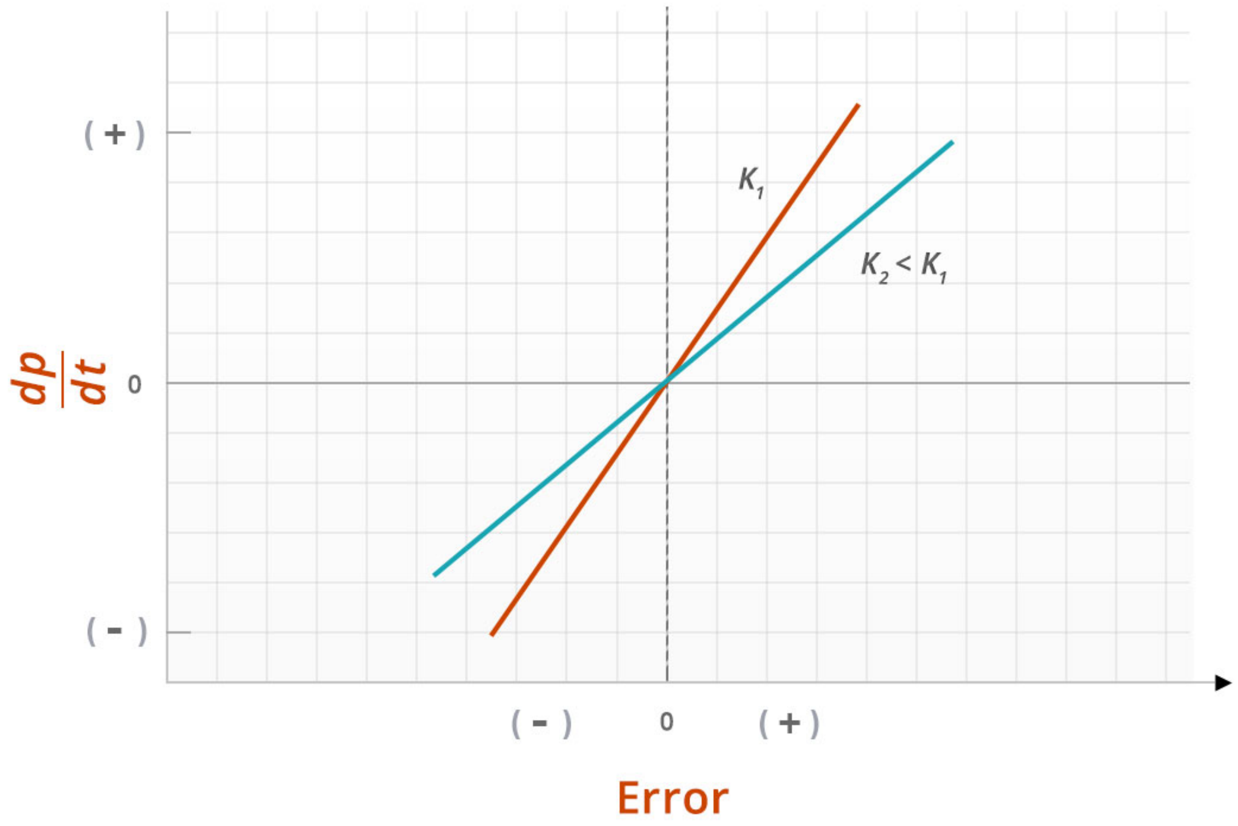


Integral control

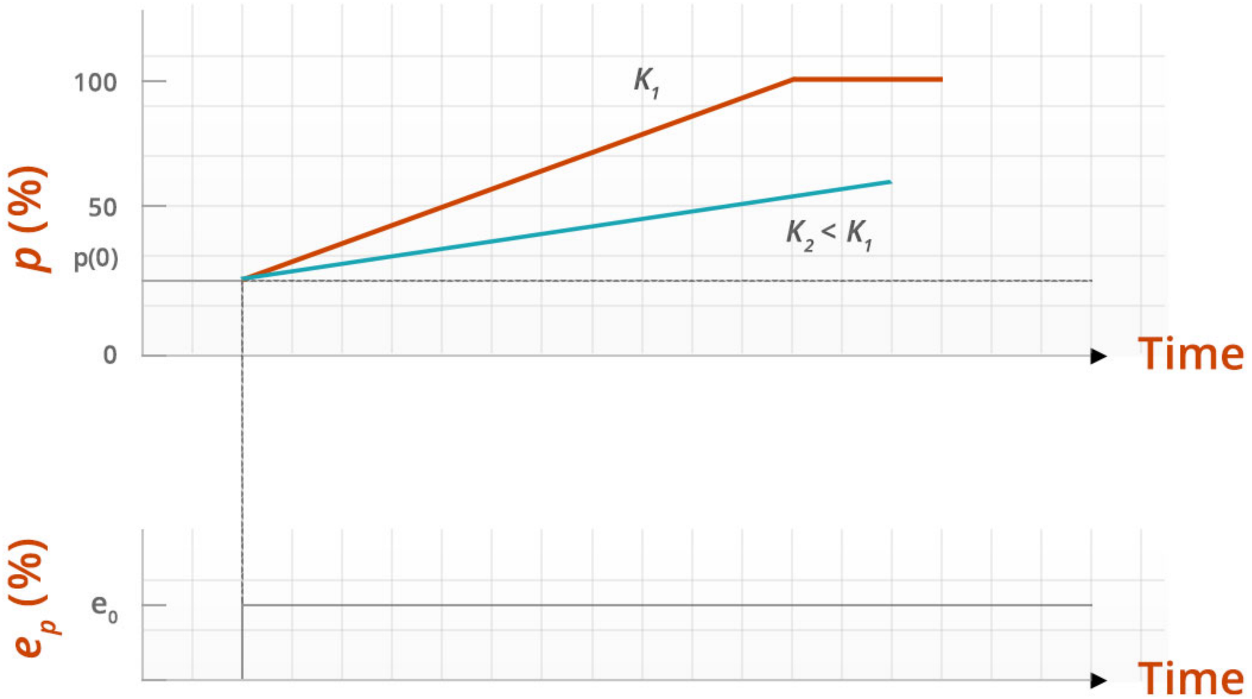
The offset problem can be important especially in very dynamic systems. To overcome this, the error is summed over time (integrated) and that sum is multiplied by a gain to be added to the present controller output:

$$p(t) = K_I \int_0^t e_p dt + p(0)$$

Where	Is the
$p(0)$	controller output when integration starts
K_I	gain



(a)



(b)

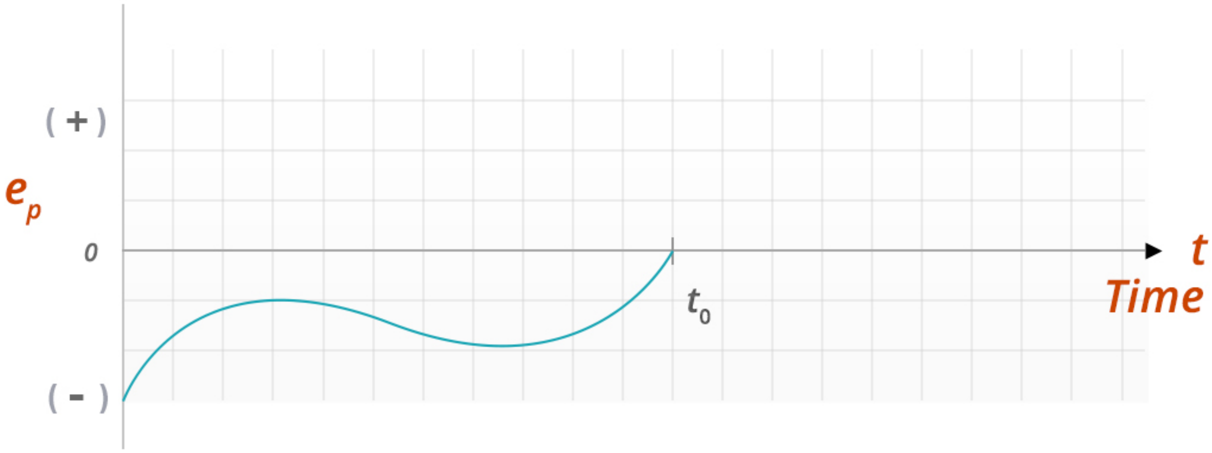
The main problem with this type of control is the response time. It obviously takes time to to do the adding and then change the control. The situation may have already changed. This is basically a dog chasing its tail problem but can lead to the control system "oscillating" and never getting back to zero error.

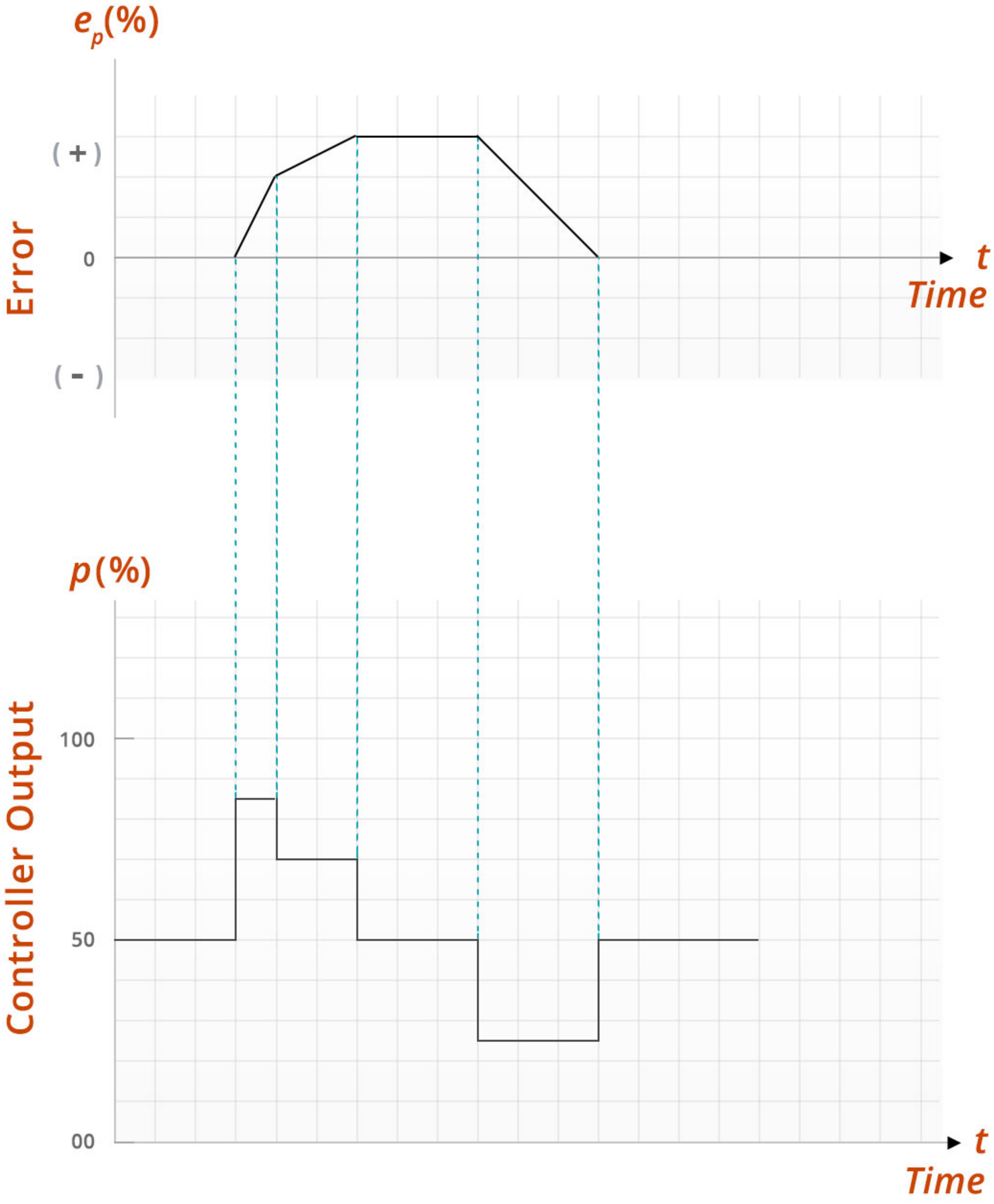
Derivative control

Derivation controller action responds to the rate at which the error is changing--its derivative. Can't be used alone because it does not actually measure the error (only its change)

$$p(t) = K_D \frac{de_p}{dt}$$

K_D is the gain

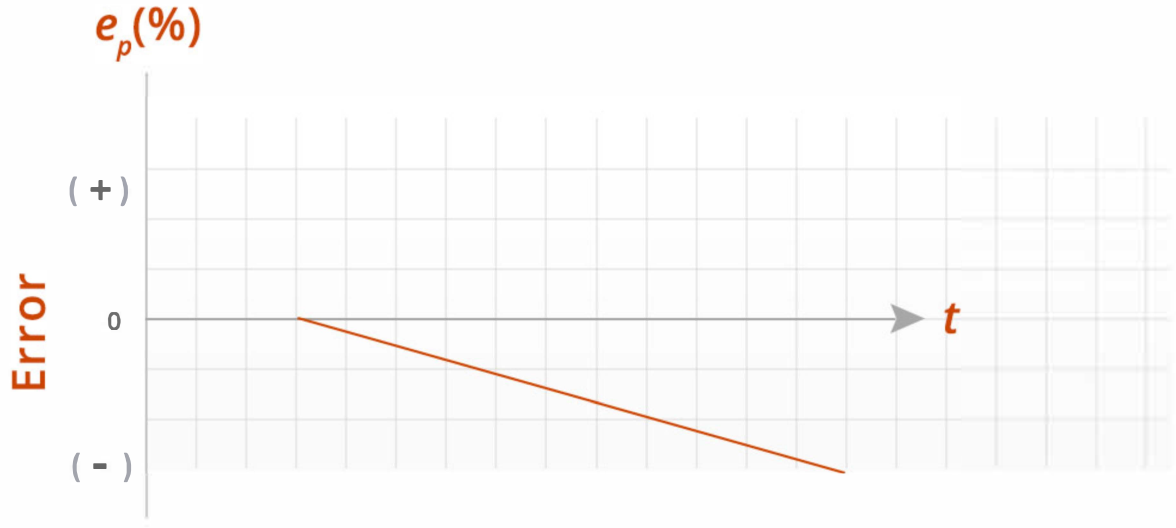


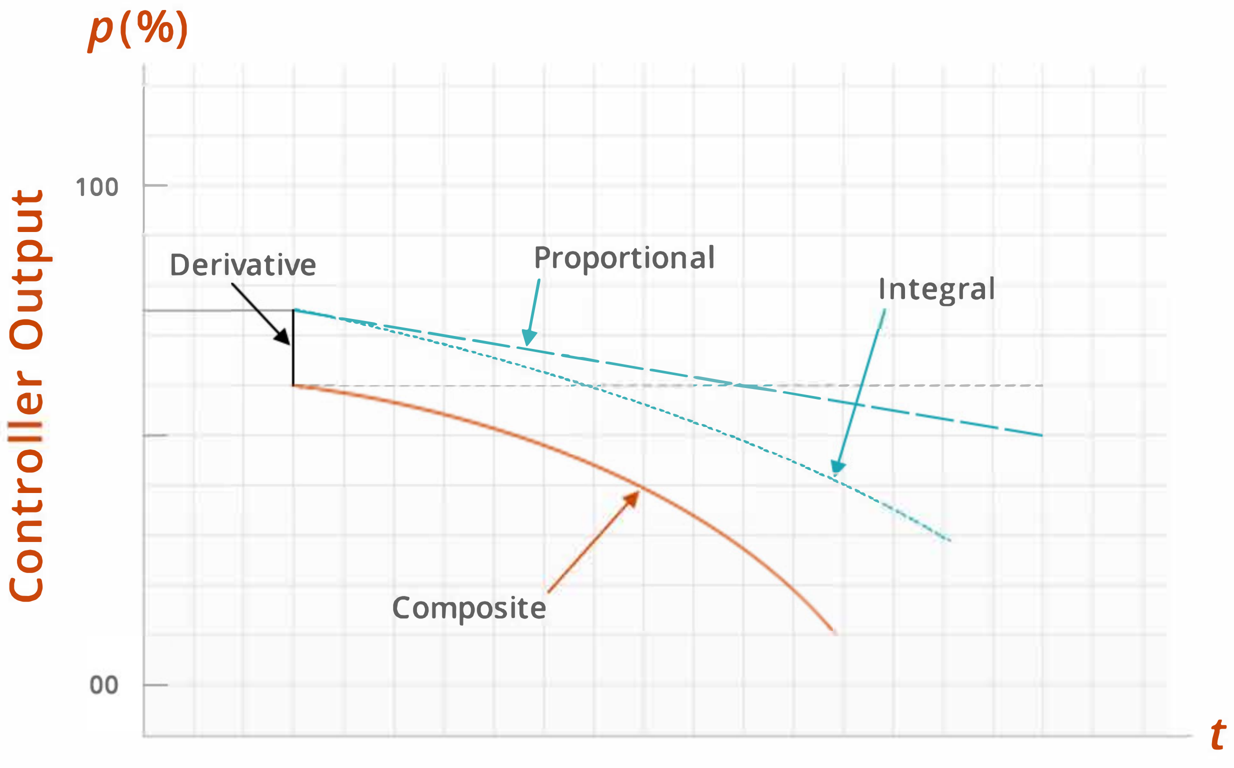

e_p(%)
(+)
0
(-)
Error
t
Time
p (%)
100
50
00
Controller Output
t
Time

Composite control (PID)

All the control we have talked about can be used in combination in various applications but we will just discuss one. PID is the most complex but also has the widest application:

$$p(t) = K_P e_p + K_P K_I \int_0^t e_p \, dt + K_P K_D \frac{de_p}{dt} + p_I(0)$$





Other Techniques - Scanning Tunneling Microscopy (STM)

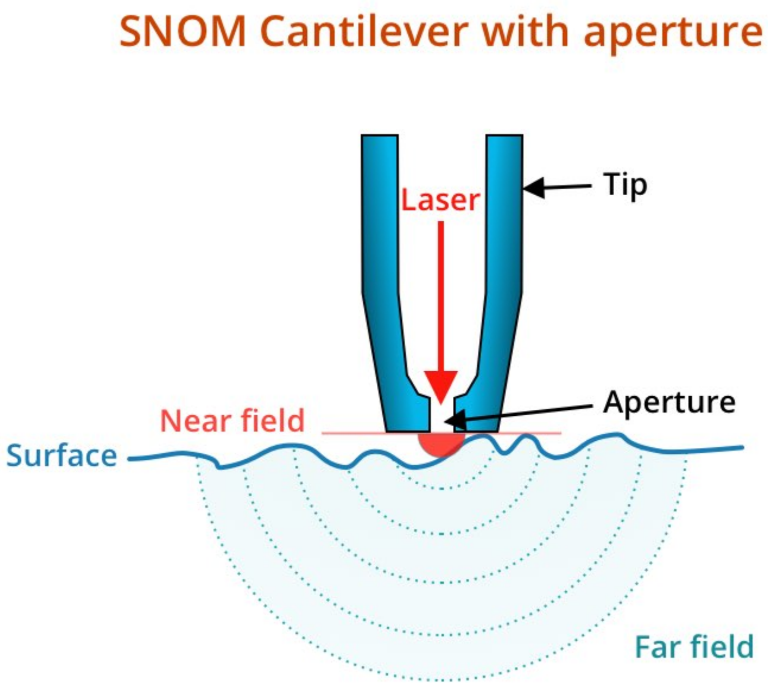
STM is the oldest of the scanning probe microscopes and involves bringing an atomically sharp tip within a couple of nanometres of the sample. When a bias is applied between the tip and sample, electrons will tunnel from the one to the other and this current can be used to measure the topography of the sample. The unique aspect of STM is that is can readily be used to acquire images with atomic resolution which is not generally true for the other SPMs. However, because a current is being measured only conducting samples can be examined and hence this limits the range of possible samples.

Scanning Tunnelling Microscopy (STM) relies on “tunnelling current” between the probe and the sample to sense the topography of the sample. The STM probe, a sharp metal tip (in the best case, atomically sharp), is positioned a few atomic diameters above a conducting sample which is electrically biased with respect to the tip. At a distance under 1 nanometre, a tunnelling current will flow from sample to tip. In operation, the bias voltages typically range from 10 to 1000 mV while the tunnelling currents vary from 0.2 to 10 nA. The tunnelling current changes exponentially with the tip-sample separation, typically decreasing by a factor of two as the separation is increased 0.2 nm. The exponential relationship between the tip separation and the tunnelling current makes the tunnelling current an excellent parameter for sensing the tip-to-sample separation. In essence, a reproduction of the sample surface is produced by scanning the tip over the sample surface and sensing the tunnelling current. The first STM operated in ultrahigh vacuum on cryogenically cooled samples. In the years following its invention in 1981, many variations on the STM theme appeared. P.K. Hansma and J. Tersoff wrote a good review article on the subject, containing experiments, theory, and over 100 references for The Journal of Applied Physics, 61 pp. R1-23, 1987.

STM relies on a precise scanning technique to produce very high-resolution, three dimensional images of sample surfaces. The STM scans the sample surface beneath the tip in a raster pattern while sensing and outputting the tunnelling current to the computer control station. The digital signal processor (DSP) in the workstation controls the Z position of the piezo based on the tunnelling current error signal. The STM can operate in both constant height and constant current modes. The DSP always adjusts the height of the tip based on the tunnelling current signal, but if the feedback gains are low the piezo remains at a nearly constant height while tunnelling current data is collected. This is constant height mode. With the gains high, the piezo height changes to keep the tunnelling current nearly constant, and changes in piezo height are used to construct the image. This is constant current mode. The exponential relationship between tip-sample separation and tunnelling current allows the tip height to be controlled very precisely. For example, if the tunnelling current stays within 20 percent of the set-point value (the current to be maintained by the feedback system), the variation in the tip-sample separation is less than 0.02 nm.

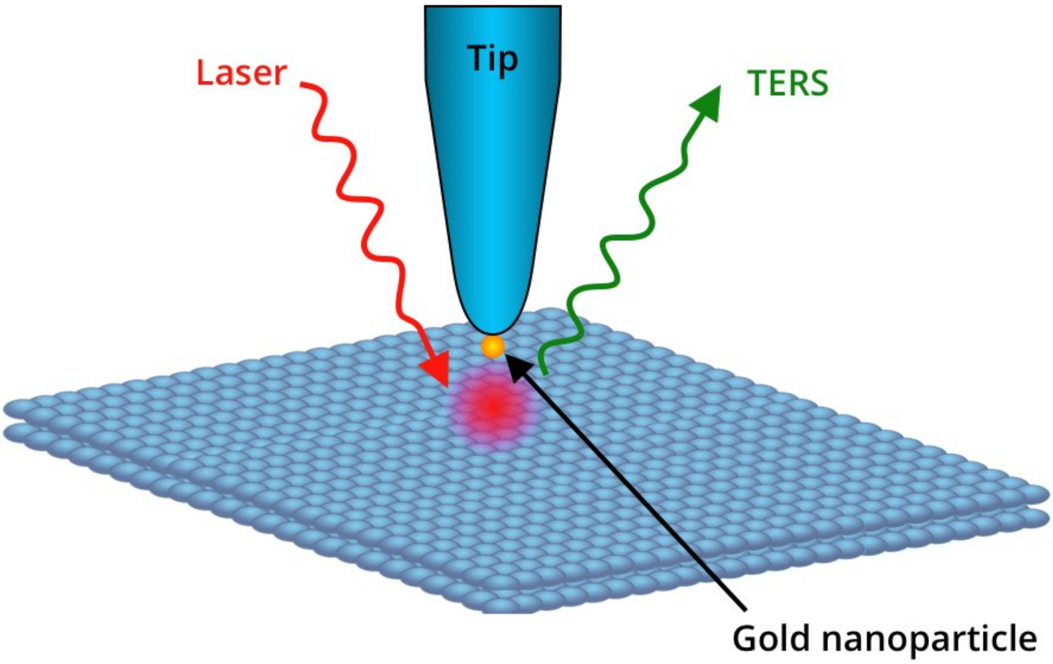
Near-field scanning optical microscope (NSOM/SNOM)

In this case, the standard AFM cantilver is replaced by something that can act as a waveguide for light (typically an optical fibre). Light is passed through the waveguide onto the sample and resulting light is detected via various means or alternatively the sample is optically excited and the resulting light is detected using the waveguide. In either approach, the aperture at the bottom of the waveguide is less than the diffraction limit, and hence this approach provides a method to get around the resolution limits of standard optical microscopy.



Tip enhanced Raman spectroscopy (TERS)

Raman spectroscopy is a light scattering technique where the specimen is excited by a laser. Some of this laser light scatters with a wavelength determined by the nature of the chemical bonds within the specimen. This is called Raman scattering, and provides information about composition, phase, crystallinity and molecular interactions. Most Raman scattering experiments use a laser with quite a large size, several micrometers at least, and are therefore only capable of coarse mapping of the specimen. In Tip enhanced Raman, the AFM tip is used to excite a very small volume, and in conjunction with laser illumination, a Raman spectrum can be obtained from an extremely small area. The AFM tip is typically metal coated to produce a roughened surface which significantly enhances the Raman signal at the tip. This allows high resolution (less than 50 nm) mapping of the chemical nature of a specimen, as well as obtaining surface topology information.



Credits

Microscopy Australia acknowledges the huge input of time and expertise by the many staff members and associates who have contributed to the development of MyScope over the years.

For the SPM module we thank: Chris Gibson and Steven Moody.