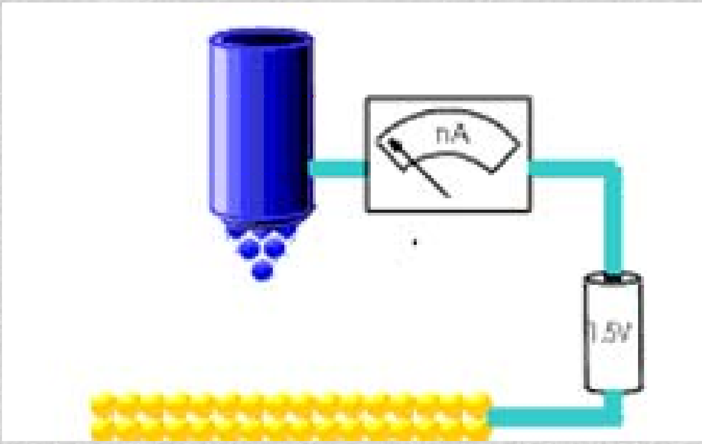
Scanning Probe Microscopy



Prof. Yongqi Fu

Lecture Plan

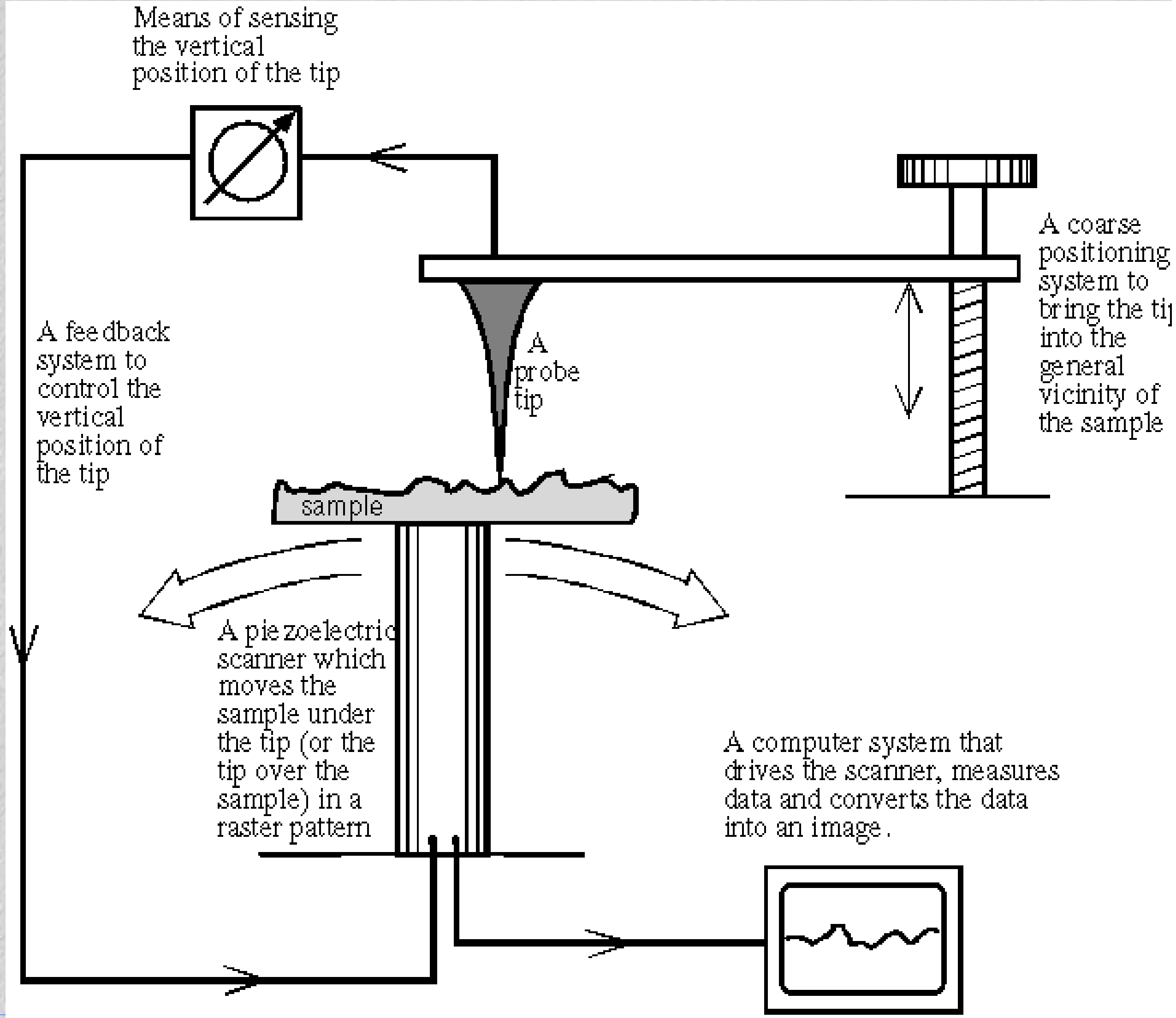
- This week
  - Scanning Probe Microscopy
- Next week
  - Atomic Force Microscopy
- Last week
  - SEM Recap
  - Electron Probe Analysis
  - Short Quiz

Lecture Plan

1. Introduction
   - Comparison with other techniques
   - Fundamentals
   - Tips
2. Construction
   - Scanner
   - Vibration
   - electronics
3. Calibration
4. Applications

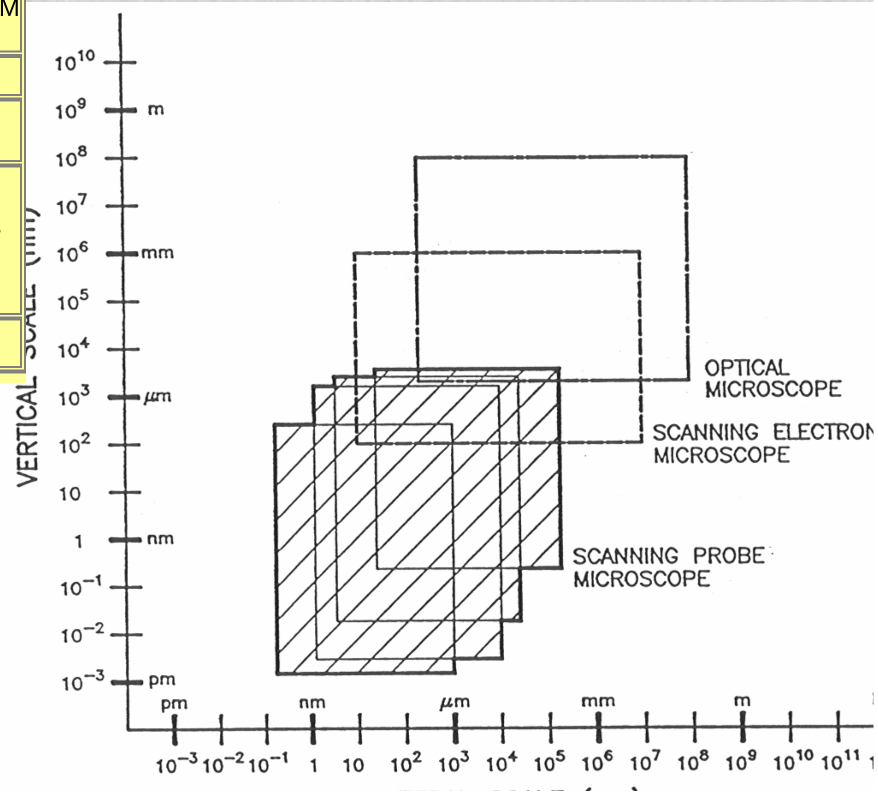
Scanning Probe Microscope

Generalised layout



Comparison of Techniques

Table 1 : Characteristics of Common Techniques for Imaging and Measuring Surface Morphology			
	Optical Microscope	**SEM**	**SPM**
Sample operating environment	ambient air, liquid, or vacuum	vacuum*	ambient air, liquid, or vacuum
Depth of field	small	large	medium
Depth of focus	medium	large	small
Resolution: x,y	1.0 µm	5nm	2-10nm for AFM 0.1nm for STM
Resolution: z	N/A	N/A	0.1nm for AFM 0.01nm for STM
Effective Magnification	1X-2x10^3X	10X - 10^6X	5 x 10^2X - 10^8X
Sample preparation requirement	little	little to substantial	little or none
Characteristics required of sample	sample must not be completely transparent to light wavelength used	surface must not build up charge and sample must be vacuum compatible*	sample must not have local variations in surface height >10µm
** Environmental SEMS can be operated at higher pressures and low eV, but resolution is sacrificed.*			



Fundamentals

⌘ Tunneling current is originated from the wavelike properties of particles (electrons, in the case) in quantum mechanics. When an electron is incident upon a vacuum barrier with po
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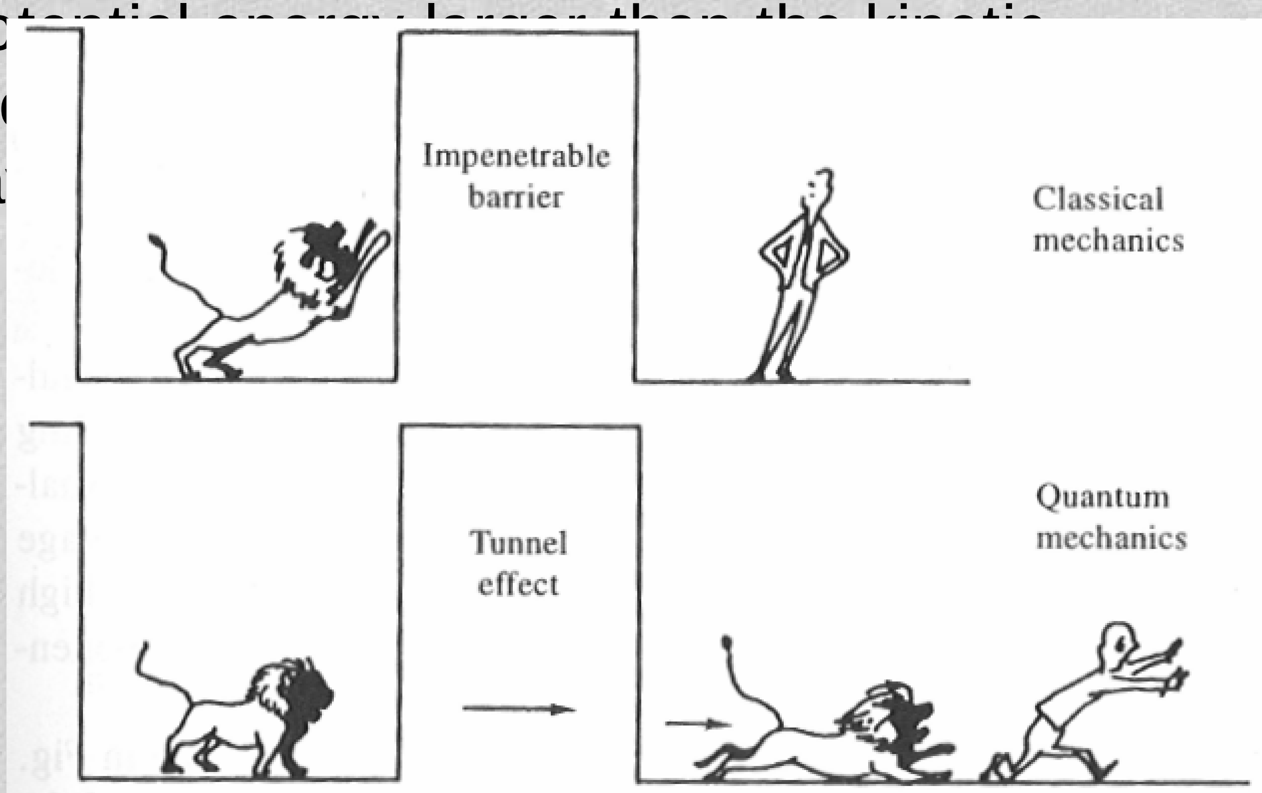
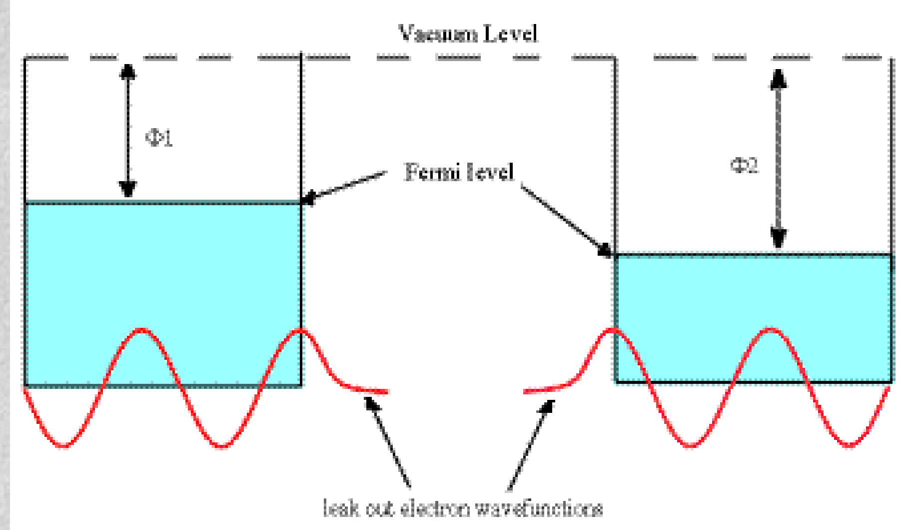
that it may tra
the other side



Fundamentals

- It is shown by the leak out electron wavefunction.



- If two conductors are so close that their leak out electron wavefunctions overlap. The electron wavefunctions at the Fermi level have a characteristic exponential inverse decay length K given by

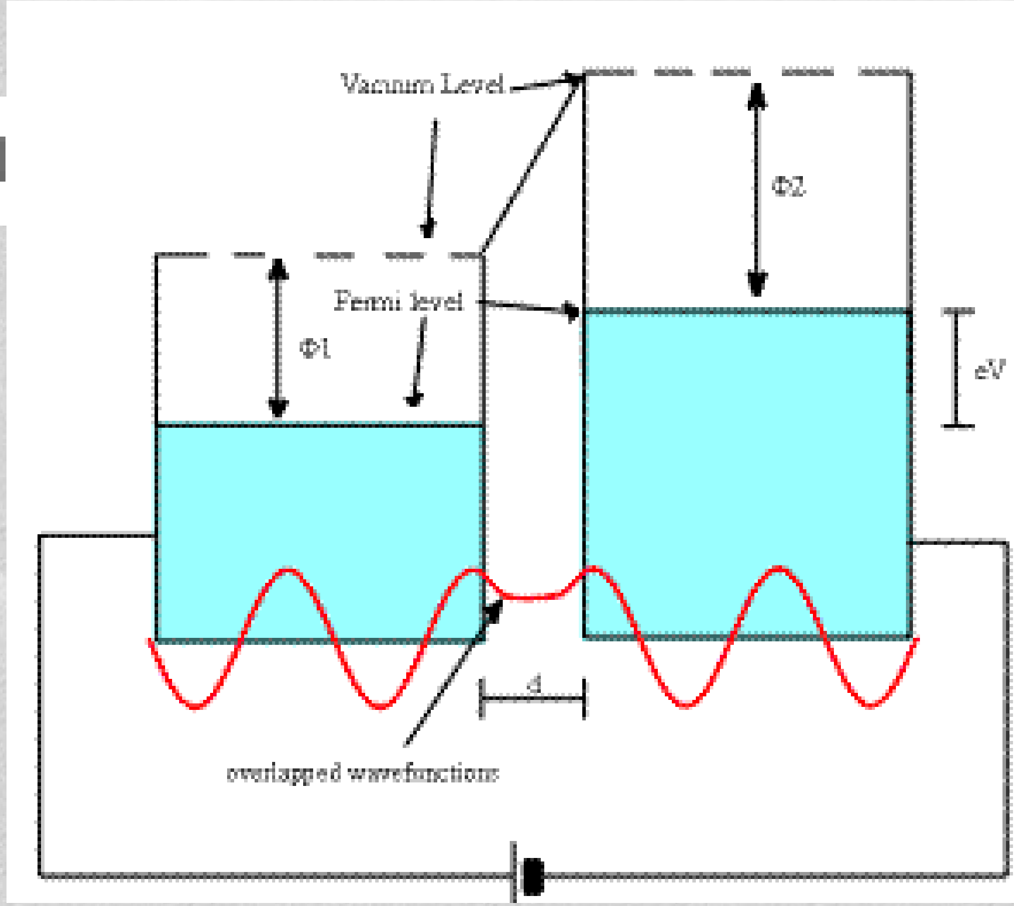
$$K = \frac{\sqrt{2m\Phi}}{\hbar}$$

- m is mass of electron, is the local tunneling barrier height or the average work function of the tip and sample. When a small voltage, V is applied between the tip and the sample, the overlapped electron wavefunction permits quantum mechanical tunneling and a current, I will flow across the vacuum gap.

Fundamentals

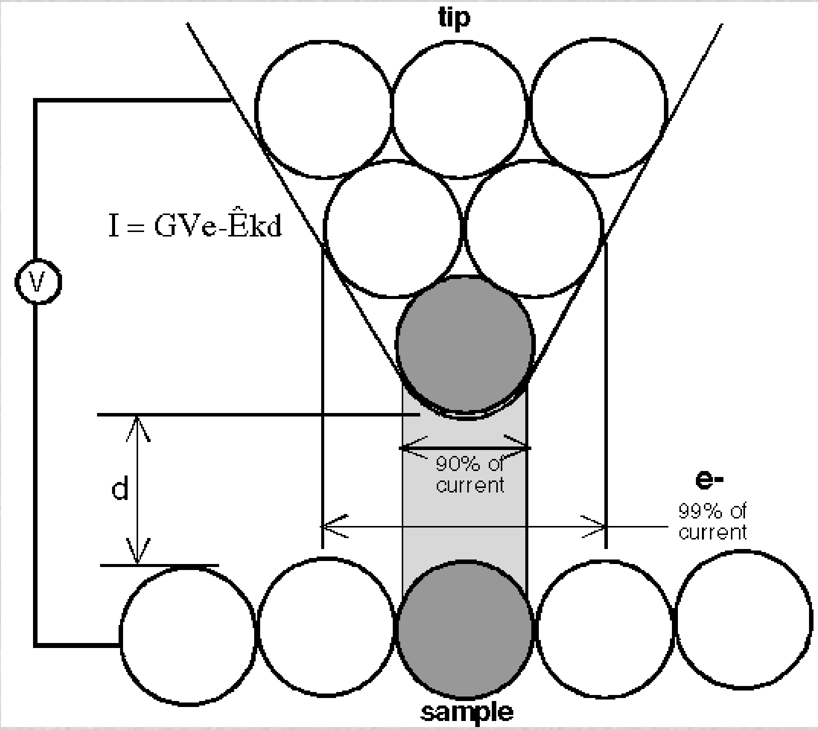
- At low voltage and temperature

$$I \propto \exp(-2Kd)$$



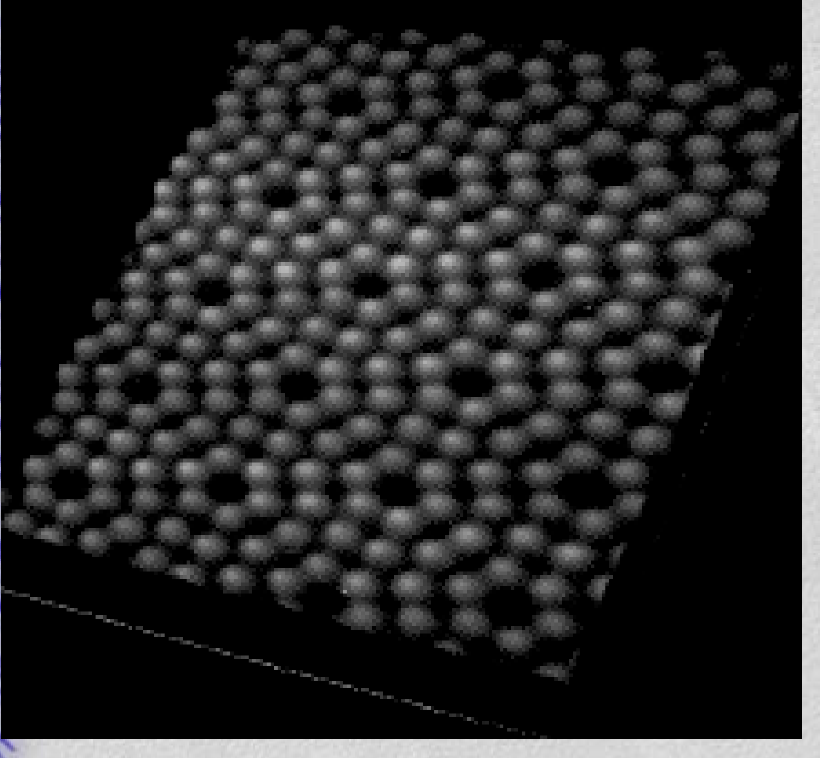
Fundamentals

- The tunneling current is an exponential function of distance;
- If the separation between the tip and sample changes by 10% (i.e. by 1 angstrom)
- This relationship gives STMs their remarkable sensitivity.
- STMs can image the surface of the sample with *sub-**angstrom*** precision vertically, and ***atomic*** resolution laterally.



Fundamentals

- This is the STM image of Si(111)-7x7 surface, the white spots represents the position of the atoms

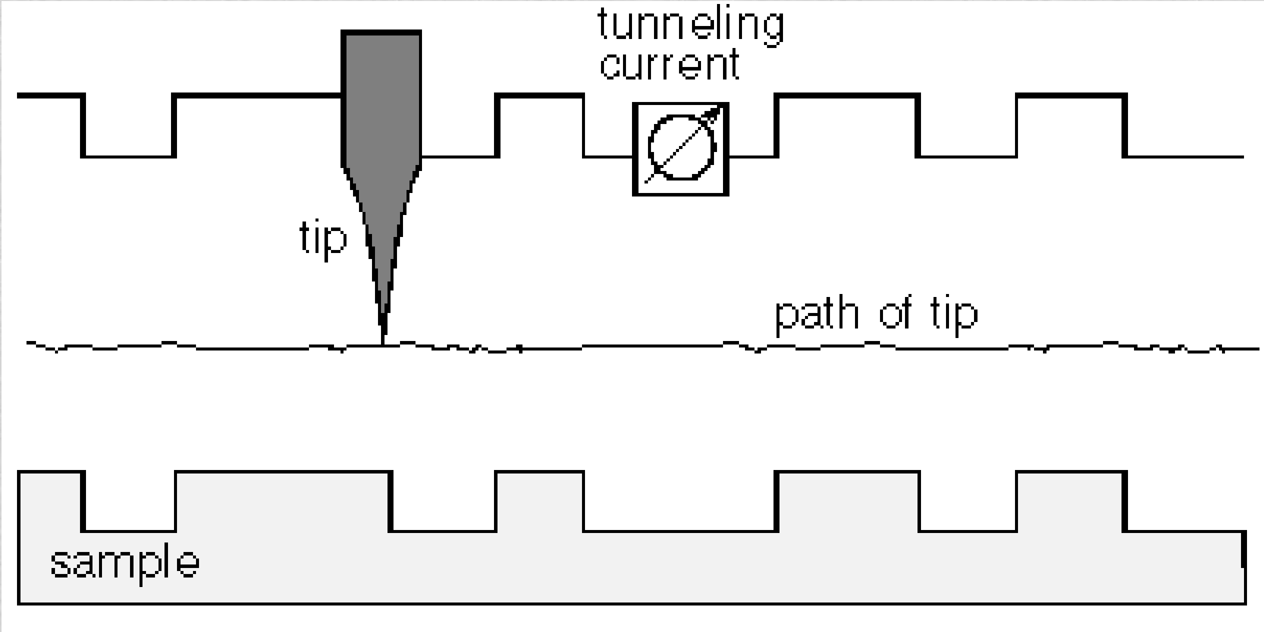


Remember !

- STM does NOT probe the nuclear position directly, but rather it is a probe of the electron density, so STM images do not always show the position of the atoms, and it depends on the nature of the surface and the magnitude and sign of the tunneling current.

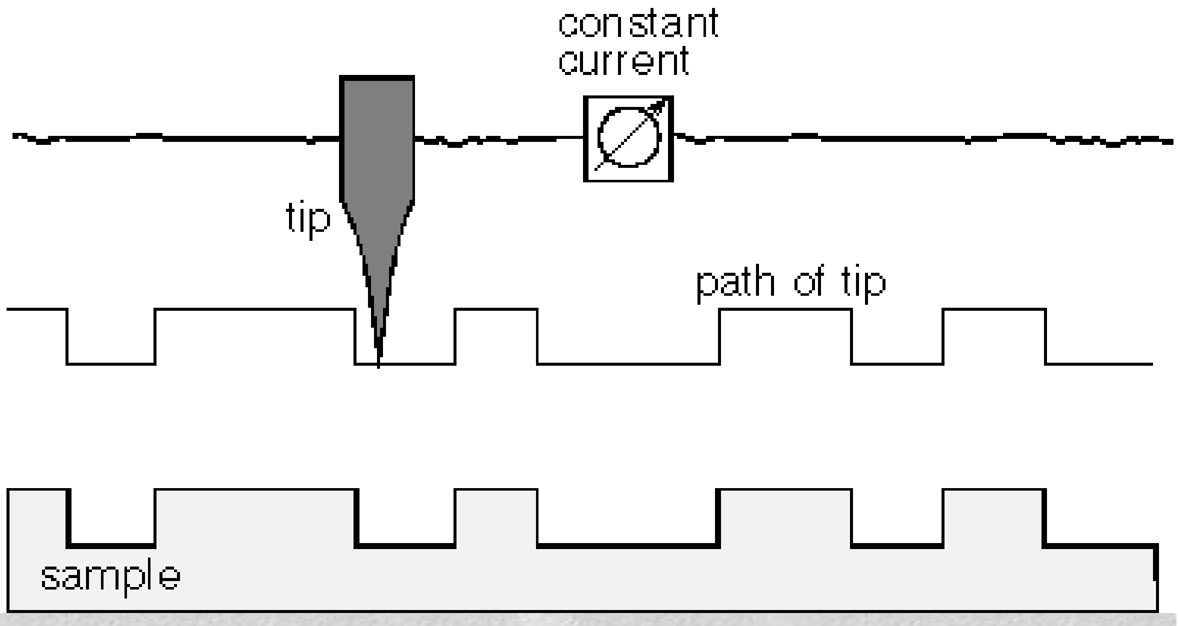
Constant Height

- Tip travels in a horizontal plane
- Tunneling current will vary
- Only suitable for smooth surfaces
- Relatively fast



Constant Current

- The STM uses feedback to keep the tunneling current constant.
- Tip-to-sample distance constant to a few hundredths of an angstrom
- Can measure irregular surfaces
- time longer

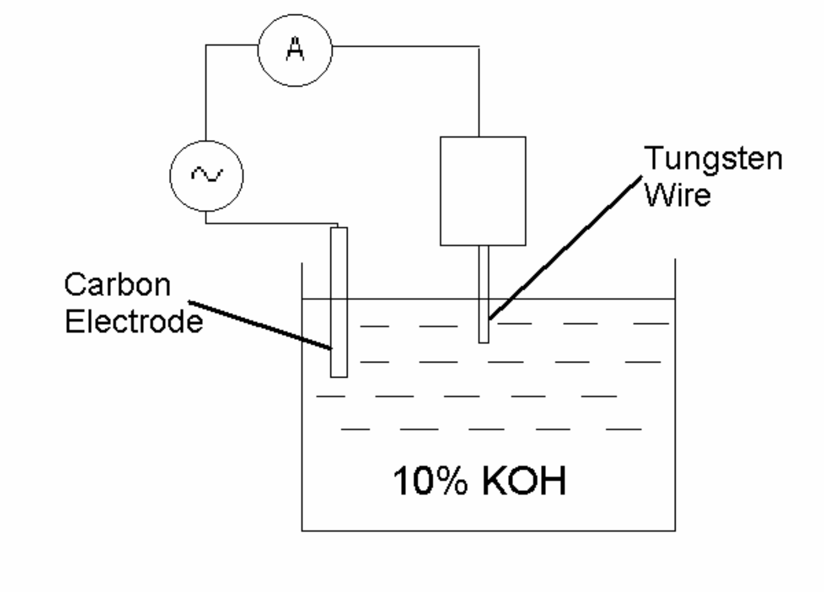


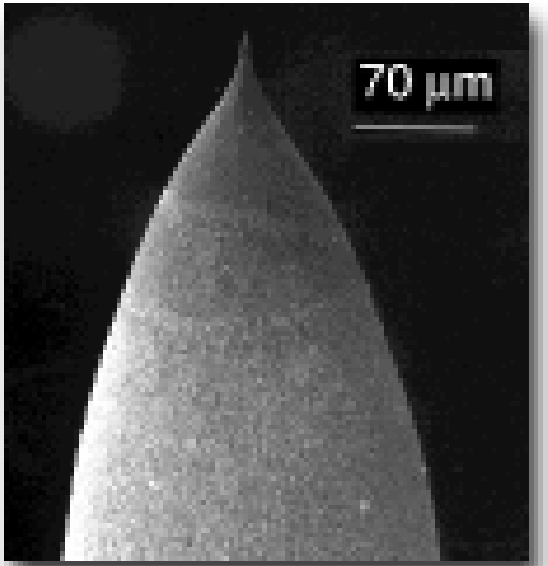
Tip

- To ensure atomic resolution it is essential the tip is of one atom dimension at its end.
- Easy!!!!!!!
- But hard to check :)

Tip

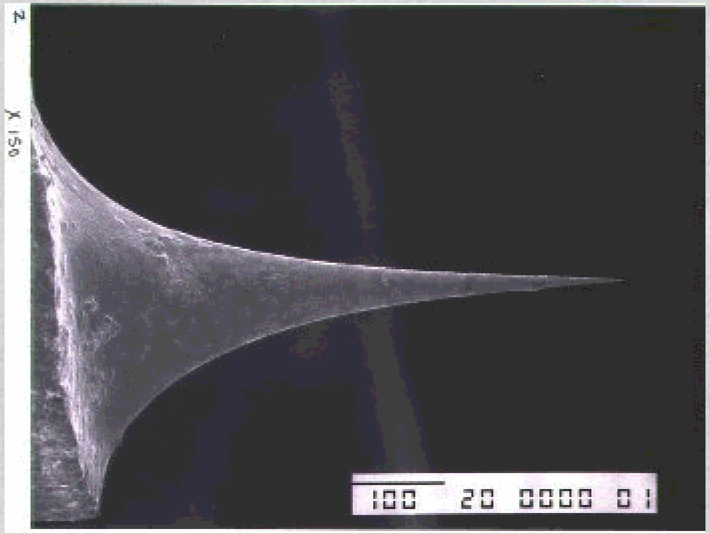
- The most common types of STM tips are electrochemically etched tungsten wires and cut platinum-iridium wires.
- Use of materials that do not oxidise in air such as Pt-Ir or Au might be advantageous as far as the stability of tunneling in air.
- However, tungsten is most commonly used in ultra-high vacuum, where oxidation is not a factor.





Tip

- This tip was made by electro-chemically etching tungsten wire in KOH at 3 Volts and 18 milliAmps for 67 minutes.

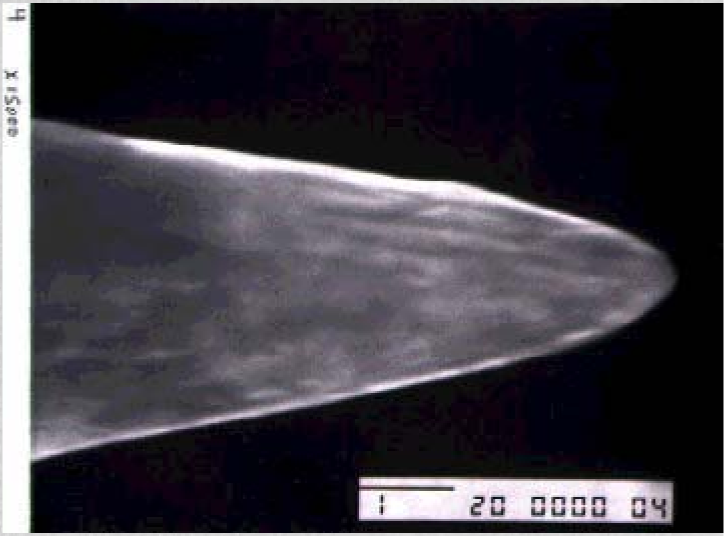


http://www.phys.unt.edu/stm/tips.htm

Tip

- KOH at 3 Volts and 18 milliAmps for 67 minutes

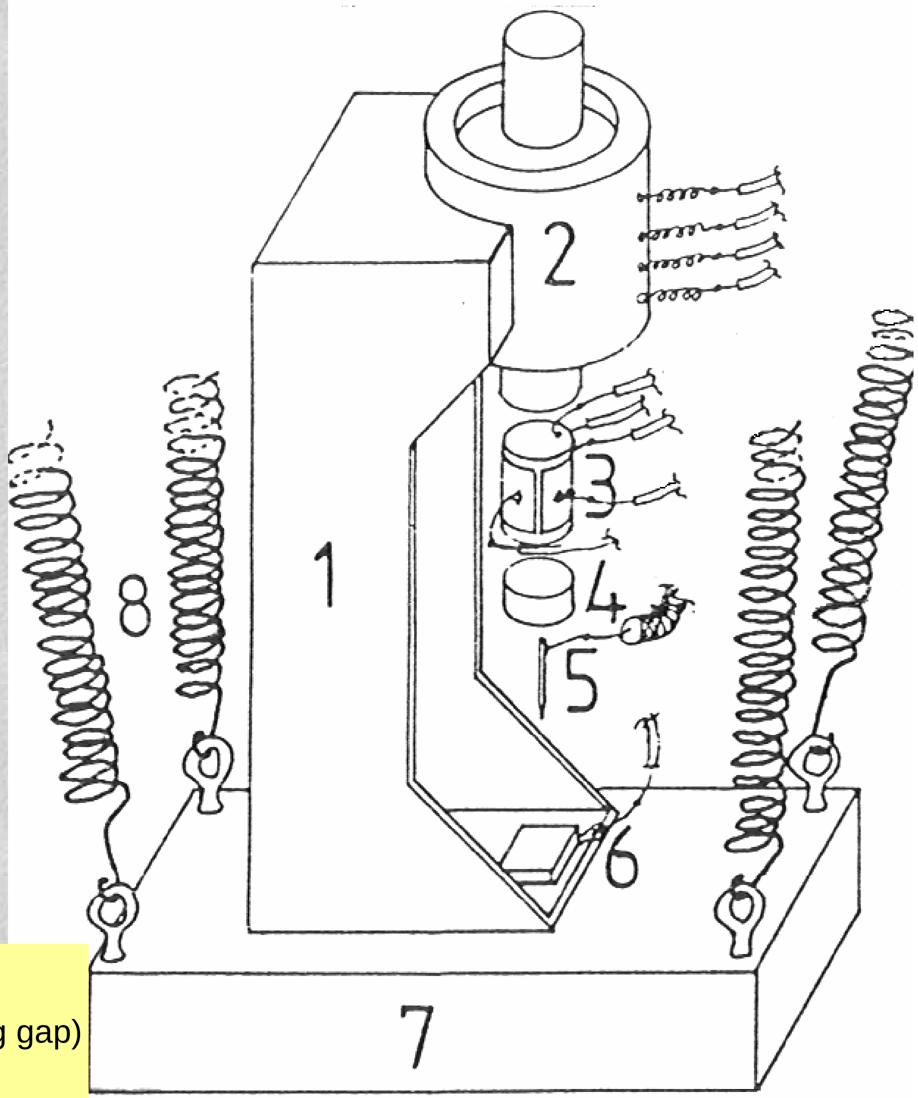
- KOH at 4 Volts and 19 milliAmps for 45 minutes.



Tip Crashing

- During STM operation it is inevitable that the tip will crash into the sample, even with well-designed control electronics and with extreme care by the operator.
- Sometimes crashing can ***improve*** the tip condition, but in general it will lead to blunting.

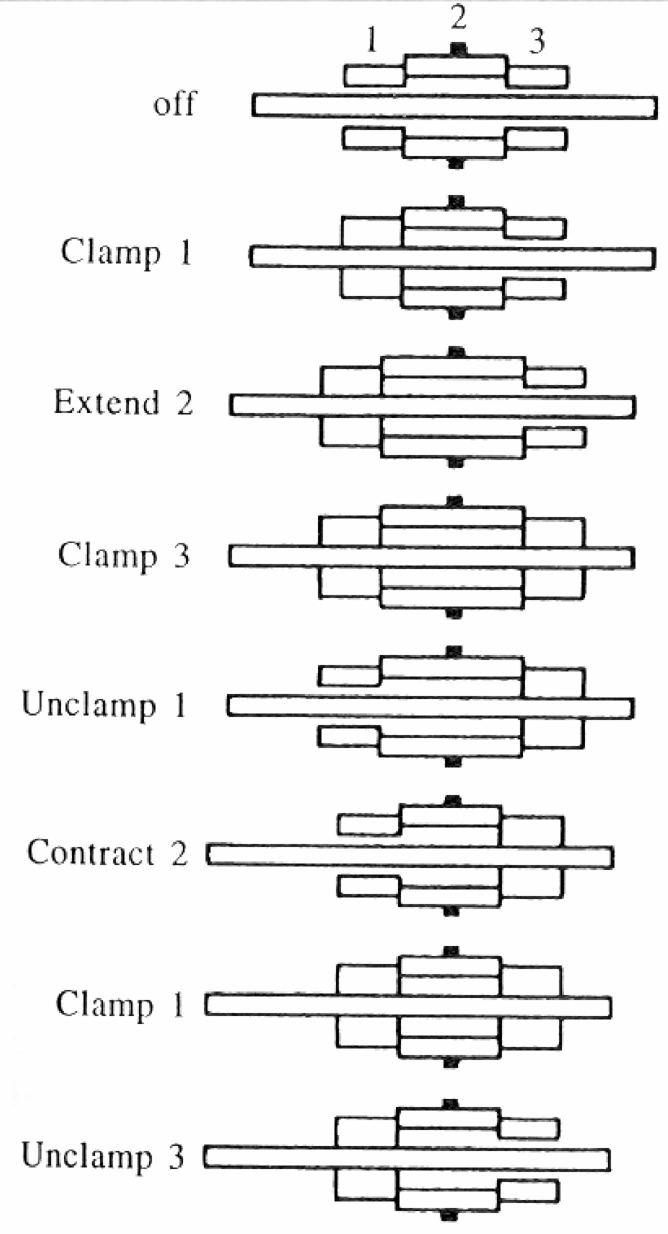
SPM Construction



1	Light rigid connection between tip and specimen
2	Inchworm for tip approach
3	Piezo cylinder for control of x and y (scanning) and z (tunneling gap)
4	Insulating shield/ tip holder
5	Tip with low noise/ bias connection
6	Specimen
7	High mass base
8	Soft spring/ damping system

Coarse Approach

- Tip approach is usually controlled by a piezoelectric "inch worm". The probe is mounted on a piezo shaft that runs through cylindrical piezo clamps. By alternately clamping at opposite ends and expanding and contracting the region of the shaft between the clamps, the shaft and therefore the probe can be moved a distance of several millimetres in 1nm steps.



Scanner

- The requirements of a good scanner are:
  - *high resolution* - the necessary resolution is less than 1 angstrom in the lateral direction (x,y) and 0.05 angstroms in the vertical direction (z),
  - *orthogonality* - movement in each of the three axes should be independent,
  - *linearity* - the amount of movement should be proportional to the applied voltage,
  - *mechanical rigidity* - a rigid scanner will have a high resonant frequency which is desirable for both vibration isolation and feedback performance, and
  - *large range* - it is desirable to cover as large a sample area as possible.

Scanner

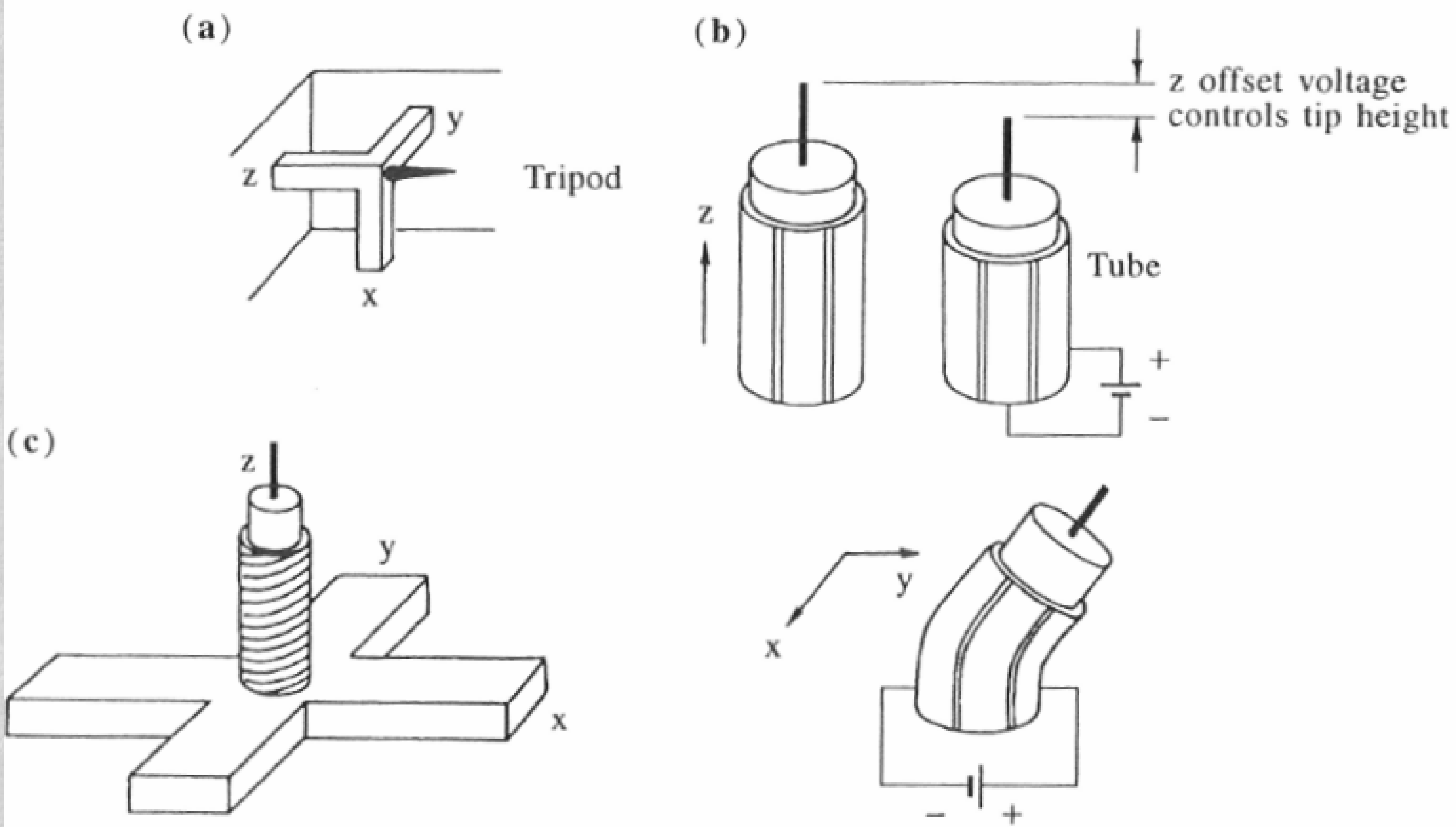
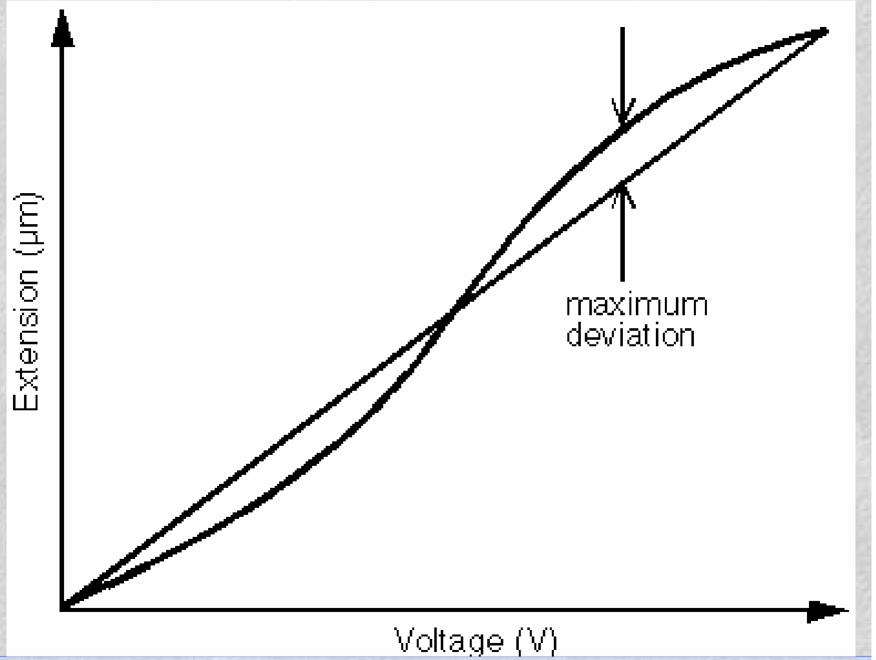


Fig. 4.4. Common three-dimensional scanners: (**a**) tripod, (**b**) single tube, and (**c**) a cross combined with a single tube

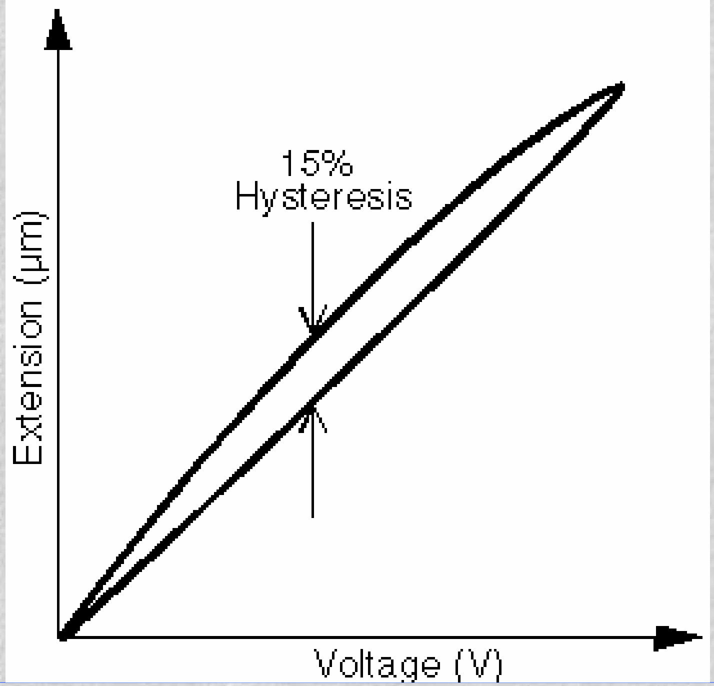
Scanner - Nonlinearity

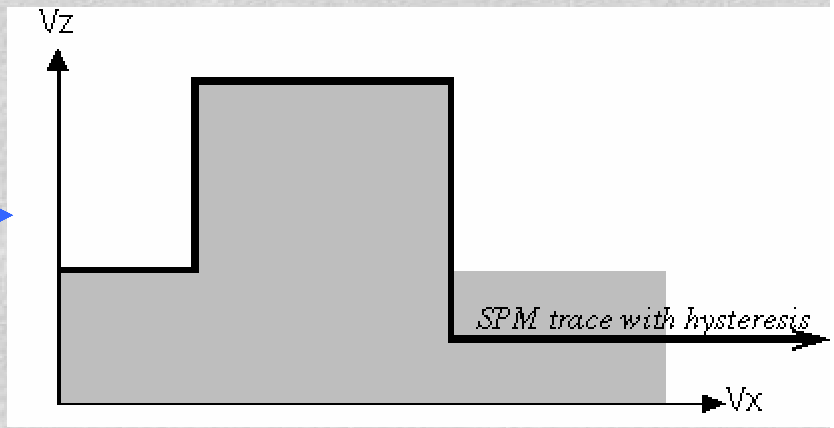
- *Nonlinearity* - this is the ratio of the maximum deviation from the linear behaviour to the ideal linear extension at that voltage.
- It is expressed as a percentage
- Typically ranges from 2% to 25%.



Scanner - Hysteresis

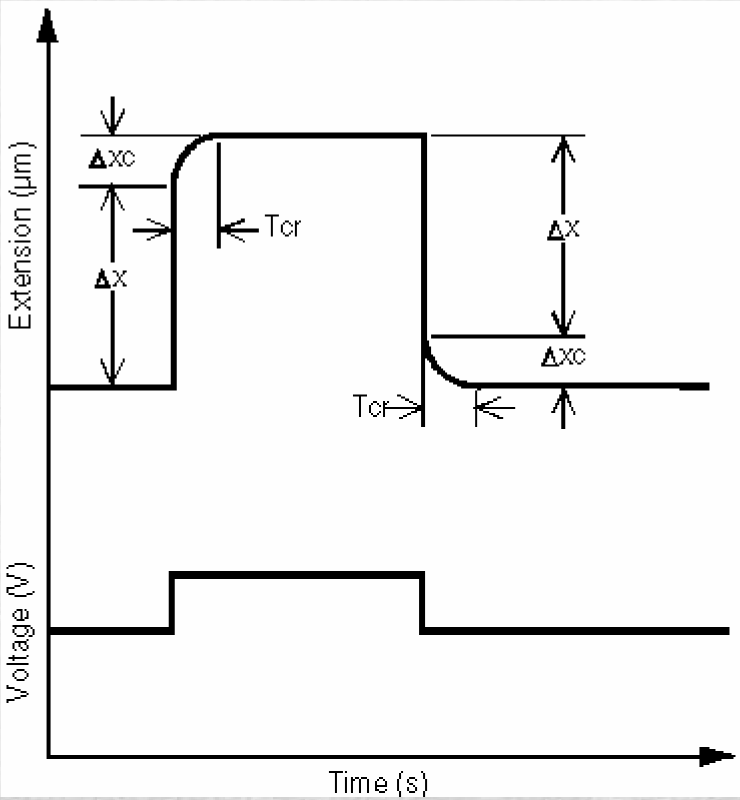
- Ratio of the maximum divergence between the two curves to the maximum extension that a voltage can create in the scanner (the change in Y/Ymax).
- Hysteresis can be as high as 20% for piezoelectric materials.

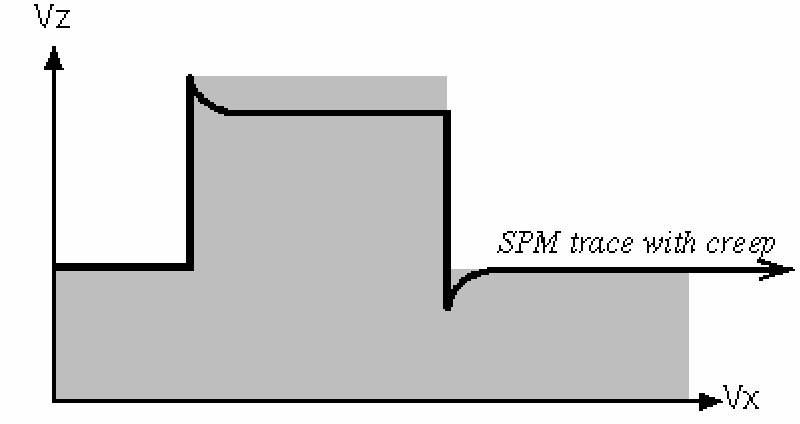




Scanner - Creep

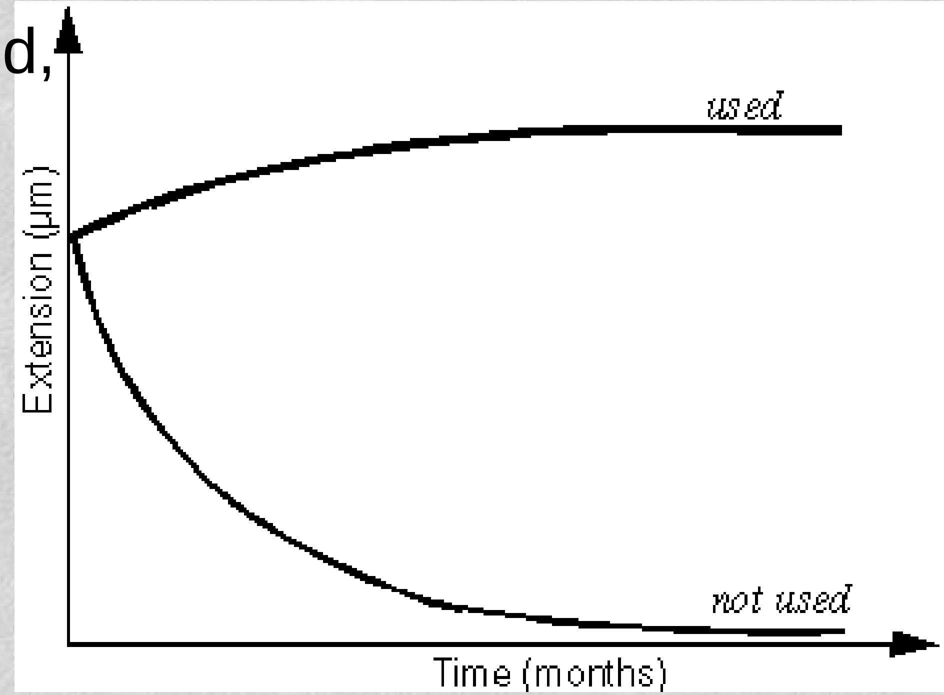
- *Creep* - when an abrupt change in voltage occurs, the material does not change dimensions all at once.
- Creep is expressed as the ratio of the second dimensional change to the first.
- Typical values are in the range of 1% to 20% for times of 10 to 100 seconds.





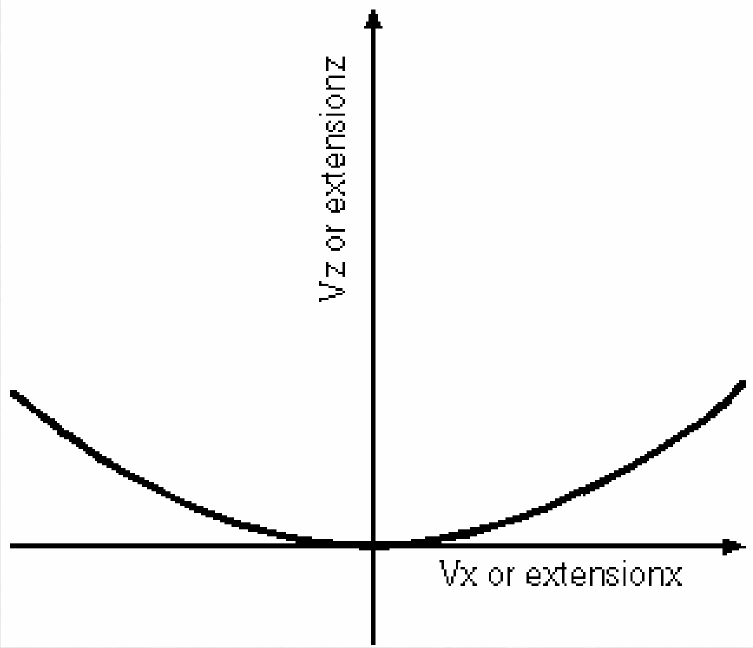
Scanner - Aging

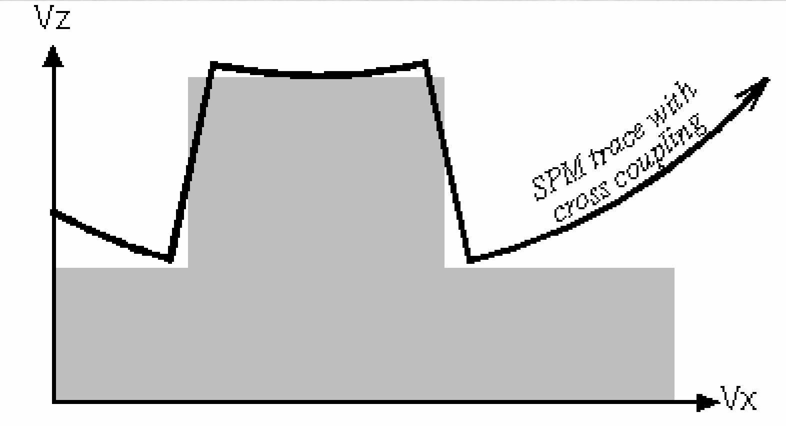
- *Aging* - the strain coefficient *d* of piezoelectric materials changes exponentially over time and use.
- The aging rate is the change in strain coefficient per decade of time.
- When a scanner is not used,
  - the deflection decreases.,
- When a scanner is used
  - the deflection increases



Scanner - Cross-coupling

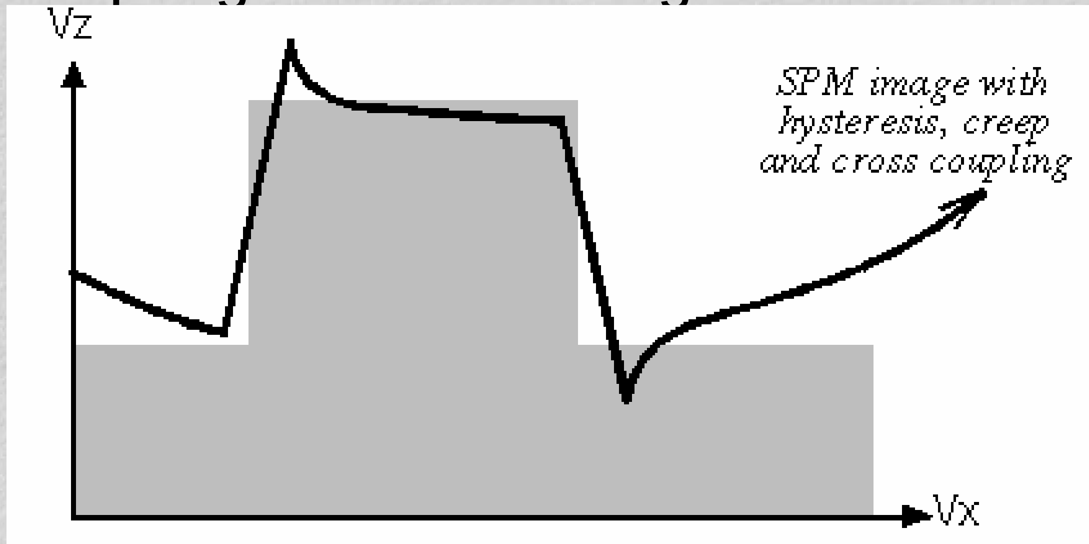
- *Cross-coupling*- this refers to the tendency of the x-axis or y-axis scanner movement to result in a spurious z-axis motion.
- This can be attributed to non-uniform electric fields and scanner construction which leads to geometric errors





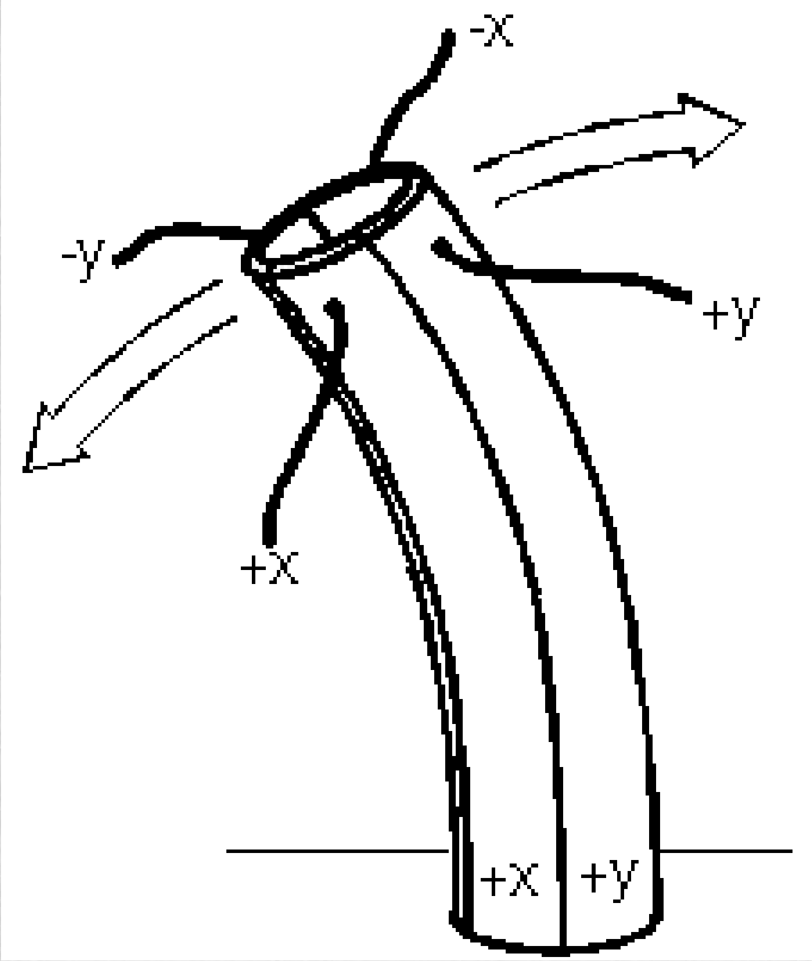
Scanner - Combined

- Scanner hysteresis causes the upward and downward sidewalls of the step to have different slopes, because the voltage required to contract the scanner as the tip goes up the step is smaller than the voltage required to extend the scanner as the tip goes down the step.
- Creep causes the exponential decay that makes the left side of the top of the step higher than the right side.
- Cross coupling adds ***curvature*** to the entire profile



Scanner - Mechanism

- Simple tube design.
- In this design the scanner is a hollow tube.
- Electrodes are attached to the outside of the tube, segmenting it electrically into vertical quarters, for +x, +y, -x, and -y travel.



Scanner - Mechanism

- The maximum scan size that can be achieved with a particular piezoelectric scanner depends upon:
  - the length of the scanner tube,
  - the diameter of the tube,
  - its wall thickness, and
  - the strain coefficients of the particular piezoelectric ceramic from which it is fabricated.

Vibration Isolation

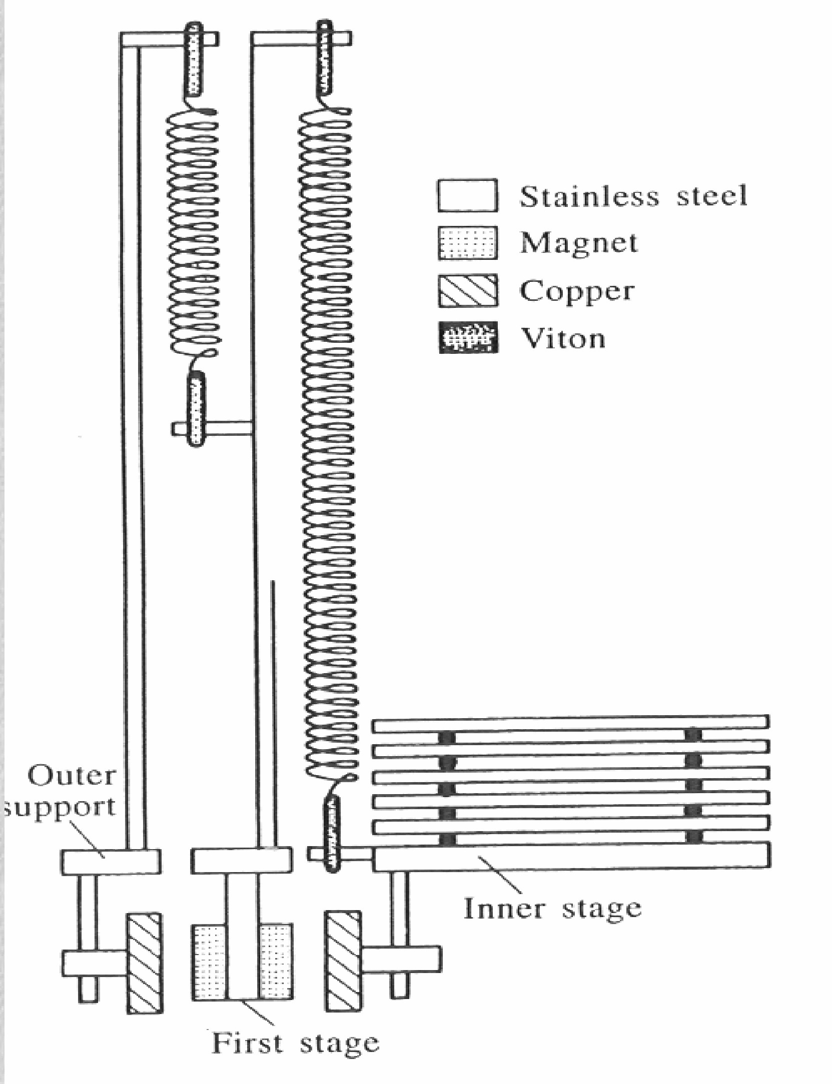
- Mainly vibrations, thermal drift and electronic noise limit the accuracy with which the tip-sample gap can be controlled and monitored.
- There are three main sources of vibration:
  - acoustic room vibration,
  - internally generated resonance, and
  - external vibrations from the floor.

Vibration Isolation

- Operating the STM in a quiet room or isolating the system acoustically solves the first problem.
- The second source of vibration can be minimised by raising the resonant frequency and therefore the scan frequency of the system, the tip/specimen unit has to be made as ***light*** and ***rigid*** as possible with a mechanical path containing as few components as possible (more easily achieved with compact designs with tube scanners).

Vibration Isolation

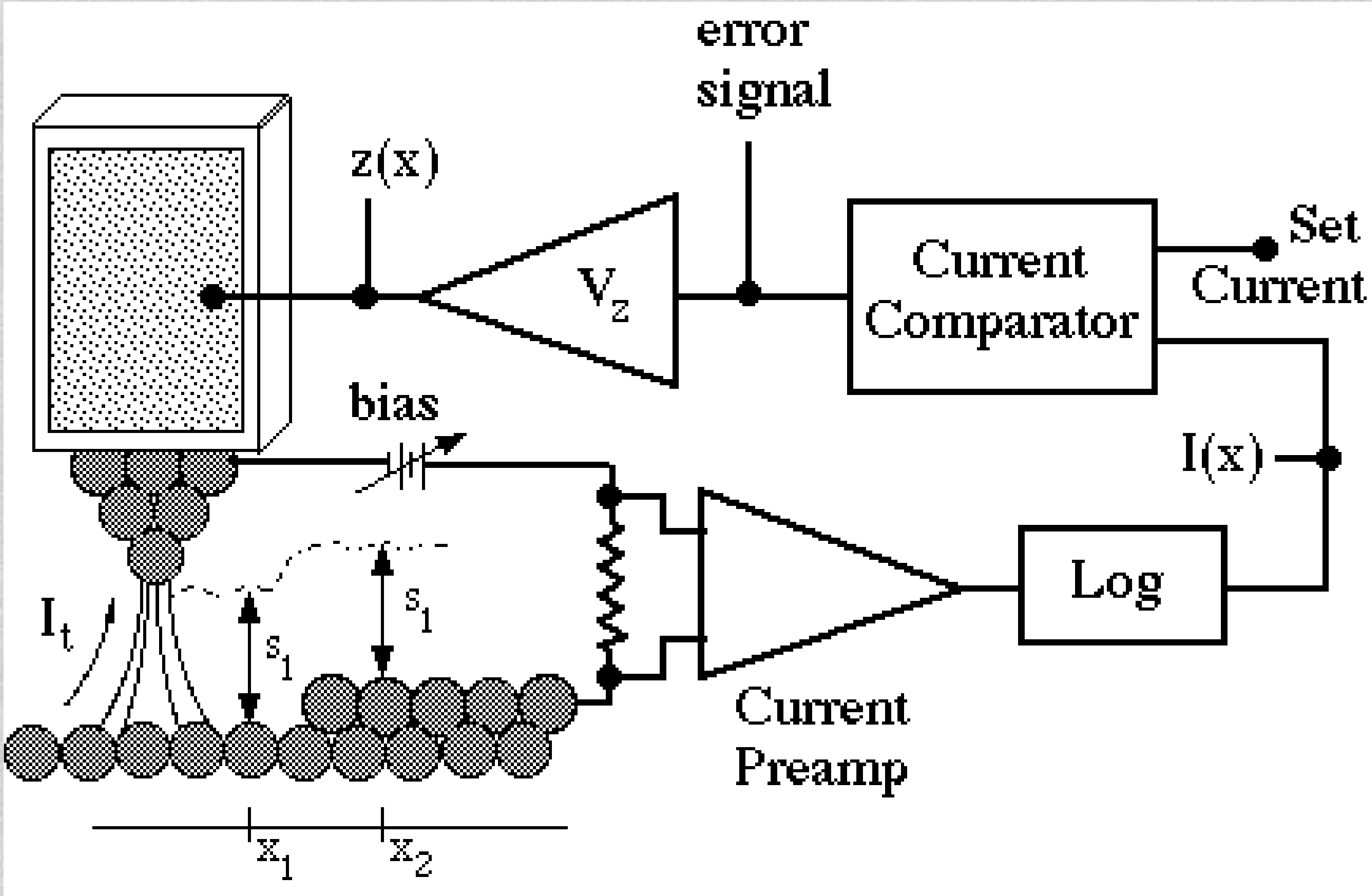
- For the third problem, vibration isolation is achieved by mounting the STM on a heavy mass isolated from the environment by damped soft springs.



Electronics

- The main aspects that have to be carefully considered when designing the electronics for controlling such a sensitive device as a STM, are noise and drift.
- Today's state-of-the-art electronics provides components with intrinsically ultra low noise and with very little drift over long periods of time and over a wide temperature range.
- However, the layout of the circuitry and shielding are critical to the performance of the system as a whole.
- The grounds of low voltage (tunneling current sensing), high voltage (piezo control), and digital (computer control & image processing) signals should be kept separate and the signal lines should be made to run as far apart as possible

Electronics

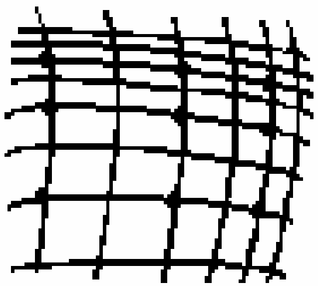


Calibration

- Calibration of the scan units can be achieved by:
  - attaching capacitors or differential transformers,
  - accurate mechanical gauging, or
  - imaging a known structure

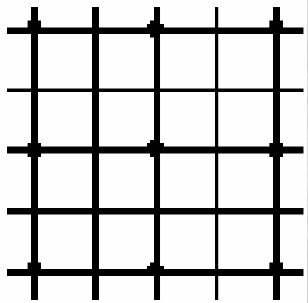
Calibration

- The usual technique for correcting for scanner nonlinearities is to image a calibration grating.
- The grating is of known form and dimension and the system will adjust the measurement data to match the expected results.
- The information is typically stored in the form of a look-up table.



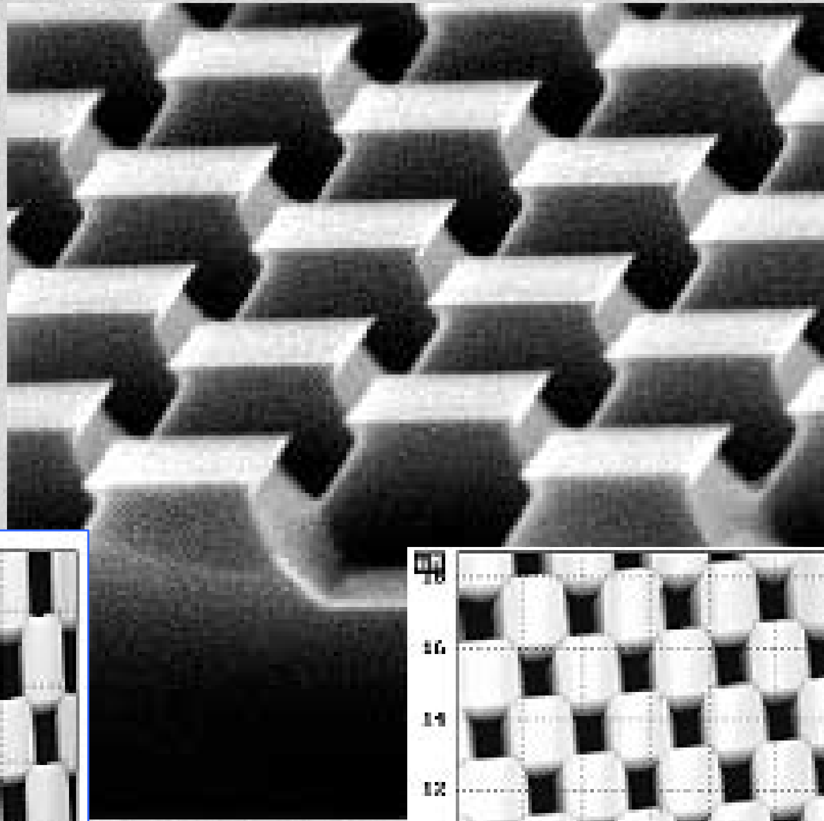
Look-Up Table

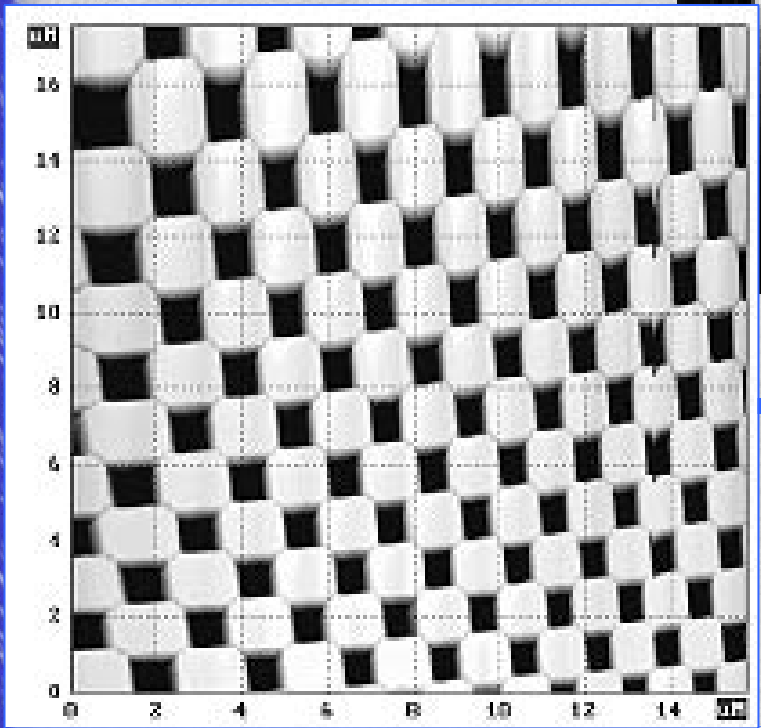
32.1	45	96	14
44.5	682.7	532	87
.347	556	.459	3321
128	32	441	.45
3	.34	21	3.68
67	.349	.5697	221
2167	29	311	65
.321.2	993	87	239
46.89	27	554	12

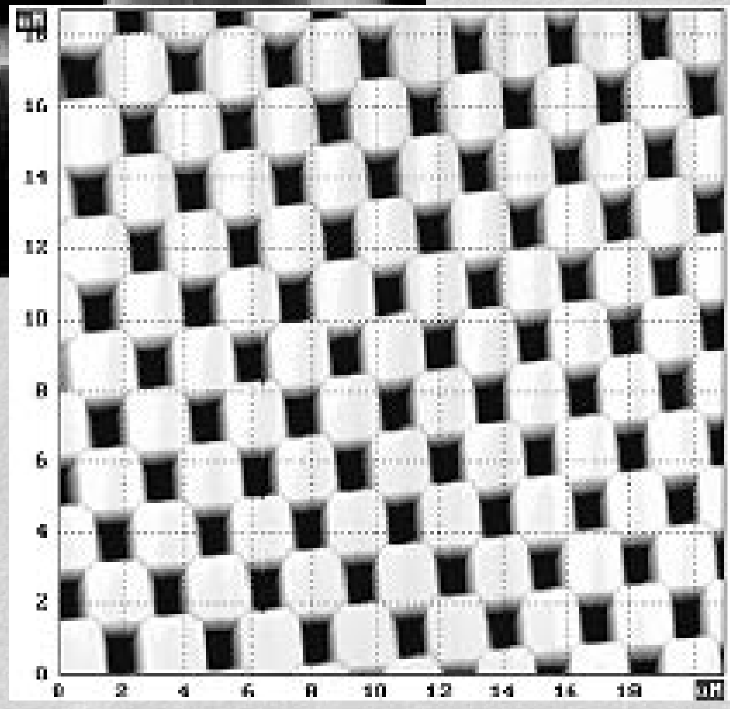


- More advanced software-correction algorithms that can model the scanner tube's non-linear response to applied voltages is also available.

Calibration



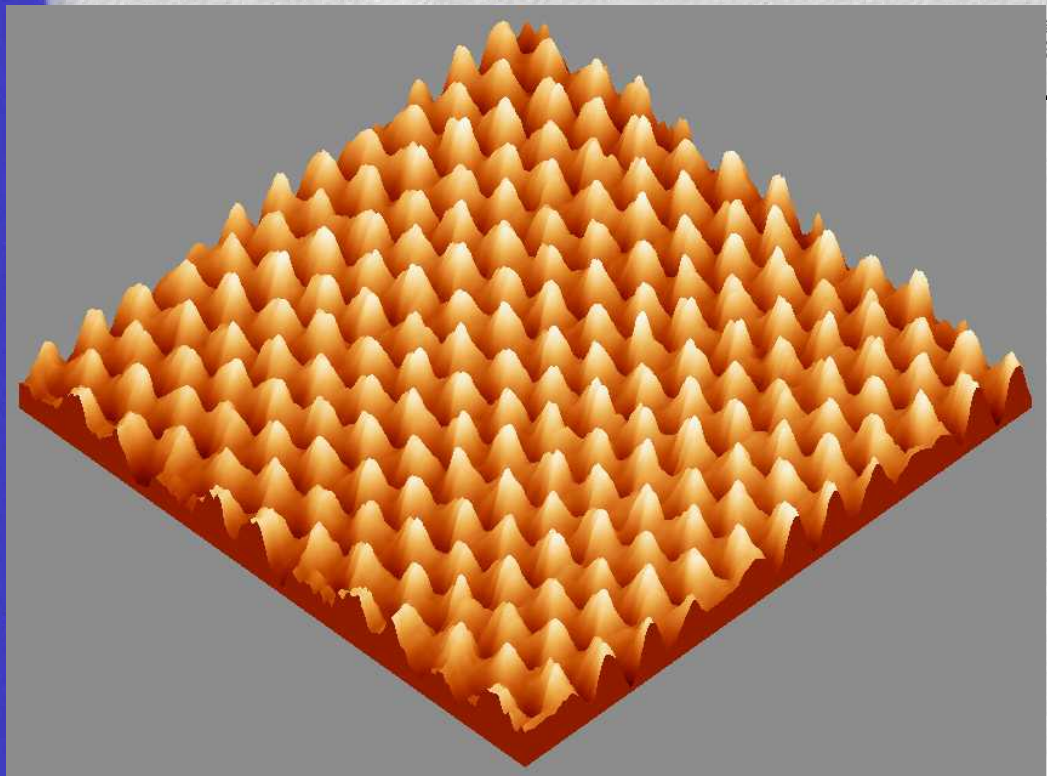


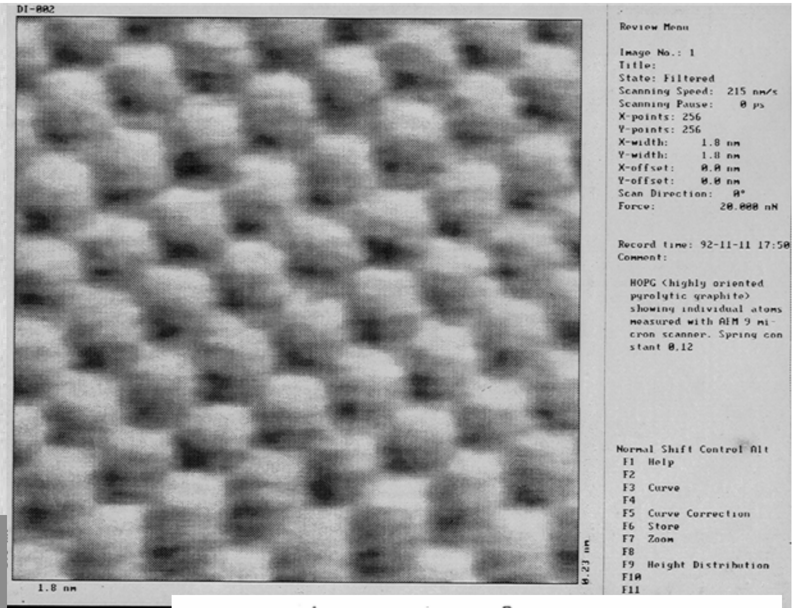


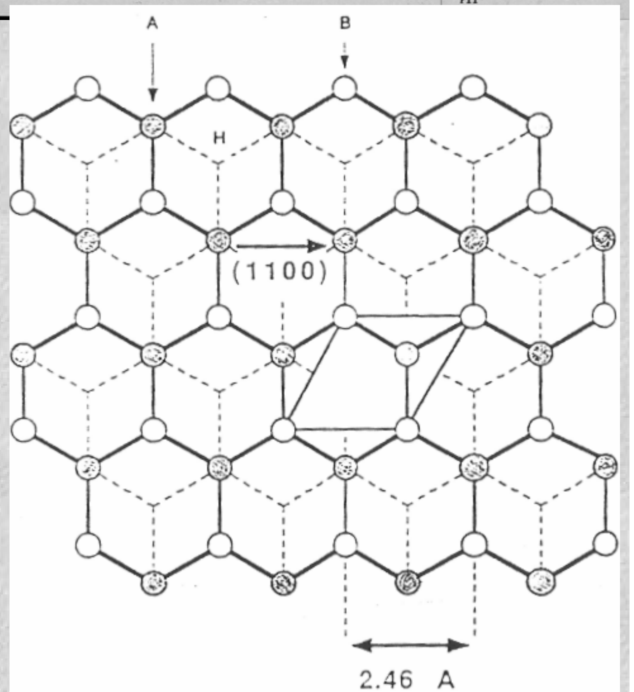
Calibration

- Software correction is generally relatively simple and inexpensive to implement.
- Their main disadvantage is they only compensate partially for scanner nonlinearities.
- Highly dependent on
  - the scan speed and
  - scan direction.

Calibration : HOPG

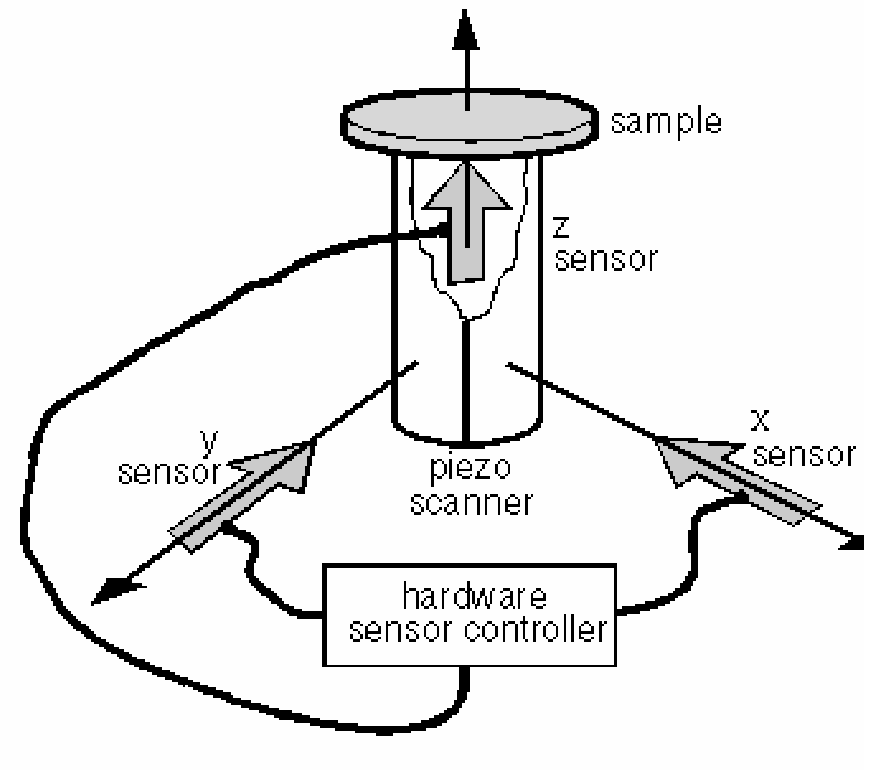




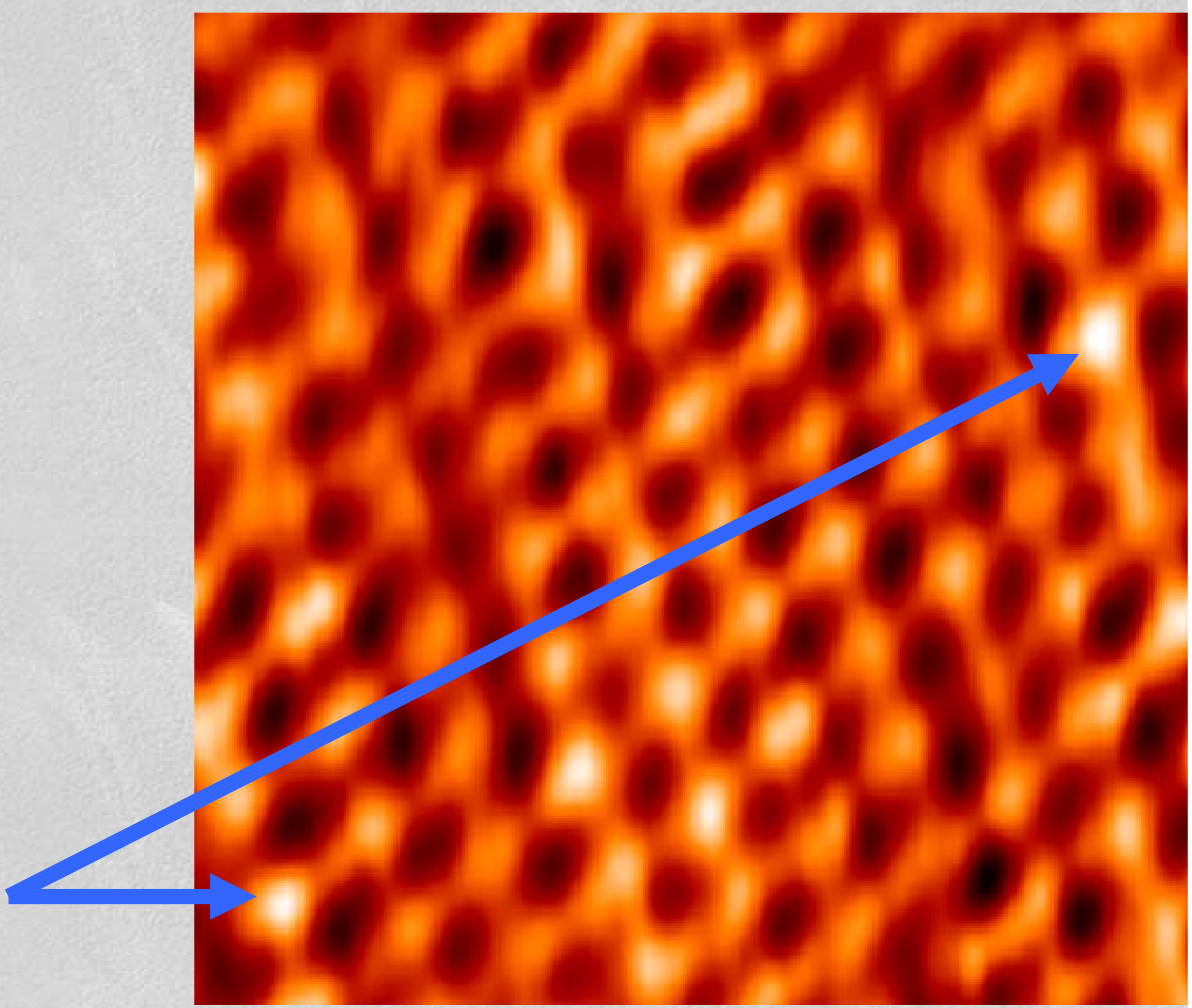


Calibration

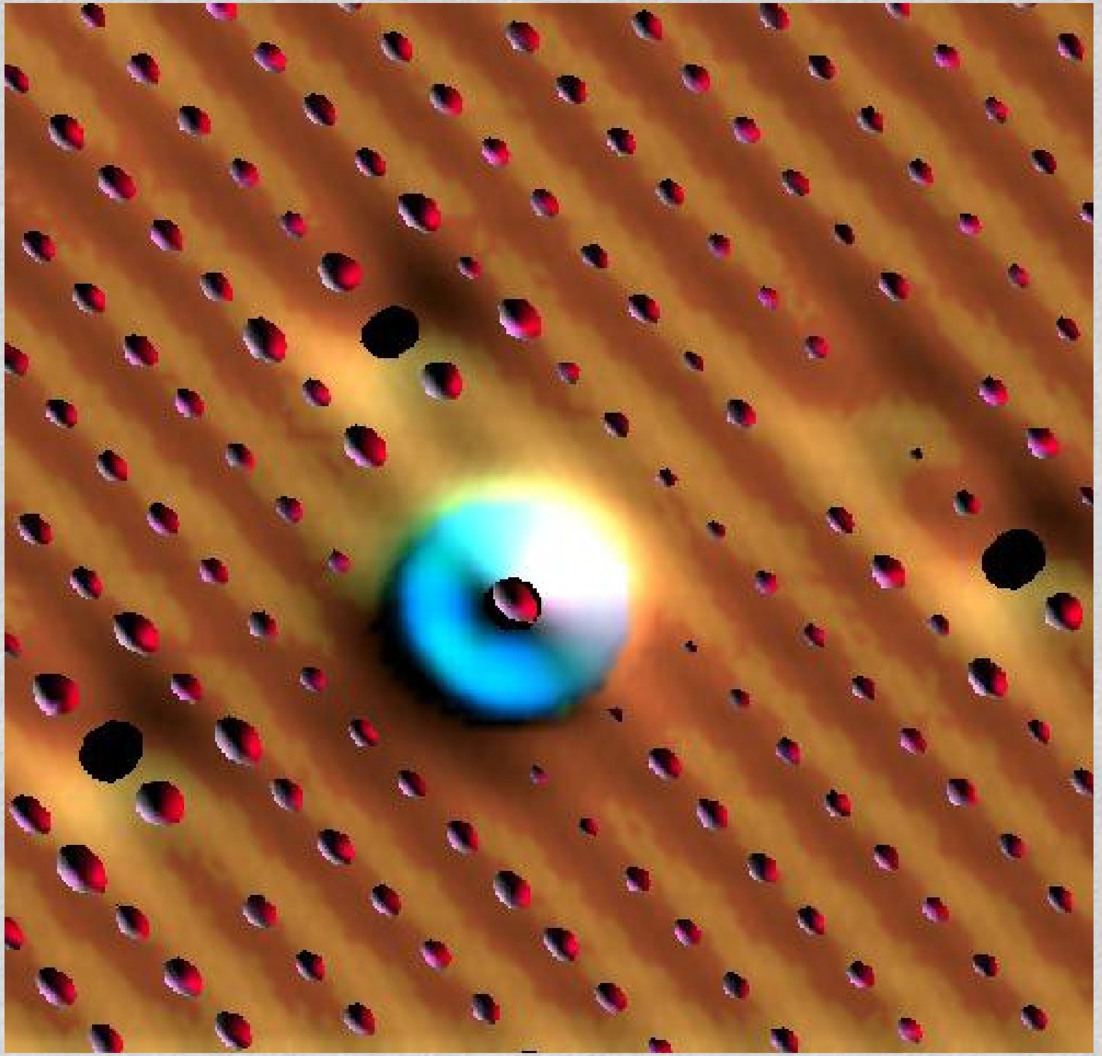
- The signal read from the sensor of each axis is compared to a signal that represents the intended scanner position along that axis.
- A feedback system applies a voltage to the scanner to drive it to the desired position. With hardware sensors it is possible to reduce total nonlinearity to less than 1%.



Applications

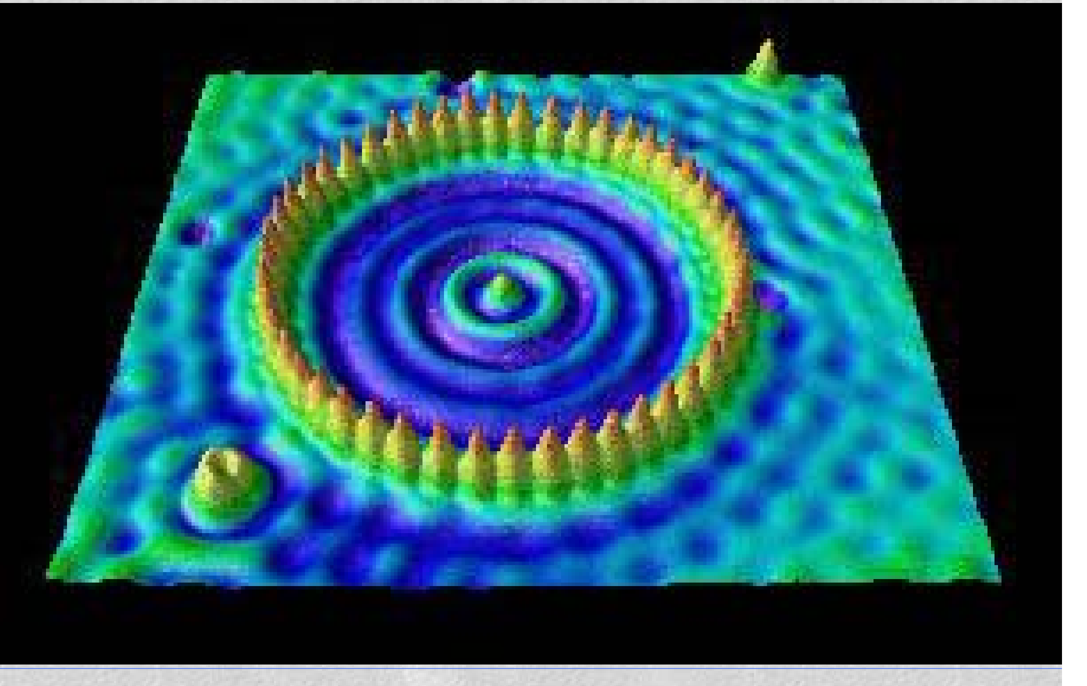


Applications



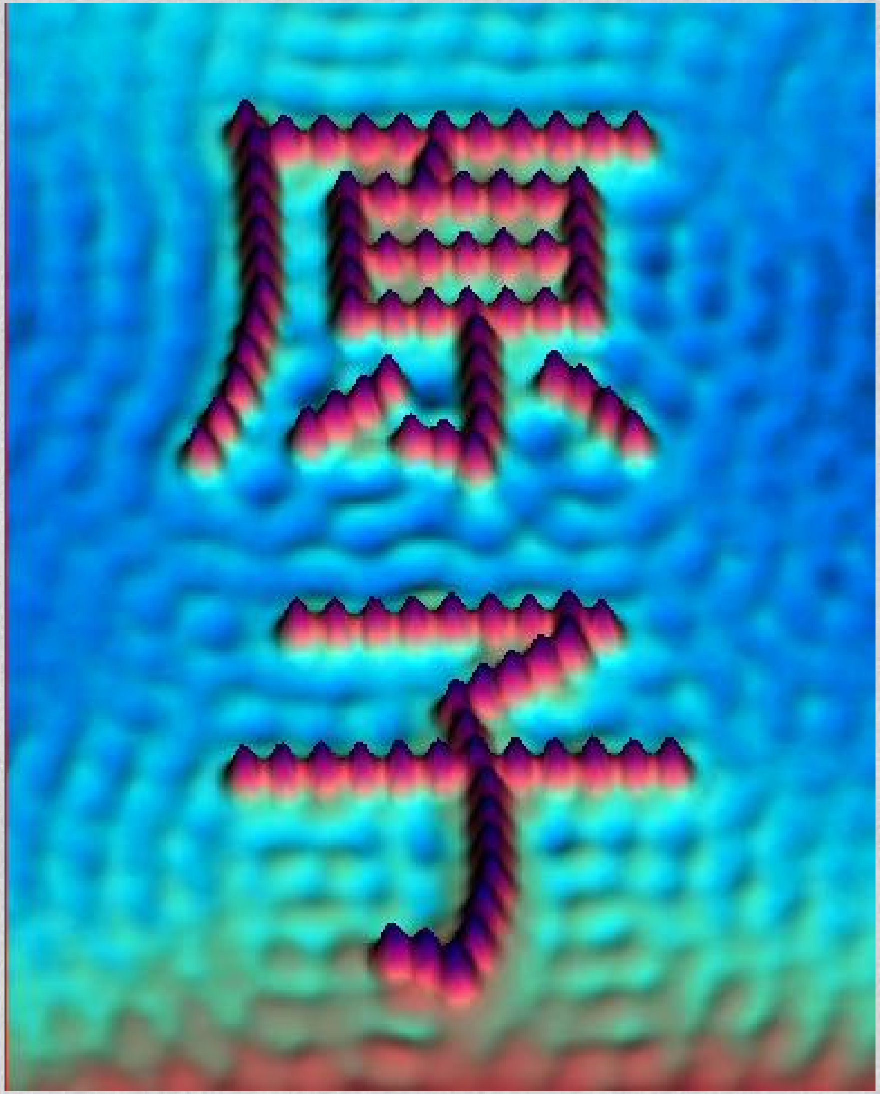
Applications

- Here is a STM image showing iron atoms adsorb on a copper (111) surface forming a "quantum corral"in a very low temperature (4K).
- Actually, the image shows the contour of the local density of electron states. The corral is about 14.3 nm in diameter.



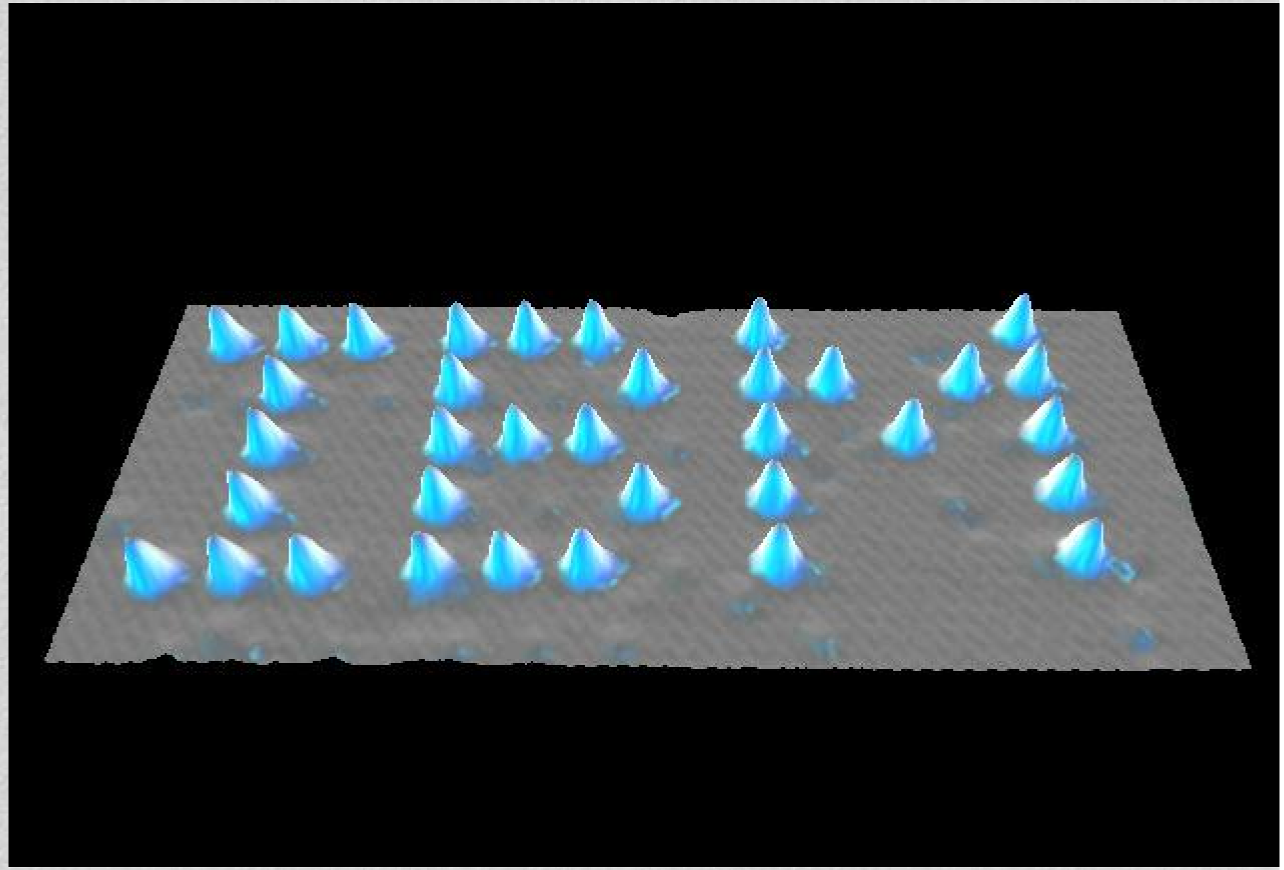
Applications : Nanotechnology

- Iron on Copper (111)



Applications : Nanotechnology

- Xenon atoms on Nickel (110)



Summary

- Introduced SPM
- STM principle
- Tip Production
- Mechanism
- Errors
- Calibration
- Applications

Next Week.....

Atomic Force Microscopy