

AP 5301/8301

Instrumental Methods of Analysis and Laboratory

Lecture 3

Microscopy (II): scanning electron
microscopy & scanning probe microscopy

Prof YU Kin Man

E-mail: kinmanyu@cityu.edu.hk

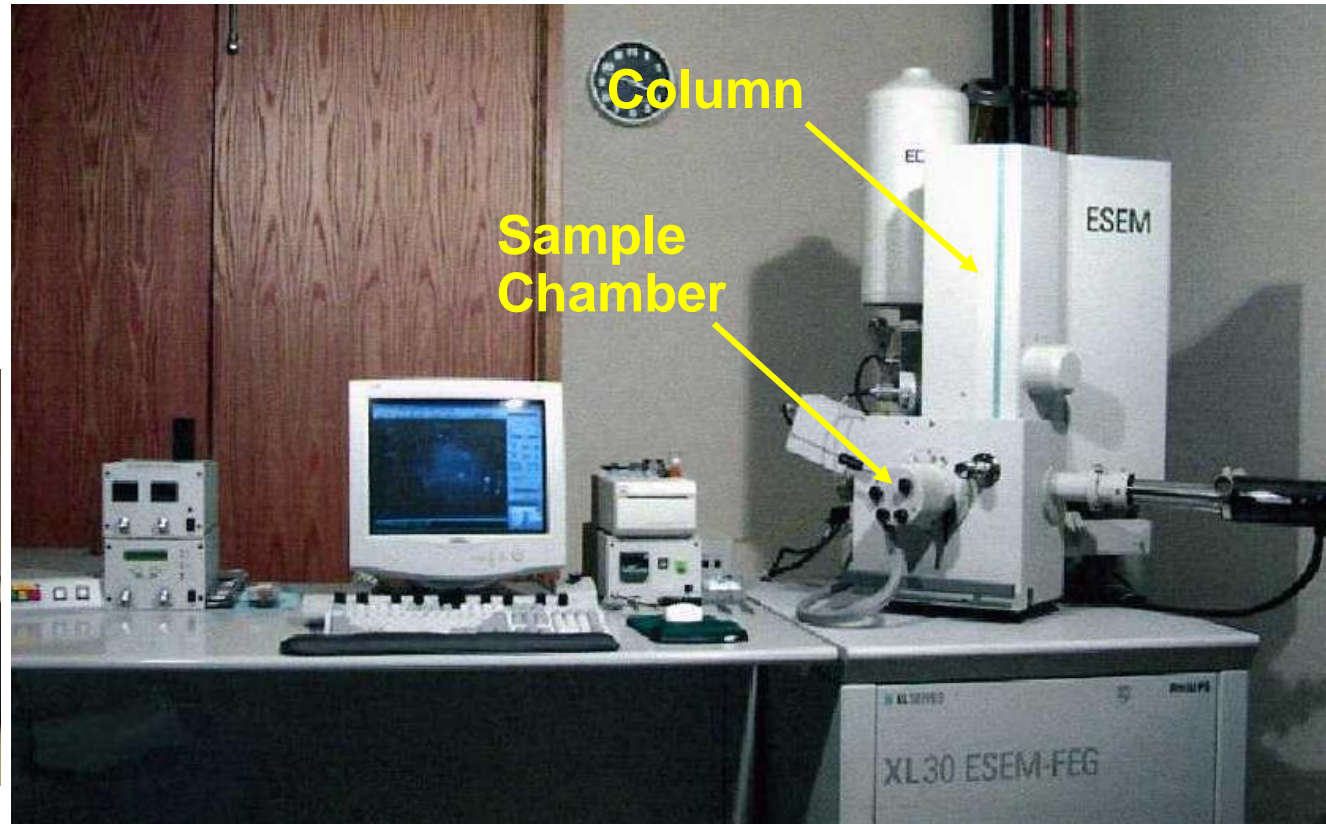
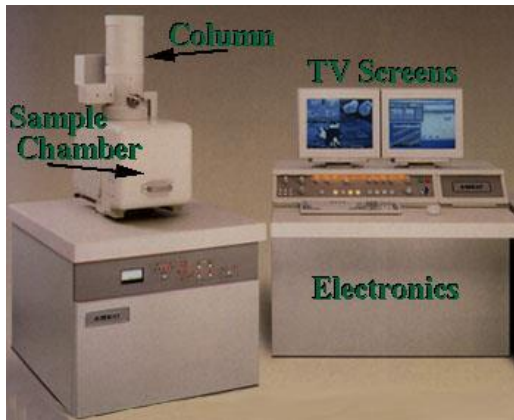
Tel: 3442-7813

Office: P6422

Lecture 3: Outline

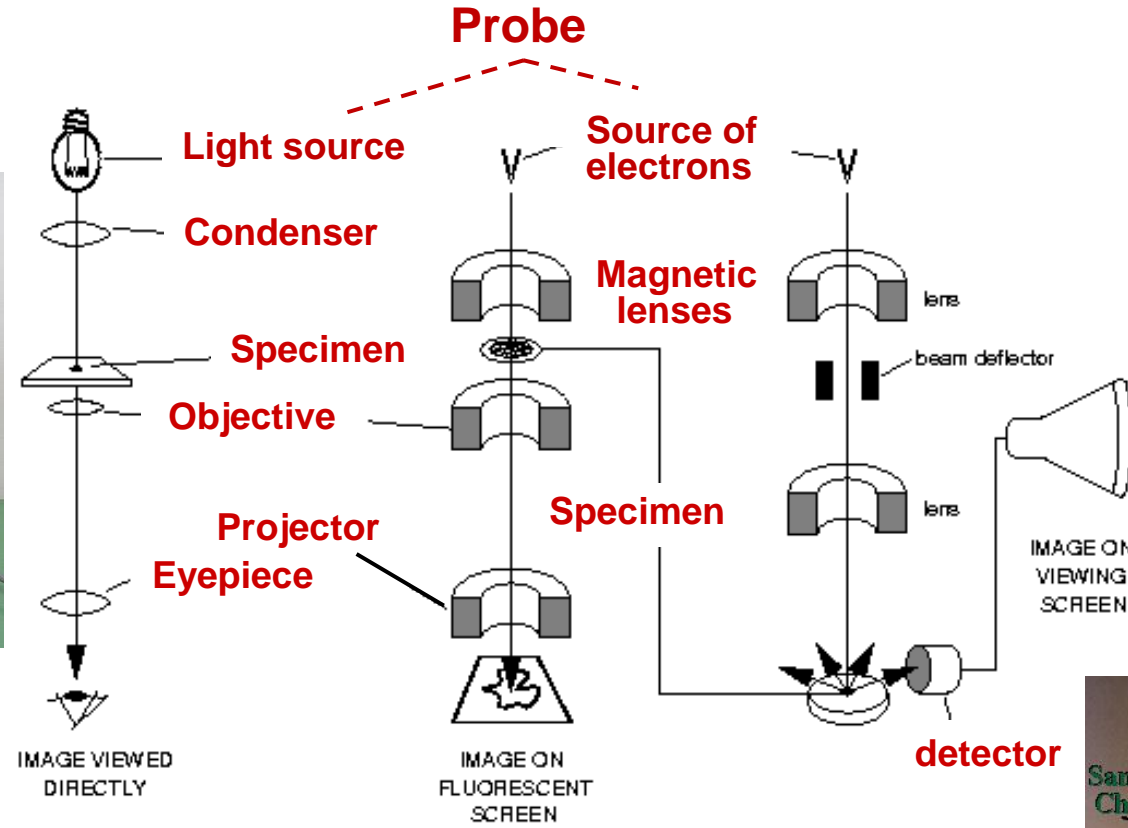
- Introduction
- Major components and functions of a SEM
 - Electron guns
 - Magnetic lenses
 - Scanning coils
 - Electron detectors
- Electron-specimen interactions
 - Secondary and backscattered electrons
 - Interaction and escape volumes
- Magnification and resolution
- Images: effects due to different parameters
 - Acceleration voltage
 - Objective lens aperture
 - Electron probe diameter
 - Sample charging
- Scanning probe microscopy
 - Scanning tunneling microscopy
 - Atomic force microscopy

Scanning electron microscope (SEM)



A scanning electron microscope (SEM) is a microscope that uses **electrons** rather than **light** to form an image. There are many advantages to using the SEM instead of a OM.

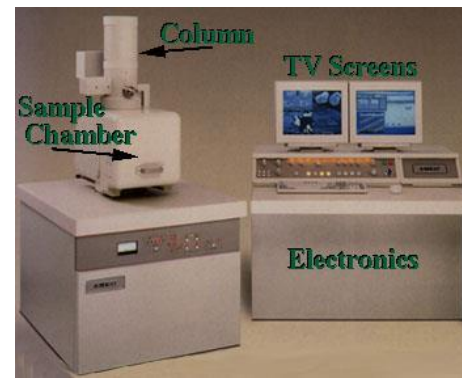
Optical and electron microscopies: a comparison



Optical microscope

Transmission electron microscope (TEM)

scanning electron microscope (TEM)



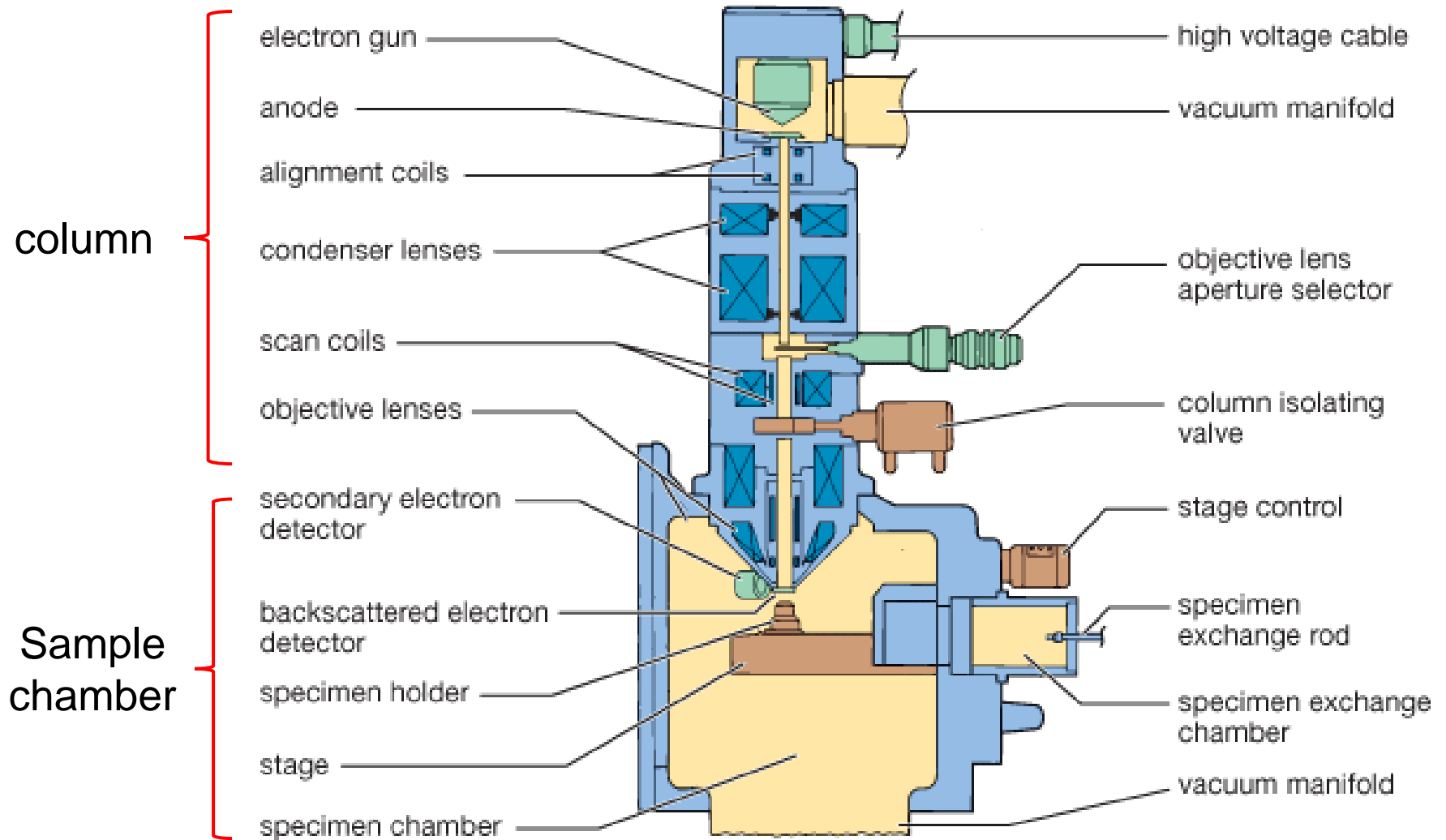
SEM: advantages

	Magnification	Depth of Field	Resolution
OM	4 – 1000x	15.5 – 0.19 μ m	~ 0.2 μ m
SEM	10 – 3000000x	4mm – 0.4 μ m	1-10nm

- SEM has a **large depth of field**
 - allows a large amount of the sample to be in focus at one time and produces an image that is a good representation of the **three-dimensional** sample.
- SEM produces images of **high resolution**, which means that closely features can be examined at a high magnification.
- SEM usually also equipped with **analytical capability**: electron probe microanalysis (energy dispersive x-ray analysis).
- The higher magnification, larger depth of field, greater resolution and compositional and crystallographic information makes the SEM one of the most useful instruments in various fields of research.

Scanning electron microscope

Parts of a scanning electron microscope



Source: JEOL U.S.A., Inc.

© 2011 Encyclopædia Britannica, Inc.

Fundamental properties of electrons

- Electron wavelength: the de Broglie's wavelength of an electron is $\lambda = \frac{h}{p}$, where p is the electron momentum, h is the Planck's constant.
- For an electron accelerated to a kinetic energy of eV, $eV = \frac{em_e v^2}{2}$ and its momentum $p = m_e v = \sqrt{2m_e eV}$; electron wavelength $\lambda(\text{\AA}) = \frac{h}{p} \approx \frac{12.27}{\sqrt{V(\text{volt})}}$
- Considering relativistic effect (e.g. 100 keV electrons will have $v > \frac{1}{2}c$):

$$\lambda(\text{\AA}) \approx \frac{12.27}{\sqrt{V(1 + 0.978 \times 10^{-6}V)}}$$

Accelerating voltage (kV)	Nonrelativistic wavelength (nm)	Relativistic wavelength (nm)	Mass ($\times m_0$)	Velocity ($\times 10^8$ m/s)
100	0.00386	0.00370	1.196	1.644
120	0.00352	0.00335	1.235	1.759
200	0.00273	0.00251	1.391	2.086
300	0.00223	0.00197	1.587	2.330
400	0.00193	0.00164	1.783	2.484
1000	0.00122	0.00087	2.957	2.823

Resolution: electron vs. optical microscopes

$$resolution = \frac{0.6\lambda}{n \sin \alpha}$$

Optical microscope

$$\lambda \sim 0.5 \mu\text{m}$$

$$n = 1.5 \text{ (glass)}$$

$$\alpha \sim 70^\circ$$

$$\text{Resolution} \sim 0.2 \mu\text{m} \text{ or } 2000 \text{ \AA}$$

Electron microscope

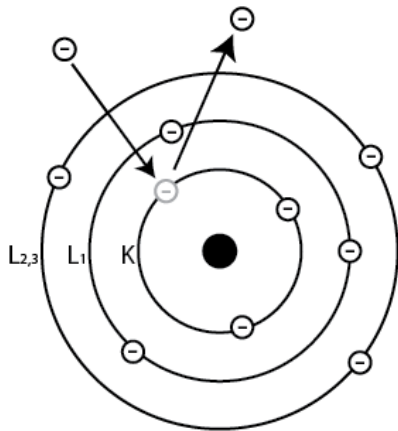
$$\lambda(\text{\AA}) = \frac{h}{p} \approx 0.068 \text{ \AA} \text{ (at 30 kV)}$$

$$n = 1.0 \text{ (vacuum)}$$

$$\alpha \sim 1^\circ$$

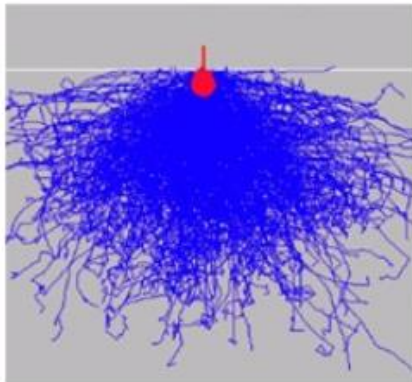
$$\text{Resolution} \sim 4.1 \text{ \AA}$$

Electron-solid interactions

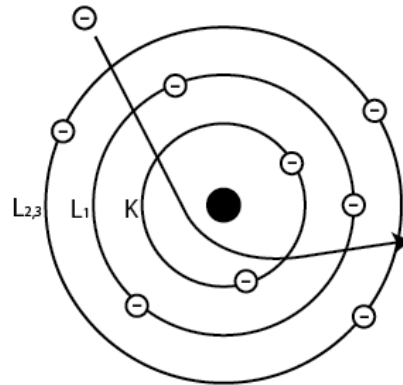


Secondary electron region for imaging

(SE is actually produced wherever primary electron goes, but only those near surface can go to SE detector for imaging)

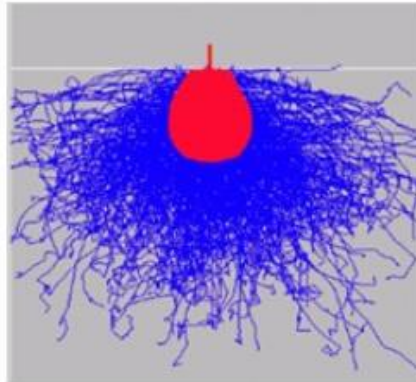


Best spatial resolution for SEM

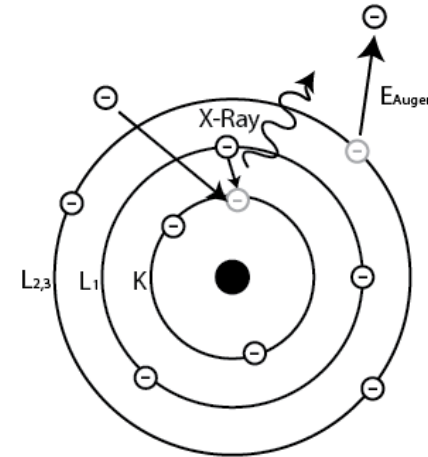


Back-scattered electron region for imaging

(Again, BSE is actually produced wherever primary electron goes. It has higher energy, so travel longer than SE)

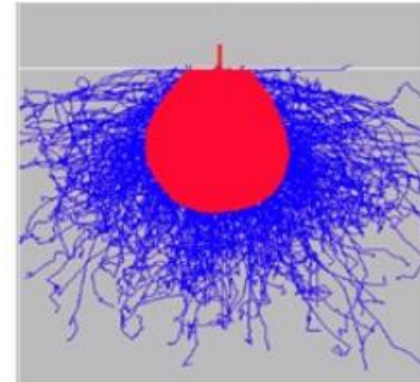


Better Z contrast for SEM
(brighter for higher Z)



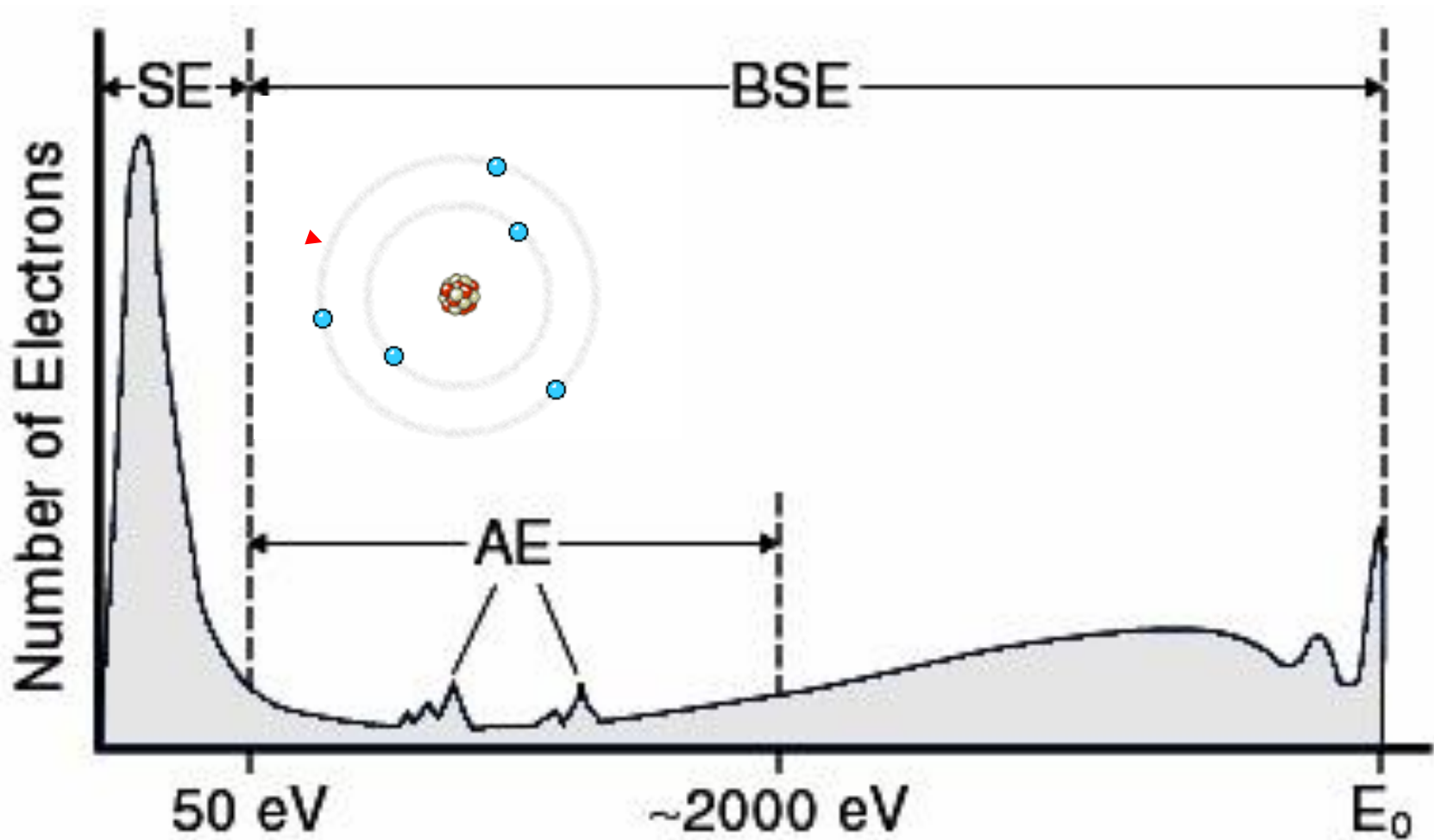
X-ray region for EDX

(x-ray is actually produced wherever primary electron goes. It travels farther than BSE)



Best analytical for EDX

Electron-solid interactions



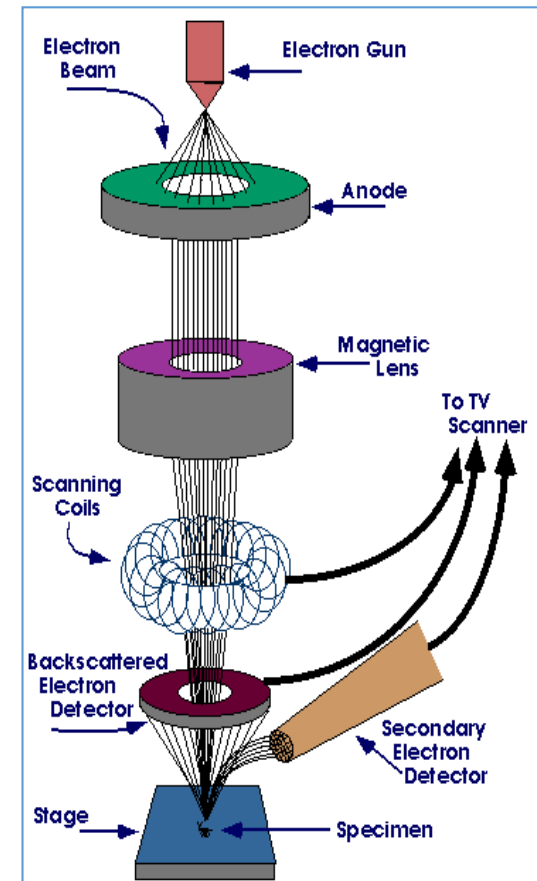
SEM: an overview

■ The column

- An **electron beam** is produced by the electron gun (thermionic or field emission) and accelerated to the desired voltage (typically 0.2-40 kV).
- The beam is focused by one or two condenser lenses to a spot of **~0.4 to 5 nm**
- The electron beam is deflected by pairs of scanning coils, **rastering** the beam in the x- and y-directions

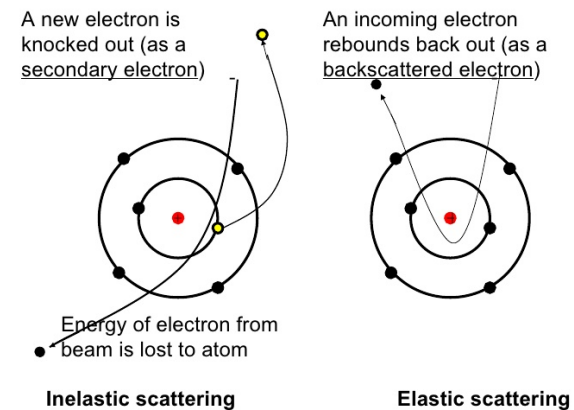
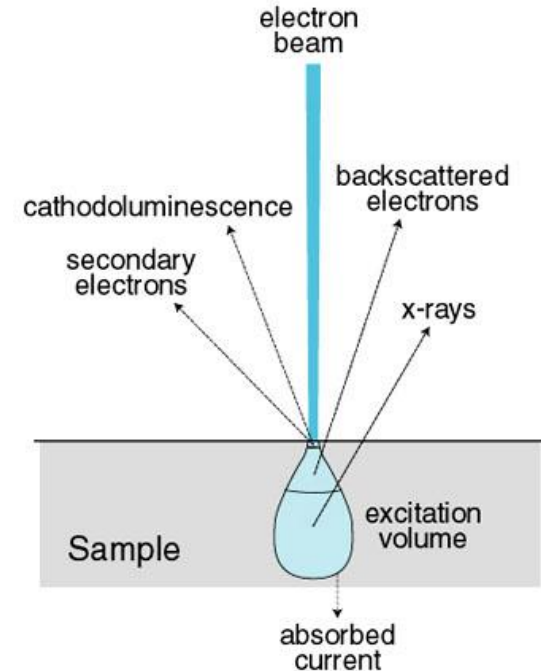
■ The sample chamber

- Specimens must be of an appropriate size to fit in the specimen chamber and are generally mounted rigidly on a specimen holder.
- Specimens must be **electrically conductive** to prevent the accumulation of electrostatic charge at the surface.
- Nonconductive specimens tend to charge when scanned by the electron beam, and are usually coated with an ultrathin coating of electrically conducting material (Au, Al)



SEM: an overview

- Electron-atom interaction: when the primary electron beam interacts with the sample, the electrons lose energy by repeated random scattering (elastic or inelastic) and absorption within a **teardrop-shaped** volume of the specimen (the interaction volume), extending from less than 100 nm to $\sim 5 \mu\text{m}$ into the surface.
 - Inelastic collision** with electrons: energy is transferred to the other electron and if the energy transferred exceeds the work function a **secondary electron (SE)** will be emitted with energy typically $< 50 \text{ eV}$ (within a few nm of the solid).
 - Elastic scattering** with atomic nuclei: primary electrons elastically scattered by nuclei. Most **backscattered electrons (BSEs)** have energies comparable to the primary electron energy ($\gg 50 \text{ eV}$).

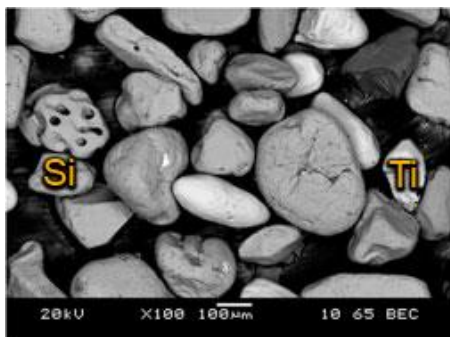


SEM: an overview

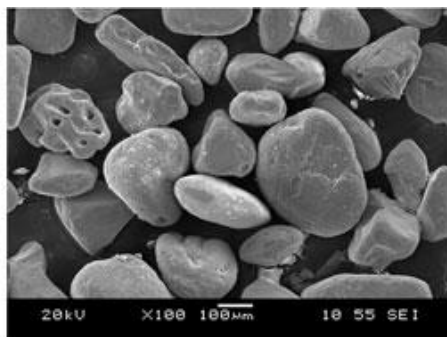
Image formation: SEM images can be formed by either the SEs or BSEs

- **SE image:** due to their low energy (<50 eV), these electrons originate within a **few nanometers** from the sample surface.
 - The SEs are detected by an **Everhart-Thornley detector** (a scintillator-photomultiplier system).
 - The amplified signal is displayed as a 2-D intensity distribution that can be viewed and eventually saved as a digital image.
 - The brightness of the signal depends on the number of secondary electrons reaching the detector.
 - Using the signal of SEs, image resolution less than 0.5 nm is possible.
- **BES image:** dedicated BSE detectors are positioned above the sample in a "doughnut" type arrangement, concentric with the electron beam, maximizing the solid angle of collection.
 - BSE detectors are usually either of scintillator or of semiconductor types.
 - Since heavy elements backscatter electrons more strongly than light elements, and thus appear brighter in the image, BSE are used to **detect contrast between areas with different chemical compositions.**
- Other types of images: signals from **cathodoluminescence** and x-ray microanalysis can also be used to form images giving different information of the specimen

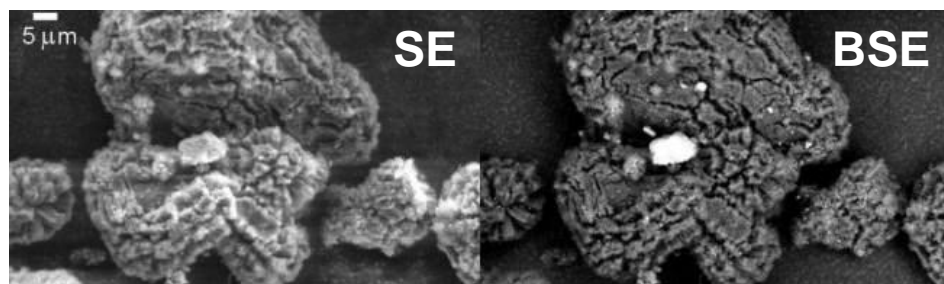
SEM: images



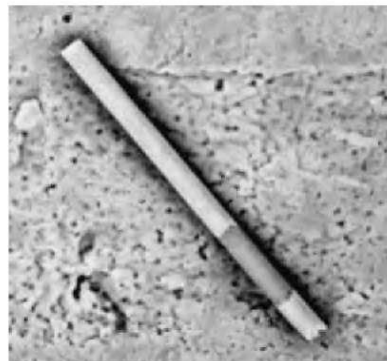
Backscattered electron image (BSE)



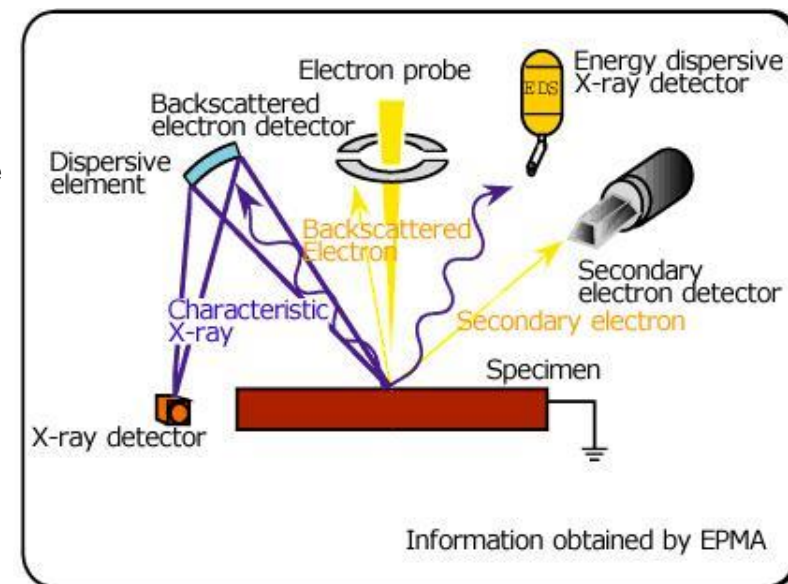
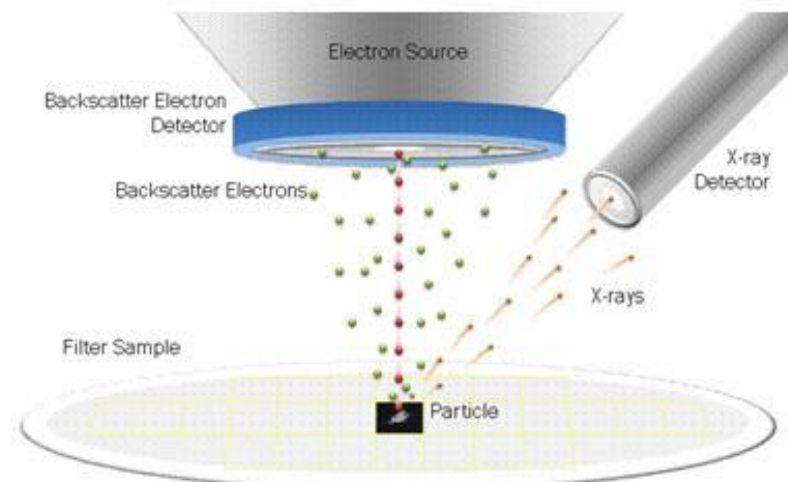
Secondary electron image (SE)



SEM images of Fe particles in carbon obtained with secondary electrons (left) and back-scattered electrons (right). The BSE image shows the Fe particles with bright contrast



Field emission SEM image of a AuAgAu nanowire, acquired with backscattered electron detection. The Ag segment appears darker as compared to the Au



SEM: the interior

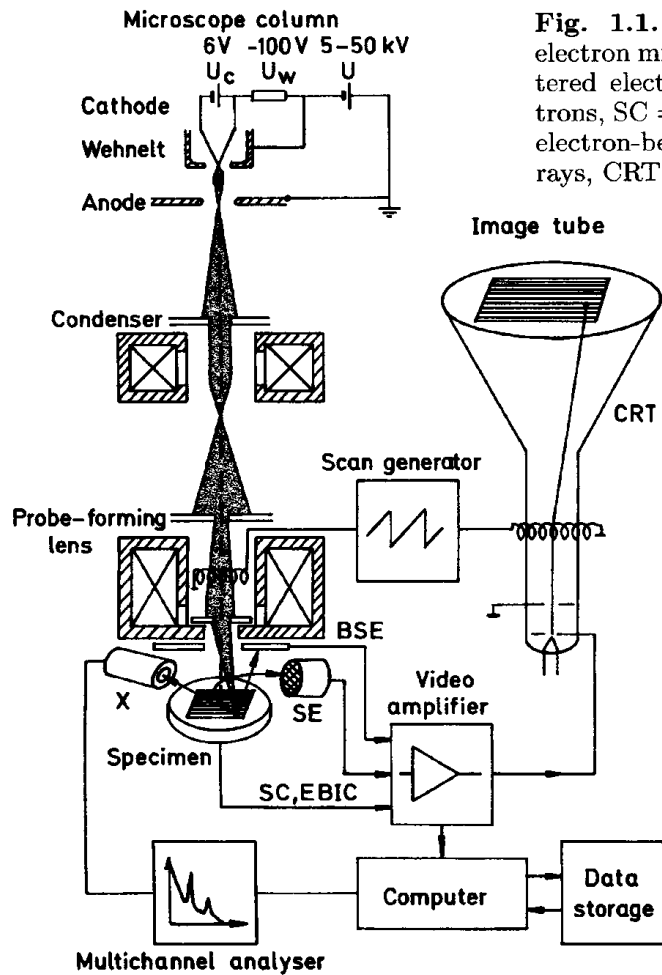
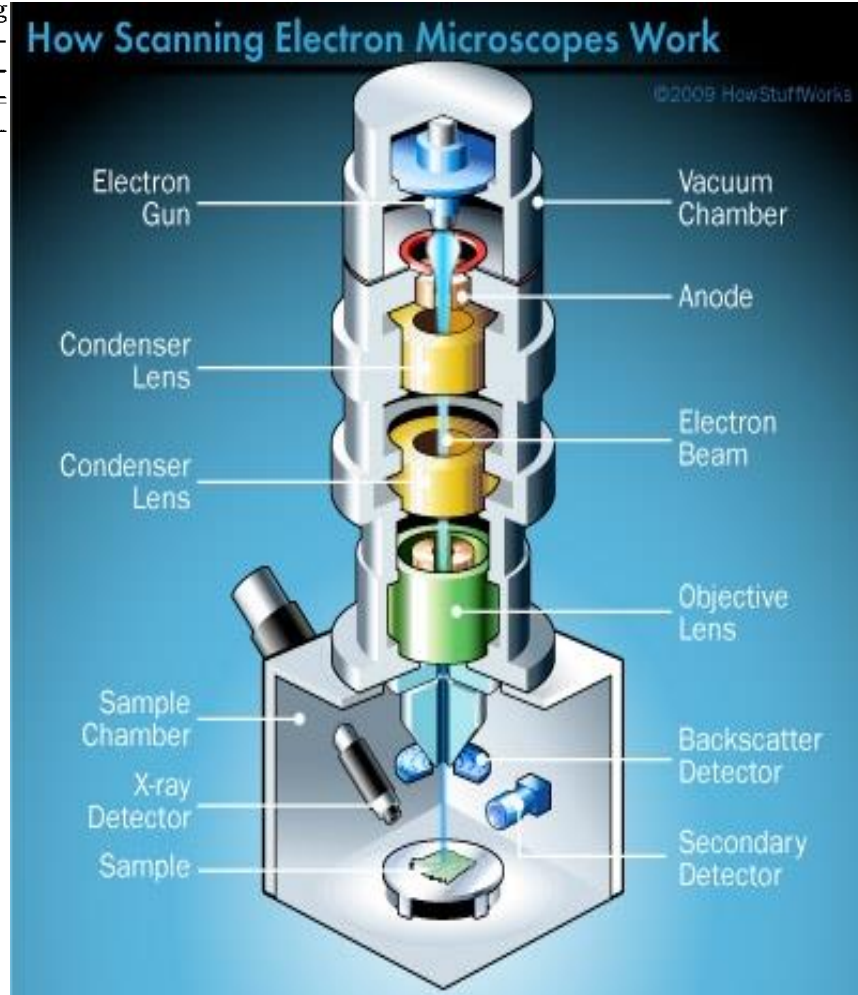


Fig. 1.1. Principle of the scanning electron microscope (BSE = backscattered electrons, SE = secondary electrons, SC = specimen current, EBIC = electron-beam-induced current, X = x-rays, CRT = cathode-ray tube)

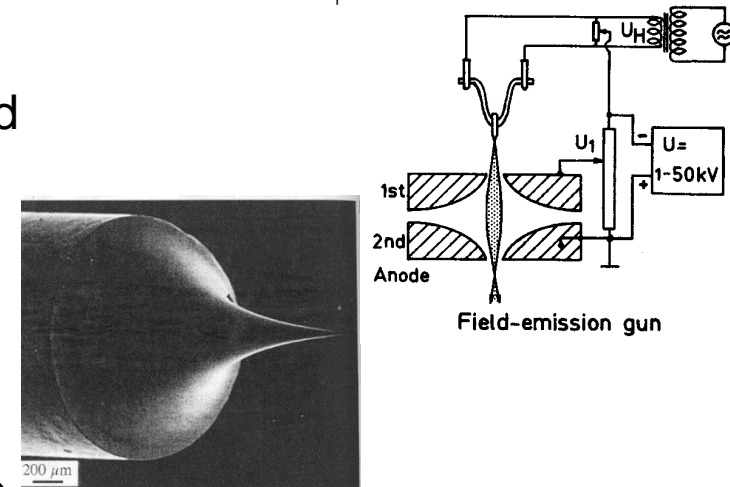
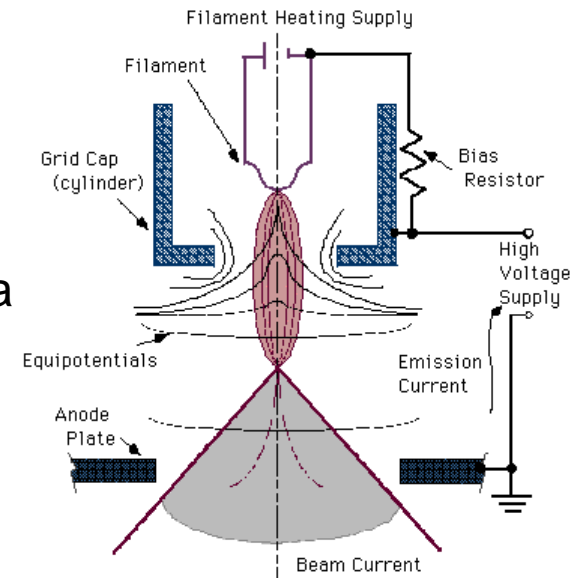


L. Reimer, "Scanning Electron Microscope", 2nd Ed., Springer-Verlag, 1998, p.2

SEM: electron gun

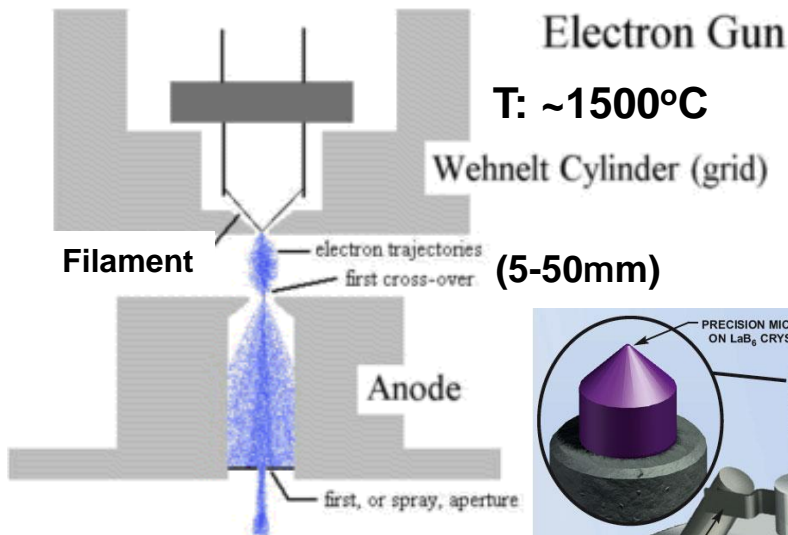
Electron guns are used to produce a fine, controlled beam of electrons which are then focused at the specimen surface.

- Thermionic emission gun:
 - A tungsten filament heated by DC to approximately 2700K or LaB₆ rod heated to around 2000K in a vacuum of 10⁻³ Pa (10⁻⁴ Pa for LaB₆), producing flux of electrons
 - Electrons are accelerated by an acceleration voltage of 1-50kV
- Field Emission Gun:
 - The tip of a tungsten needle is made very sharp (radius < 0.1 μm) so that electric field becomes very strong (> 10⁷ V/cm) and electrons emitted due to field emission
 - Ultra-high vacuum (<10⁻⁶ Pa) is needed to avoid ion bombardment to the tip from the residual gas.
 - Electron probe diameter < 1 nm is possible

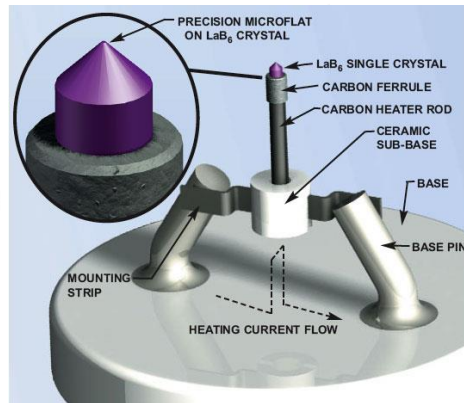


SEM: electron gun

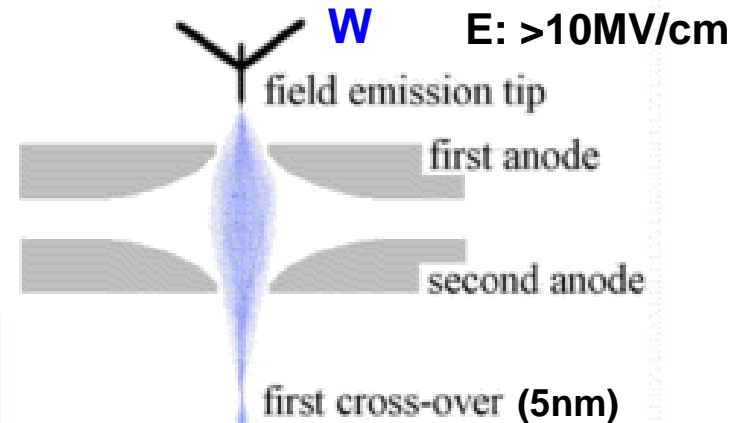
Thermionic Gun



W and LaB₆



Field Emission Gun



Cold- and thermal FEG

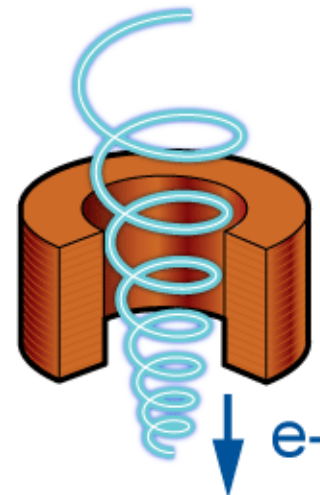
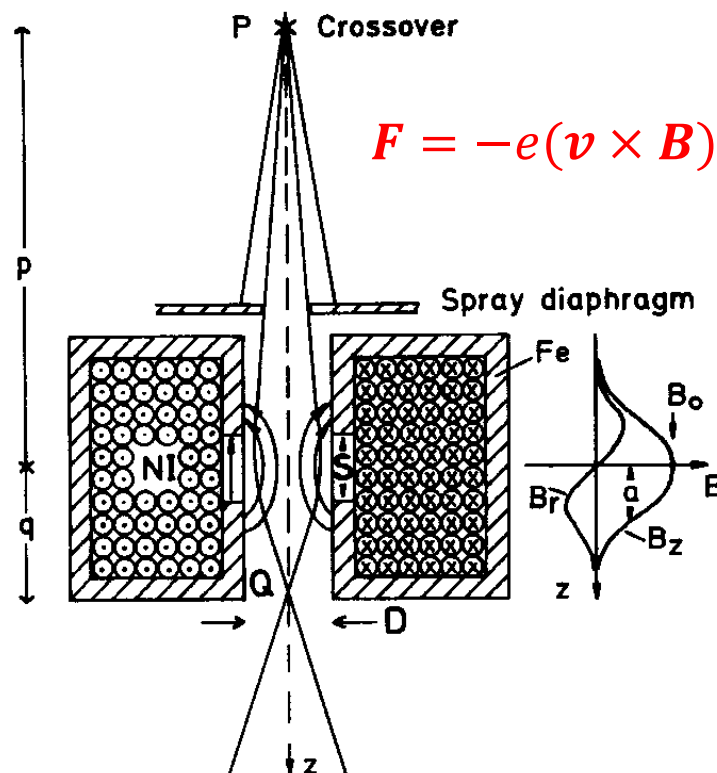


Source	Brightness	Stability (%)	Size	Energy spread (eV)	Vacuum (Torr)
W	3×10^5	~1	50 μm	3.0	10^{-5}
LaB ₆	3×10^6	~2	5 μm	1.5	10^{-6}
C-FEG	10^9	~5	5 nm	0.3	10^{-10}
T-FEG	10^9	<1	20 nm	0.7	10^{-9}

Brightness – beam current density per unit solid angle

SEM components: magnetic lenses

A magnetic lens is a solenoid designed to produce a specific magnetic flux distribution. Magnetic lenses are used for the focusing or deflection of moving electrons. They operate by use of the **magnetic Lorentz force**. Their strength can often be varied by usage of electromagnets.



Electromagnetic lenses create a circular magnetic field that **demagnify** (condense) the electron beam as it passes through.

Lens formula: $1/f = 1/p + 1/q$

$$f \propto B_0^2$$

f can be adjusted by changing B_0 , i.e. changing the current through coil.

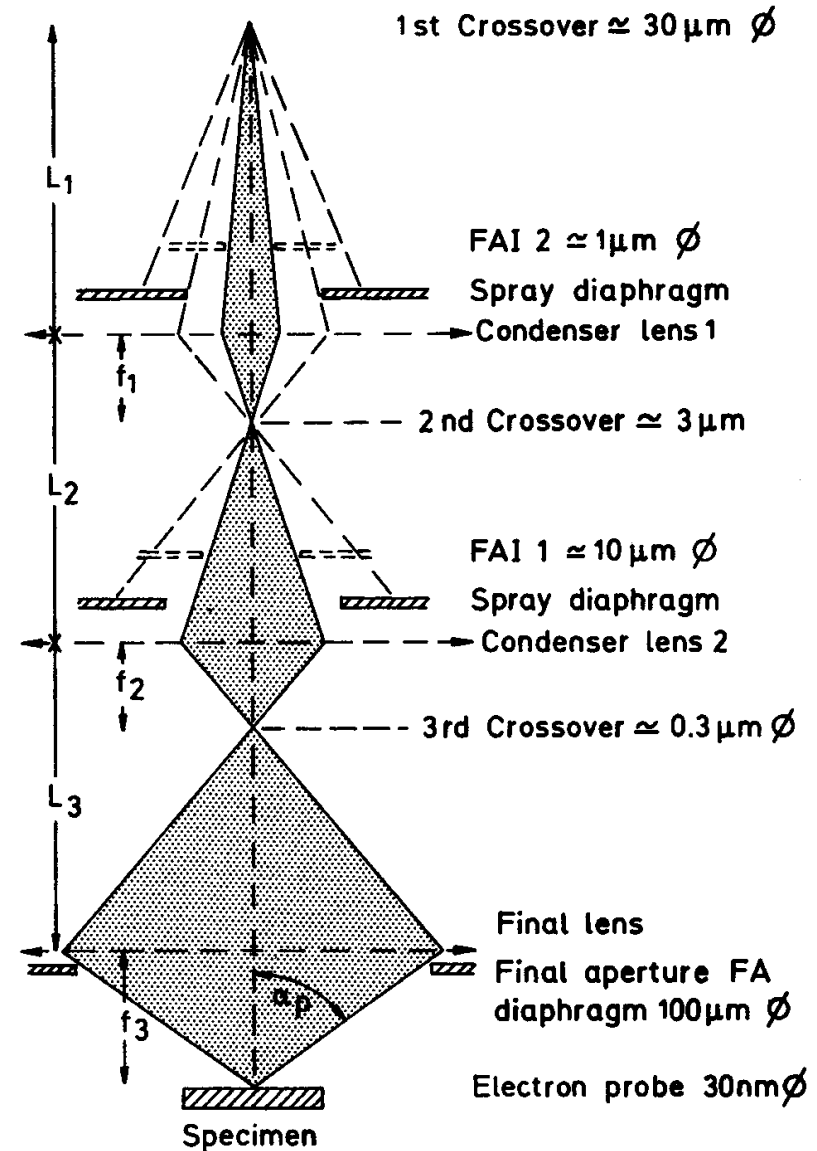
SEM components: condenser

Condenser lens – focusing

- Controls the spot size and convergence of the electron beam which impinges on the sample.
- For a thermionic gun, the diameter of the first **cross-over point** $\sim 20\text{-}50\mu\text{m}$
- To focus the beam to $< 10\text{ nm}$ on the specimen surface, the magnification should be $\sim 1/5000$, which is not easily attained with one lens (say, the objective lens) only.
- Therefore, condenser lenses are added to **demagnify** the cross-over points.

Demagnification:

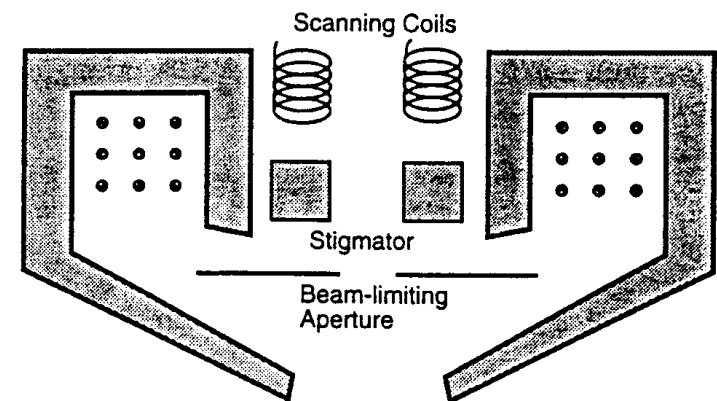
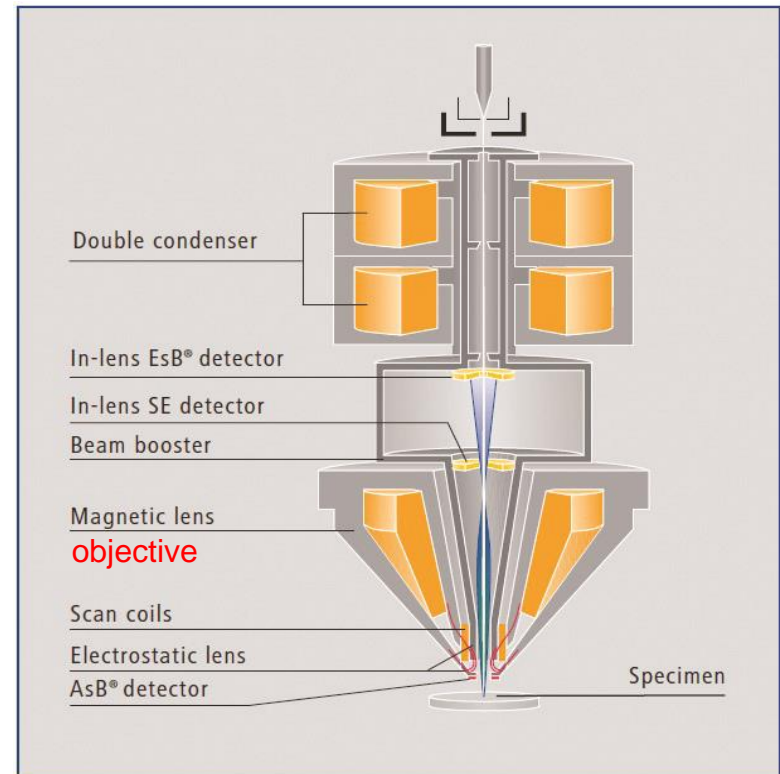
$$M = f/L$$



SEM components: objective

Objective lens – final probe forming

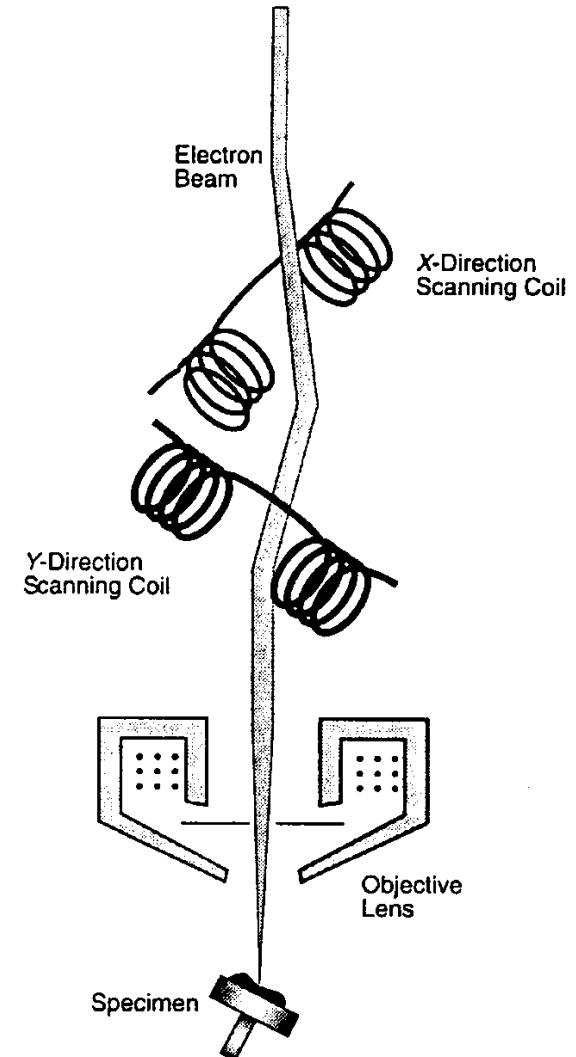
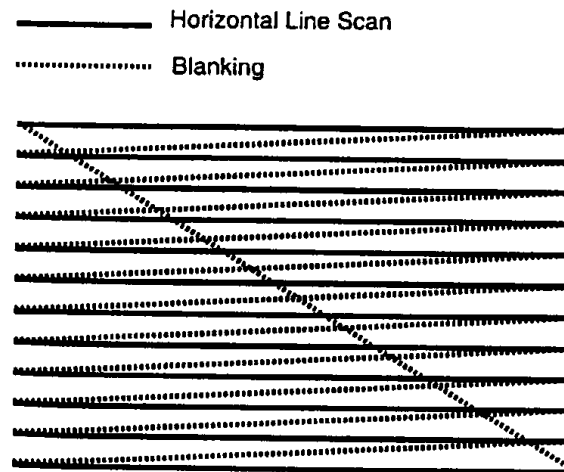
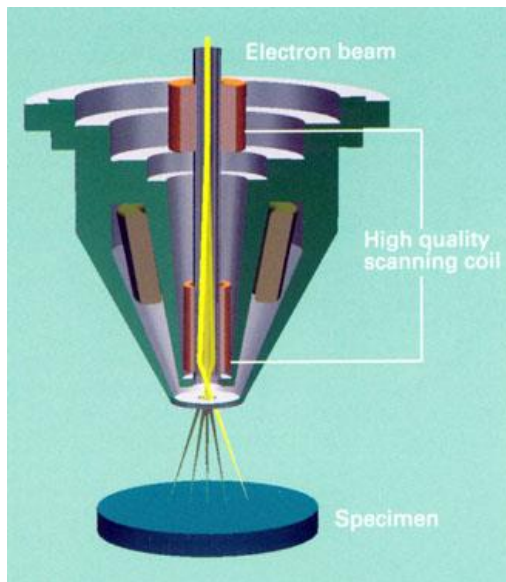
- Controls the **final focus** of the electron beam by changing the magnetic field strength
- Since the electrons coming from the electron gun have **spread in kinetic energies and directions of movement**, they may not be focused to the same plane to form a sharp spot.
- By inserting an **aperture**, the stray electrons are blocked and the remaining narrow beam will come to a narrow
- The cross-over image is finally demagnified to an **~10nm** beam spot which carries a beam current of approximately **10^{-9} - 10^{-12} A**.



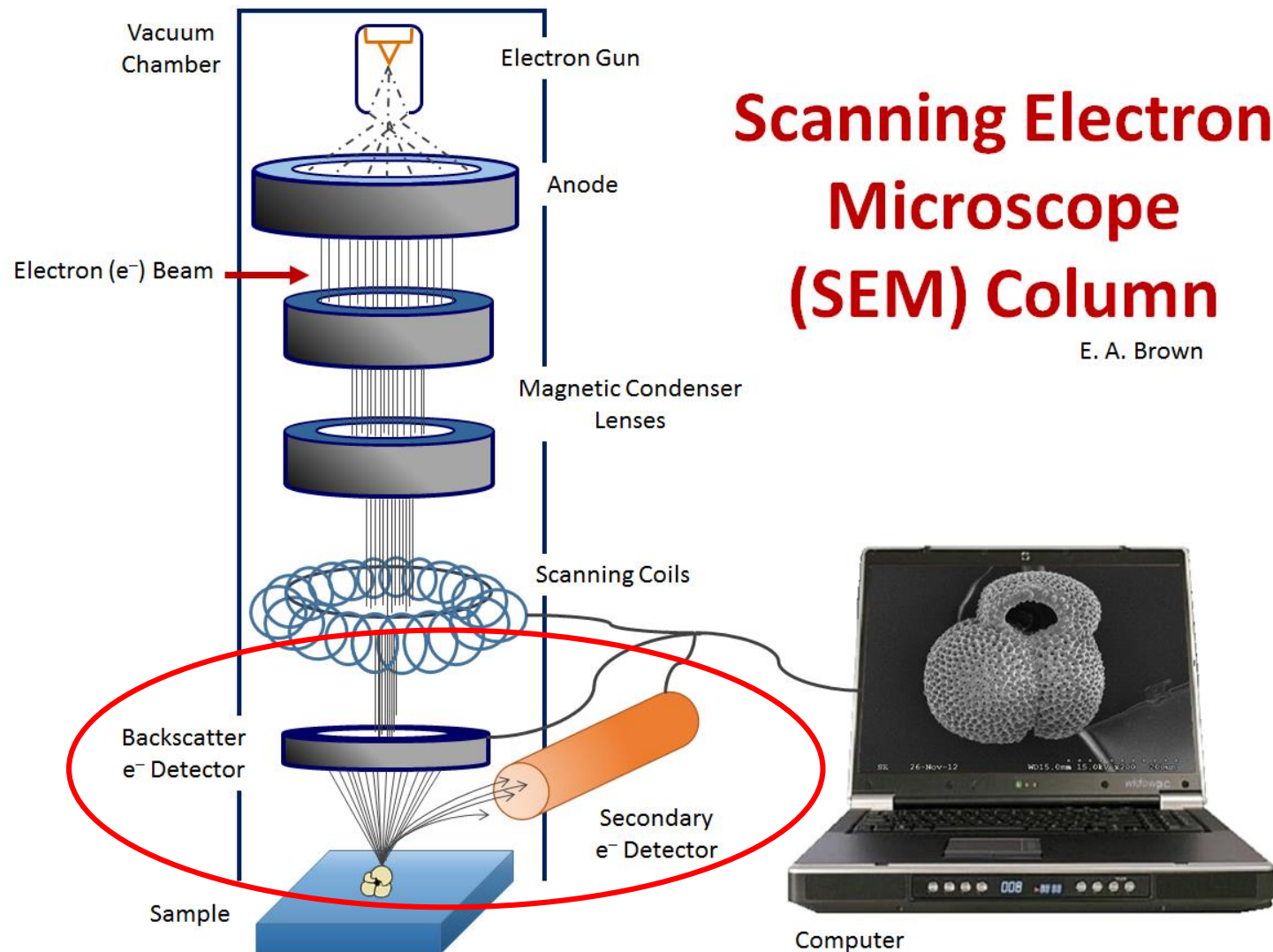
SEM components: scanning coils

Scanning coils in a SEM are used to raster the beam across the sample for textural imaging

- Two sets of coils are used for scanning the electron beam across the specimen surface in a **raster** fashion over a rectangular area of the sample surface.
- This effectively samples the specimen surface **point by point** over the scanned area.

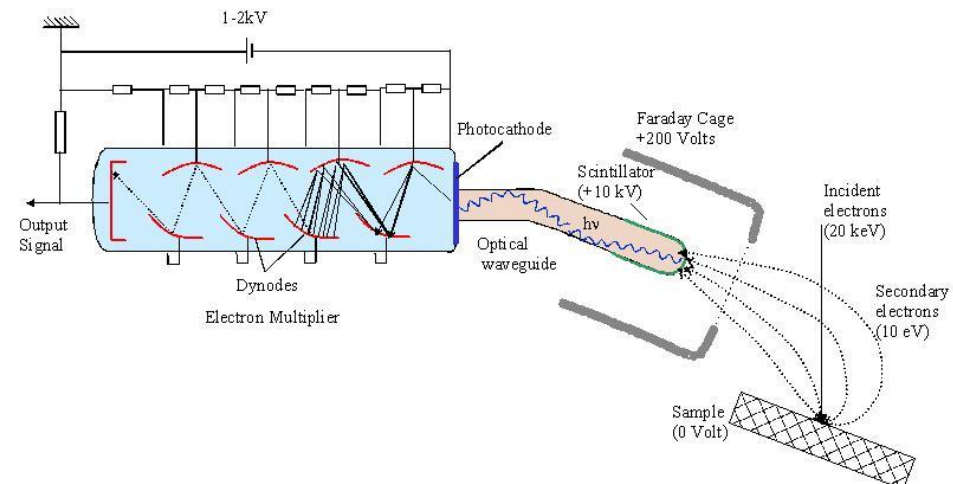
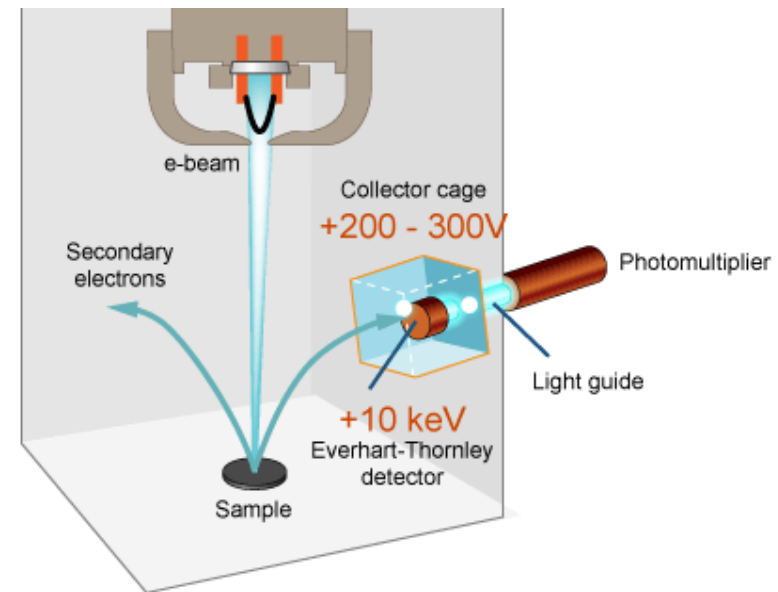


SEM components: electron detectors



SEM components: Everhart-Thornley detector

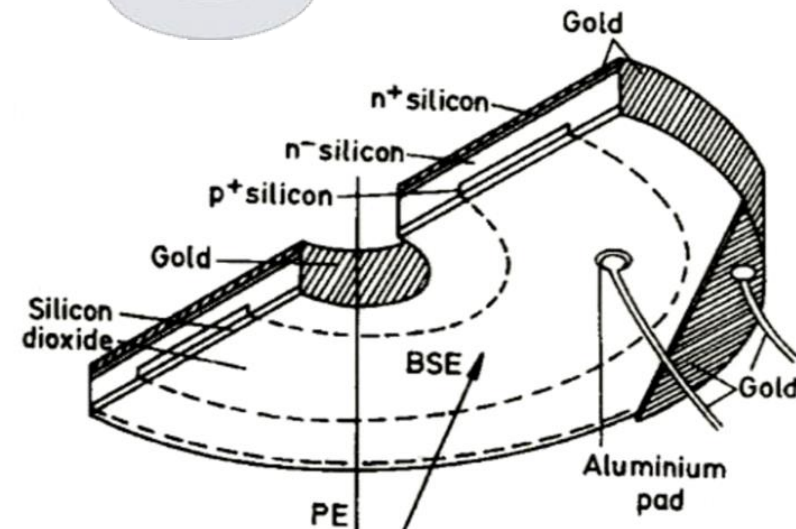
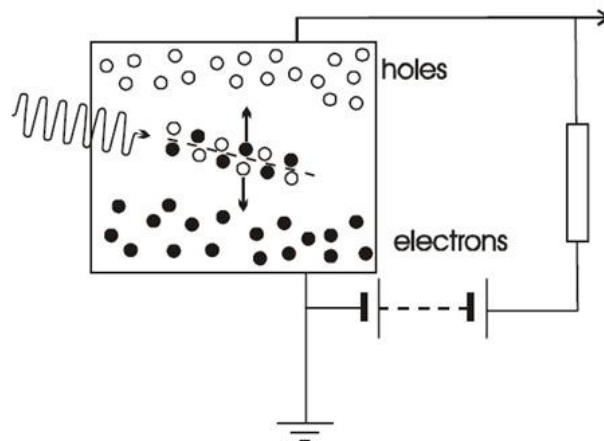
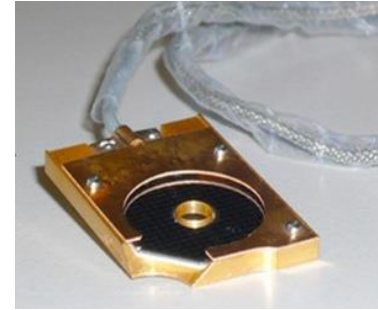
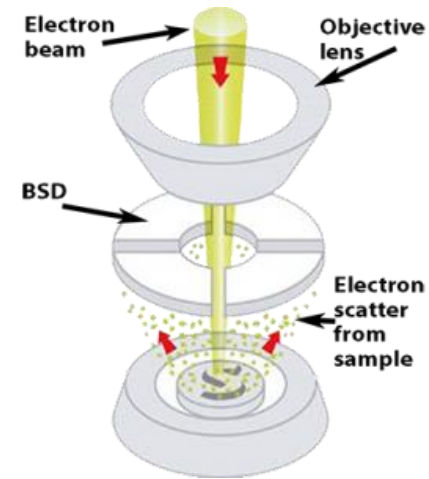
- A Everhart-Thornley Detector (E-T detector or ET detector) consists primarily of a **scintillator** inside a **Faraday cage** inside the specimen chamber of the microscope
- A low positive voltage is applied to the Faraday cage to attract the relatively low energy (<50 eV) secondary electrons.
- The scintillator has a high positive voltage (~10 kV) to accelerate the incoming electrons to it where they can be converted to **light photons**
- Can be used in backscattered electron (>50 eV) mode by either turning off the Faraday cage or by applying a negative voltage to the Faraday cage



SEM components: backscattered electron detector

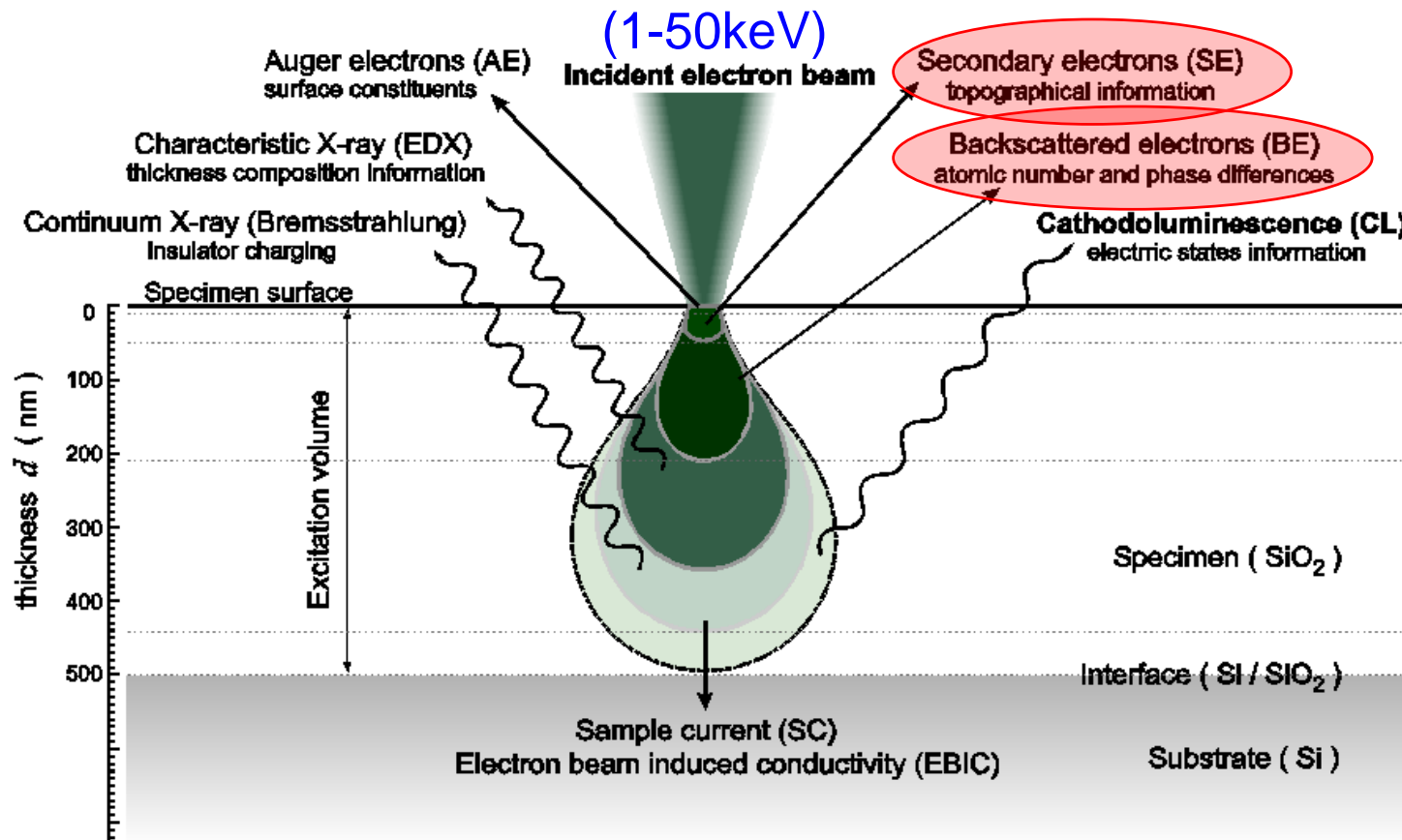
Backscattered electrons have energies close to the primary beam energy and are detected by semiconductor detectors

- High energy (>50 eV to keV) backscattered electrons (BSEs) incident on the detector creating electron-hole pairs in the bulk of the semiconductor.
- The $e - h$ pairs are separated and swept to opposite poles by an applied voltage, creating a current
- The number of $e - h$ pairs are proportional to the energy of the electrons



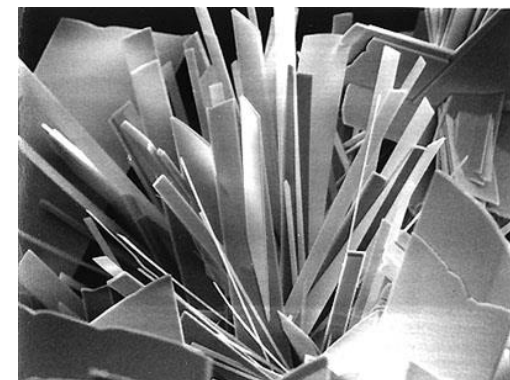
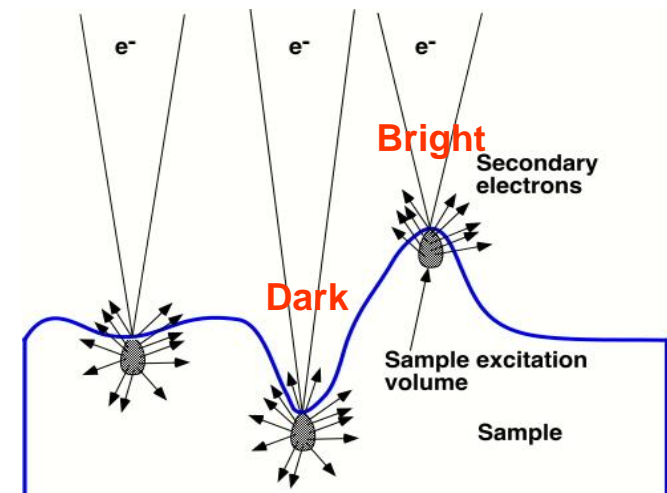
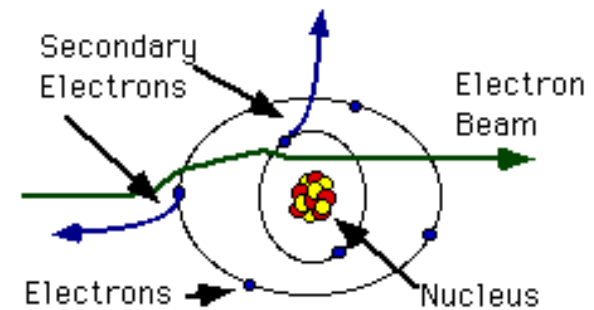
Electron beam microscopy/spectroscopy

The types of signals produced by a SEM include secondary electrons (SEs), back-scattered electrons (BSEs), characteristic X-rays and photons (cathodoluminescence) (CL), absorbed current (specimen current) and transmitted electrons. Both SEs and BSEs are used for imaging

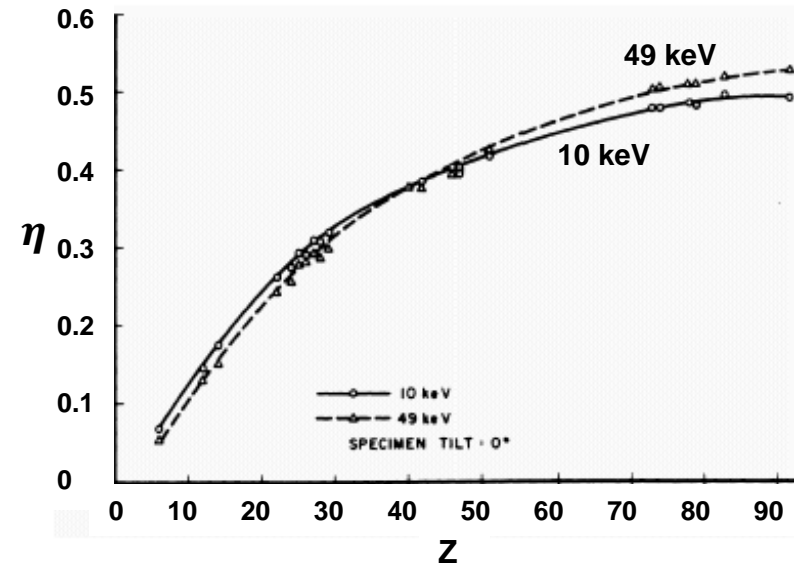
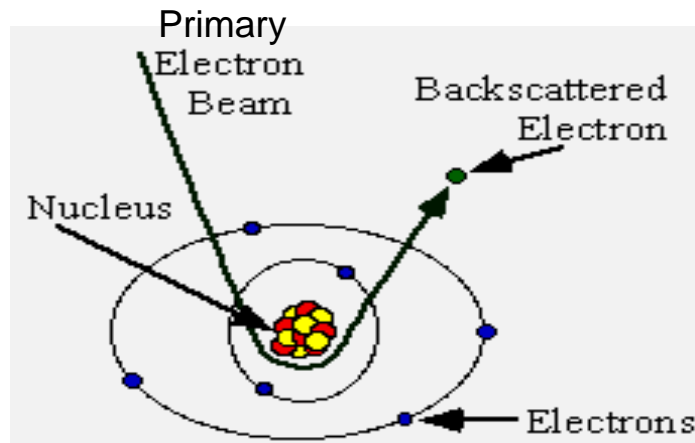


Secondary electrons (SEs)

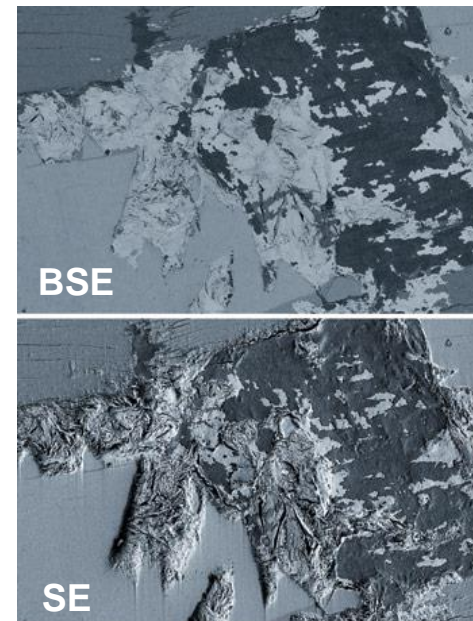
- Produced by **inelastic** interactions of high energy electrons with core electrons (K, L M shells) of atoms in the specimen, causing the ejection of the electrons from the atoms. T
- These ejected electrons have energies **<50eV**.
- SE yield: $\delta = n_{SE}/n_B > 1$ independent of Z
- δ decreases with increasing beam energy and increases with decreasing glancing angle of incident beam
- Due to their low energy, only SE that are **very near the surface** (<10nm) can exit the sample and be examined (small escape depth).
- SE generation depend on the angle of incidence, thus local variations in the angle of the surface to the beam (roughness) affects the numbers of electrons leaving from point to point. This gives rise to **topographic contrast** of the specimen



Backscattered Electrons (BSEs)



- **BSE** are produced by **elastic interactions** (scatterings) of electrons with nuclei of atoms in the specimen and they have **high energy** and **large escape depth**.
- **BSE yield**: $\eta = n_{BS}/n_B \sim$ increases with atomic number, **Z**
- BSE images show characteristics of **atomic number contrast**, i.e., high average Z appear brighter than those of low average Z .
- η increases with tilt giving rise to **topological contrast** (but not as well as SEs).

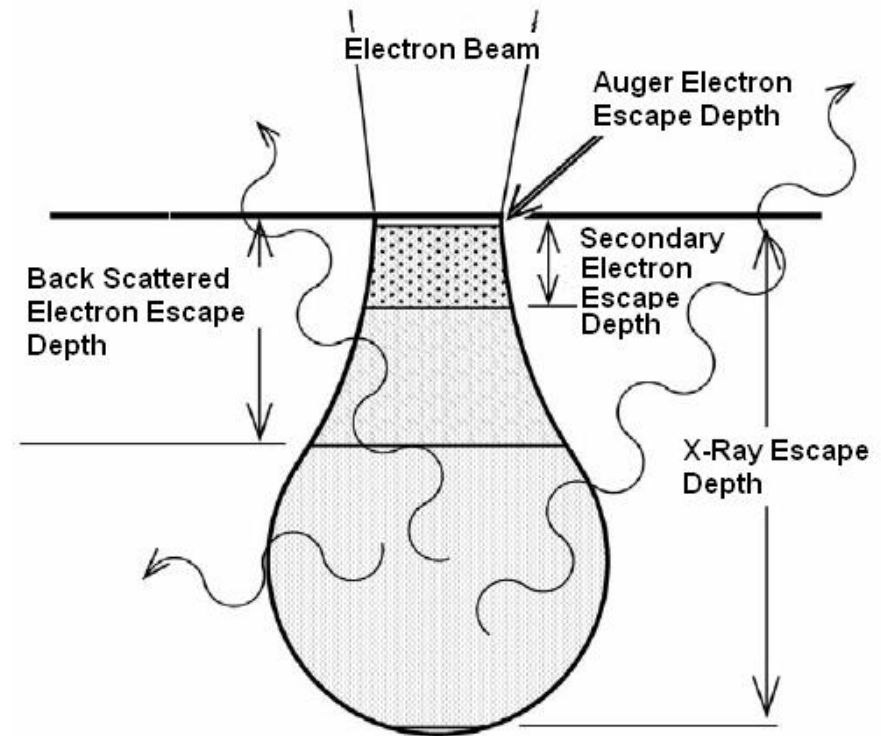


SEM images of a geological material taken with 750 V electrons. The BSE image displays minimum topography but enhanced Z-contrast

Interaction and escape volume

The combined effect of the elastic and inelastic interactions is to distribute the beam electrons over a three-dimensional **interaction volume**. The actual dimensions and shape of the interaction volume are dependent upon a number of parameters: accelerating voltage, atomic number and tilt.

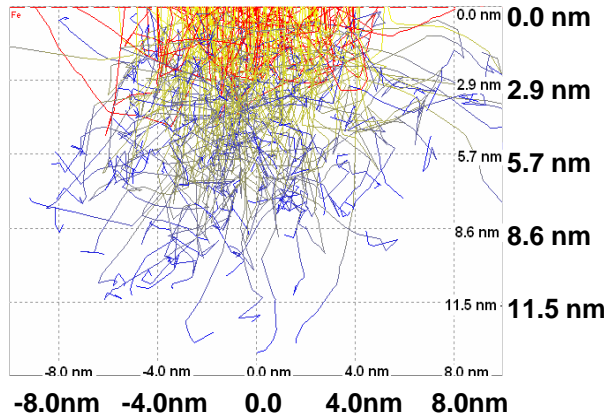
- The volume responsible for the respective signal is called the **escape volume** of that signal
- If the diameter of primary electron beam is $\sim 5\text{nm}$:
 - **Secondary electron:**
diameter $\sim 10\text{nm}$; depth $\sim 10\text{nm}$
 - **Backscattered electron:**
diameter $\sim 1\mu\text{m}$; depth $\sim 1\mu\text{m}$
 - **X-ray:** from the whole interaction volume, i.e., $\sim 5\mu\text{m}$ in diameter and depth



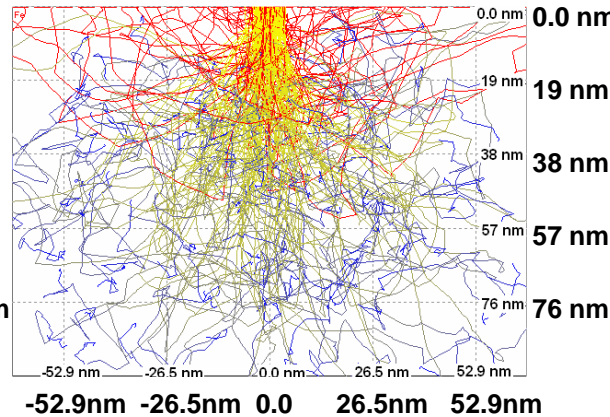
Interaction volume

The interaction volume increases while the probability of elastic scattering decrease with **accelerating voltage**

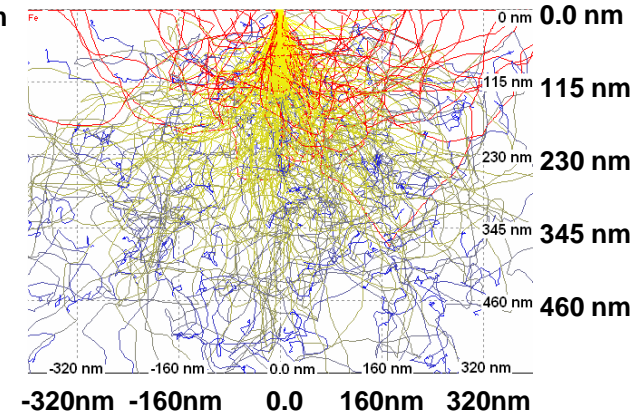
1 kV



5 kV

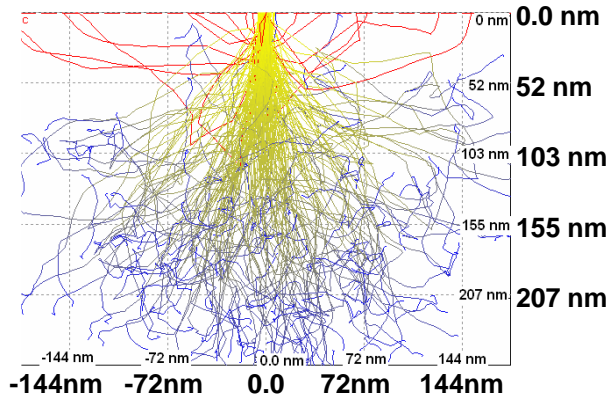


15 kV in Fe

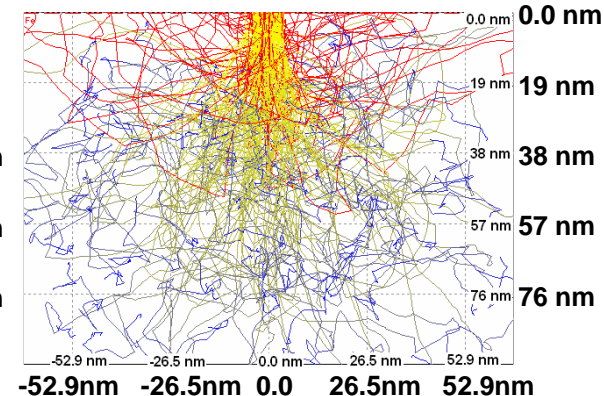


The interaction volume decreases while the probability of elastic scattering increases with higher **atomic number** elements.

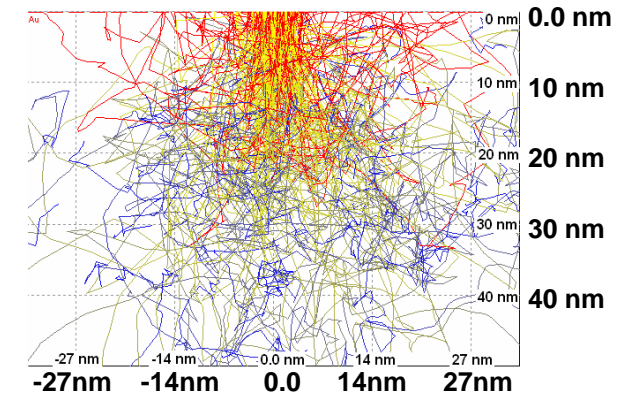
5 kV in Carbon



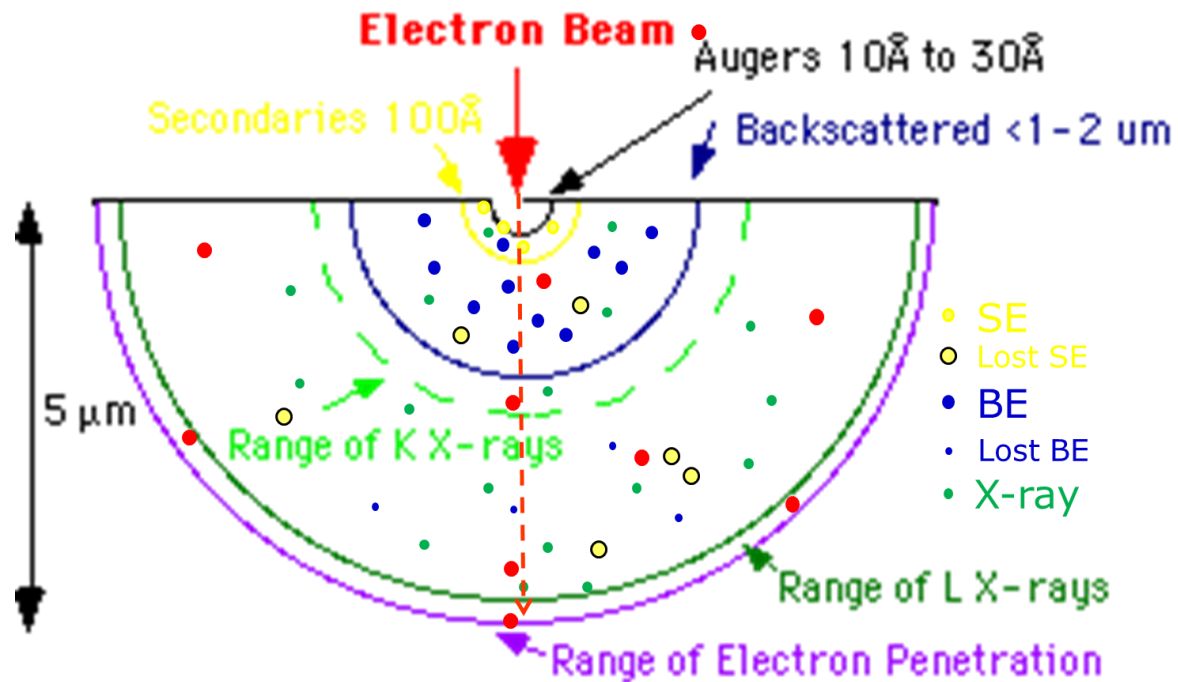
Iron



Gold



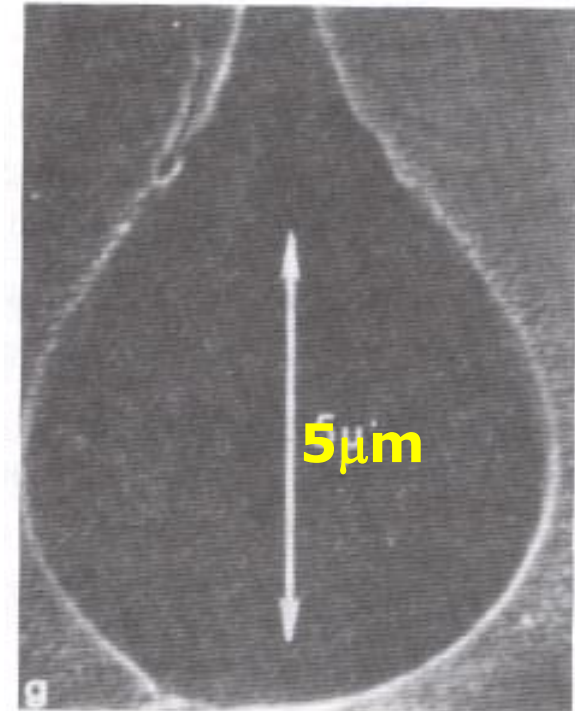
Electron interaction volume



At 20 KV Accelerating Voltage and $Z=28$

Schematic illustration of electron beam interaction in Ni

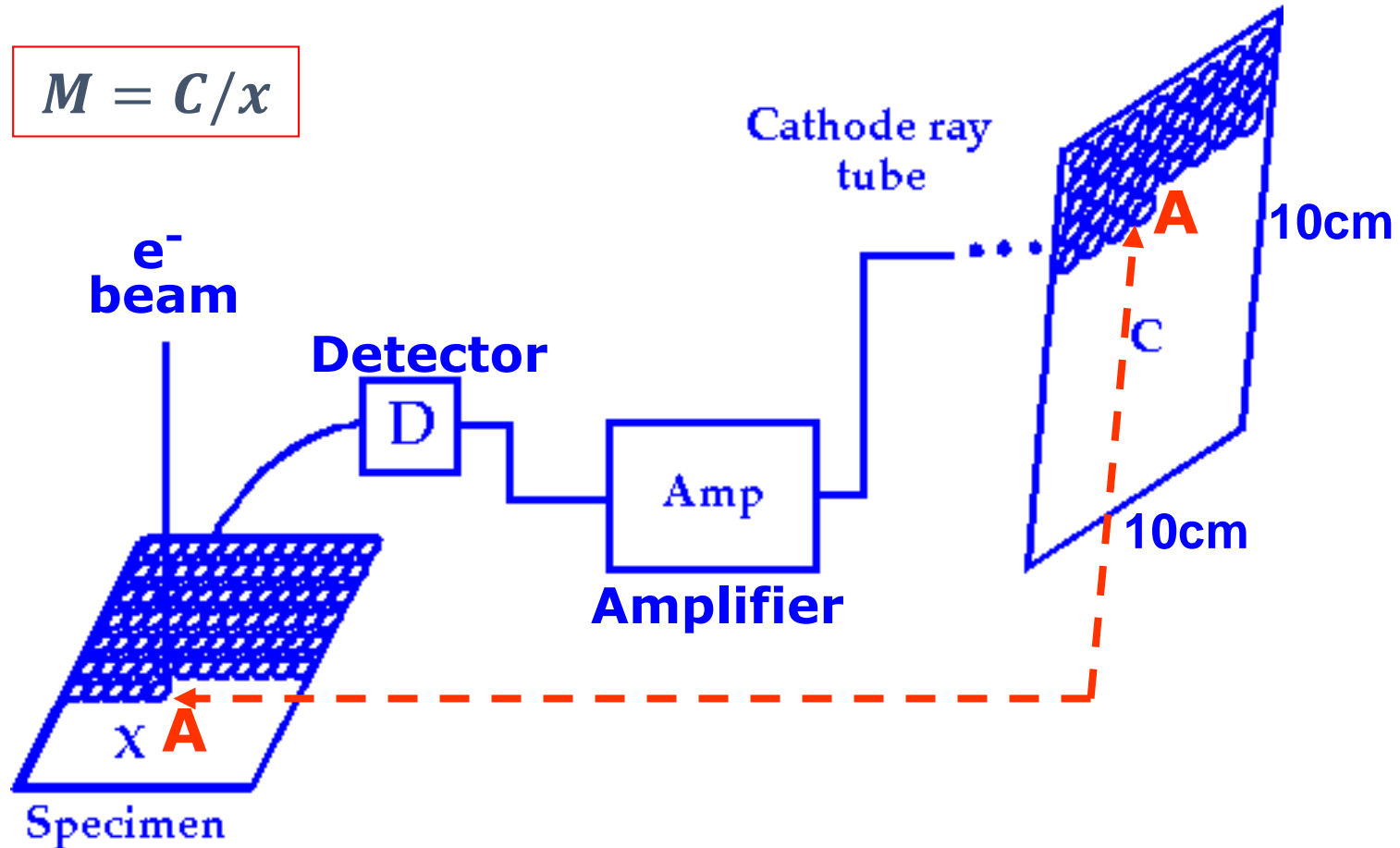
Pear shape



Electron interaction volume in polymethylmethacrylate (plastic-a low Z matrix) is indirectly revealed by etching

Image Formation & Magnification in SEM

$$M = C/x$$



- Beam is scanned over specimen in a raster pattern in synchronization with beam in CRT.
- Intensity at A on CRT is proportional to signal detected from A on specimen and signal is modulated by amplifier.

SEM magnification

- Magnification in an SEM can be controlled over a range of about 6 orders of magnitude from about 10 to 500,000x.
- Image magnification in an SEM is NOT a function of the power of the objective lens
- Magnification results from the **ratio of the dimensions of the raster on the specimen and the raster on the display device**, i.e controlled by the current supplied to the x, y scanning coils.

Magnification=area scanned on the monitor/area scanned on the specimen

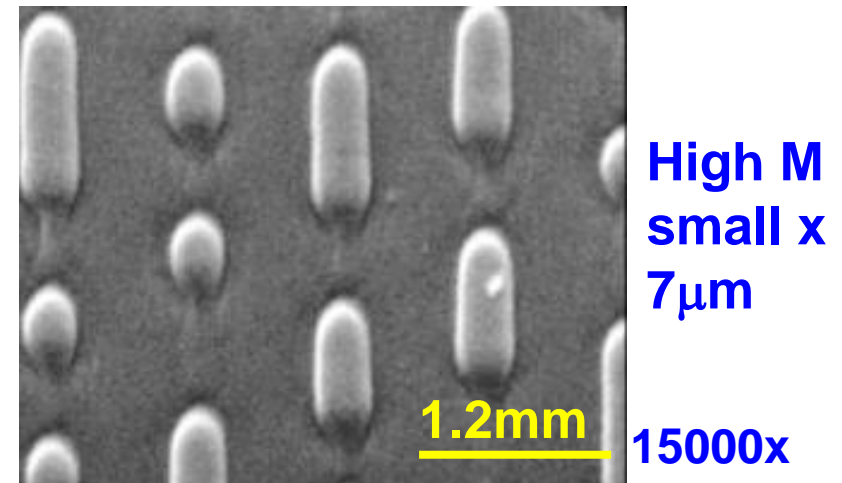
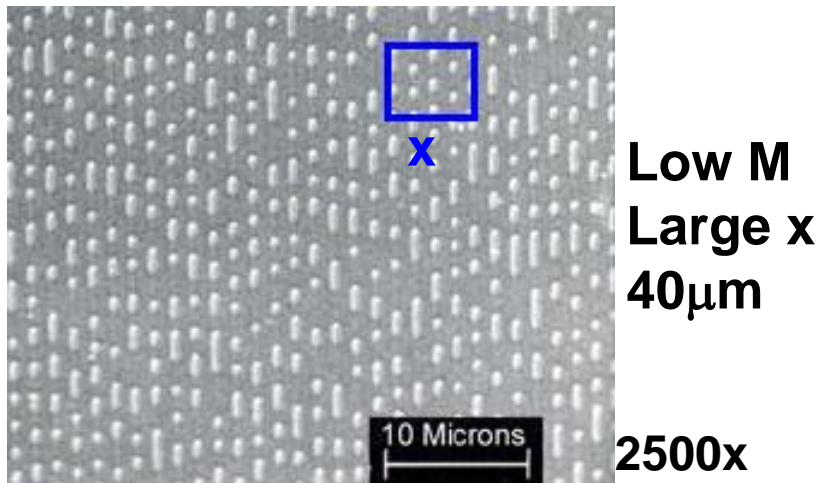
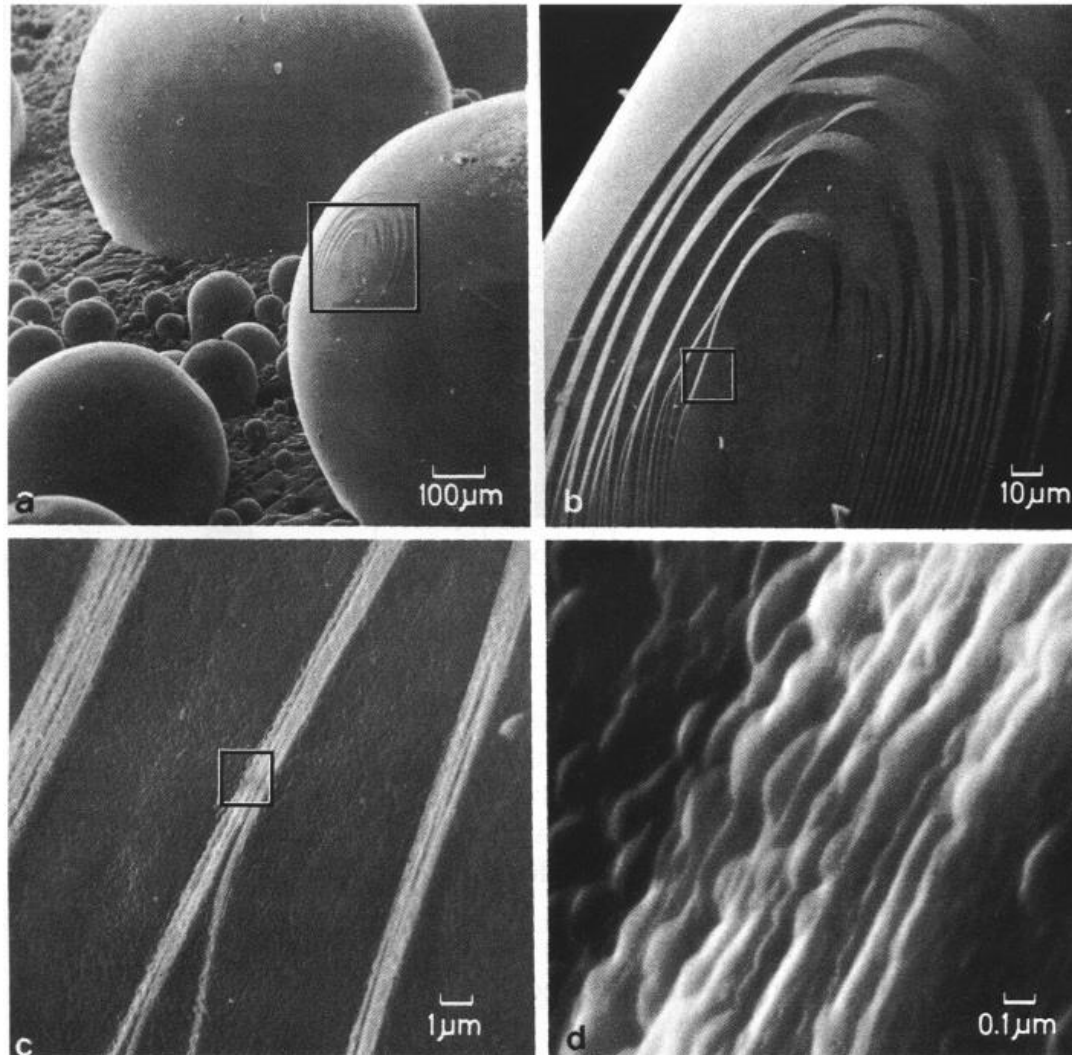


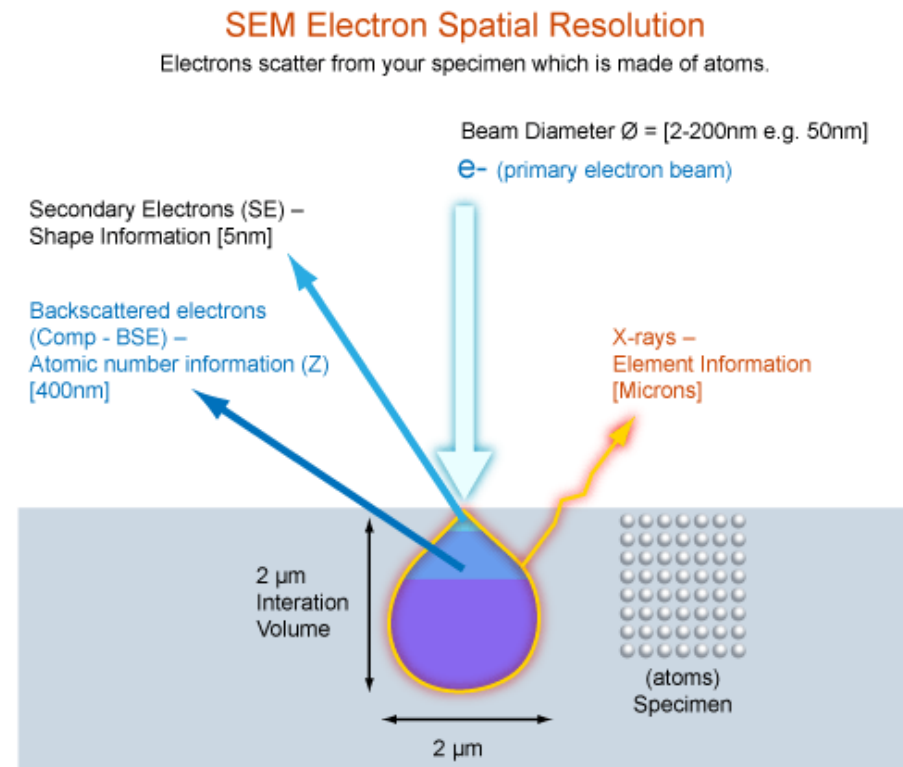
Image Magnification



Example of a series of increasing magnification (spherical lead particles imaged in SE mode)

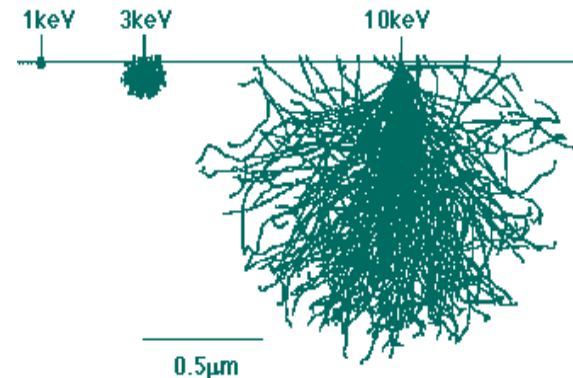
Resolution: spatial

- The resolution is the minimum spacing at which two features of the specimen can be recognized as distinct or separate.
- Unlike in an optical system, the resolution **is not limited by the diffraction limit, fineness of lenses or mirrors** or detector array resolution.
- The **spatial resolution** of the SEM depends on
 - the size of the **electron spot**, which in turn depends on both the wavelength of the electrons and the electron-optical system that produces the scanning beam.
 - the size of the **interaction volume**.
- The resolution can fall somewhere between less than 1 nm and 20 nm.

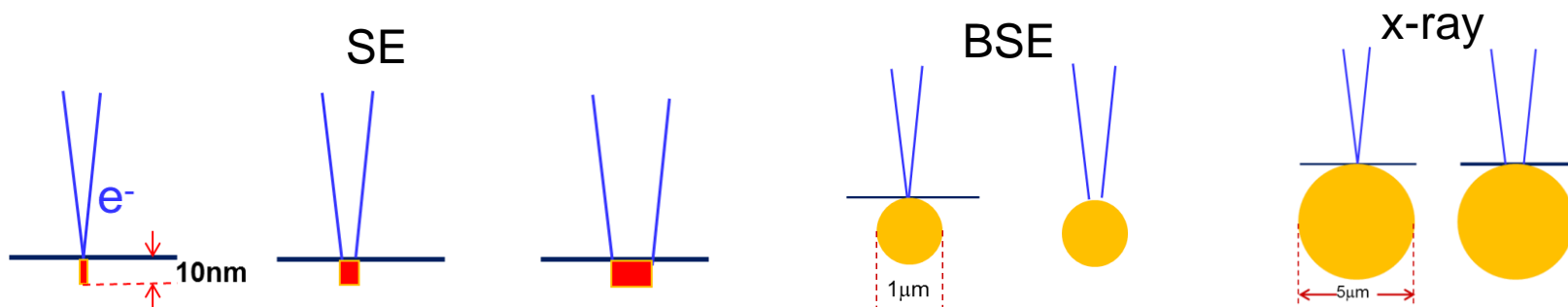


Resolution: interaction/escape volume

- The interaction volume falls with beam energy E as $\sim 1/E^5$
- If the screen can display 1000 pixels/(raster line), then there are 1000 pixels on each raster line on the specimen.
- The resolution is the pixel diameter on specimen surface.
 - If escape volume is larger than the pixel size: resolution decreased since signal from different pixel will overlap. The image will appear out of focus.
 - If escape volume equals the pixel size: optimum resolution condition
 - If escape volume is smaller than pixel size: the resolution is still achieved but signal will be weak and image appears noisy



Effect of probe size on escape volume



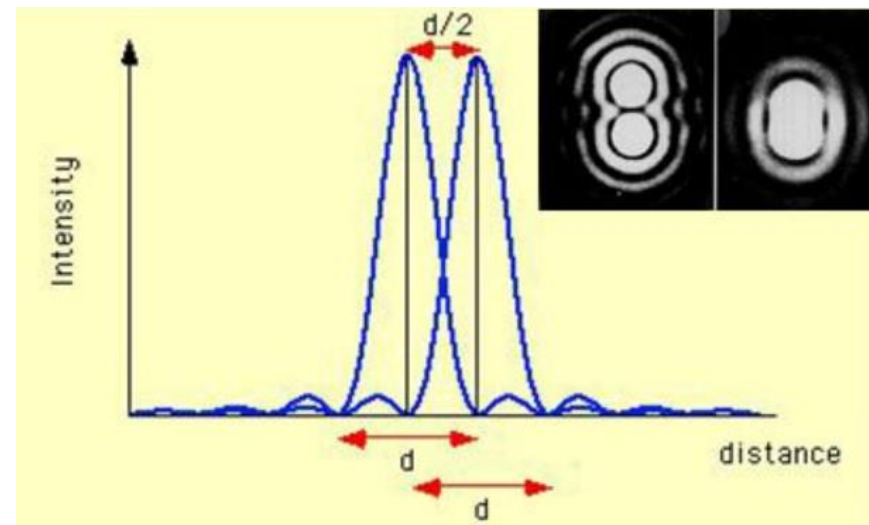
Resolution: electron optics limit

- Recall Rayleigh criterion for OM:

$$d_{min} = \frac{0.61\lambda}{(NA)}$$

- For electrons: de Broglie wavelength $\lambda = h/p$ where p is the electron momentum

$$\lambda = \frac{h}{\sqrt{2m_e(KE)}}$$



- For electron diffraction: $d_d = \frac{1.22\lambda}{\alpha}$,

for a 20-keV beam, $\lambda = 0.0087 \text{ nm}$ and $\alpha = 5 \times 10^{-3} \text{ rad}$, $d_d = 2.1 \text{ nm}$

- SEM resolution is also affected by **chromatic and spherical aberrations** (C_s) of lenses:

$$d_{min} = 1.29\lambda^{3/4}C_s^{1/4}$$

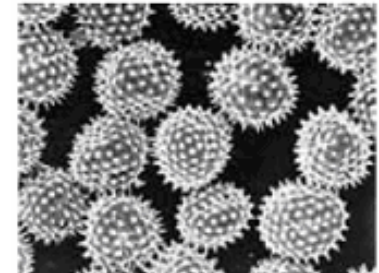
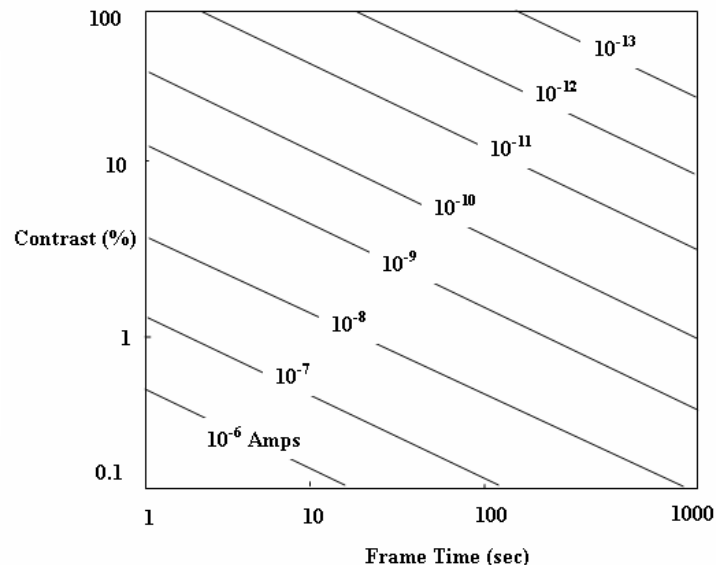
- A SEM fitted with an FEG has an **achievable resolution** of $\sim 1.0 \text{ nm}$ at 30 kV due to smaller C_s ($\sim 20\text{mm}$) and λ .

Resolution: specimen contrast

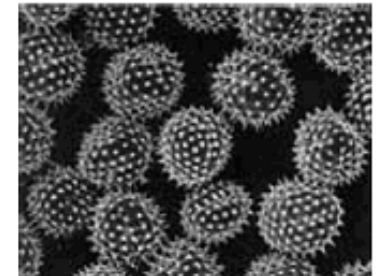
Contrast can be defined as $(S_2 - S_1)/S_2$ where S_1 and S_2 are the background and feature signals, respectively and $S_2 > S_1$.

- Topological contrast: the size, shape and texture of three dimensional objects, dependent upon the number of SEs and BSEs being emitted from different areas of the specimen
- Compositional contrast: different numbers of backscattered electrons being emitted from areas of the sample differing in atomic number

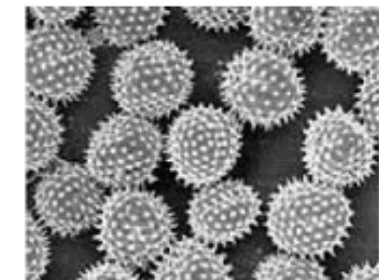
Contrast	d_{min} (nm)
1.0	2.3
0.5	4.6
0.1	23
0.01	230



Excessive contrast



Insufficient contrast



Optimum contrast & brightness

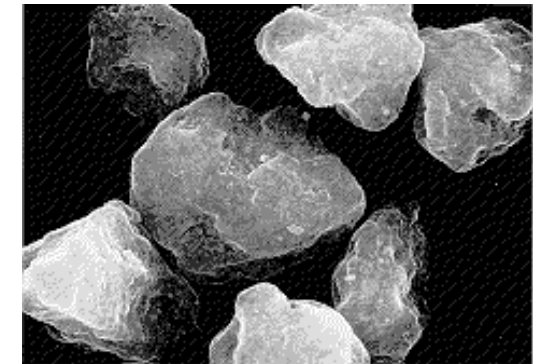
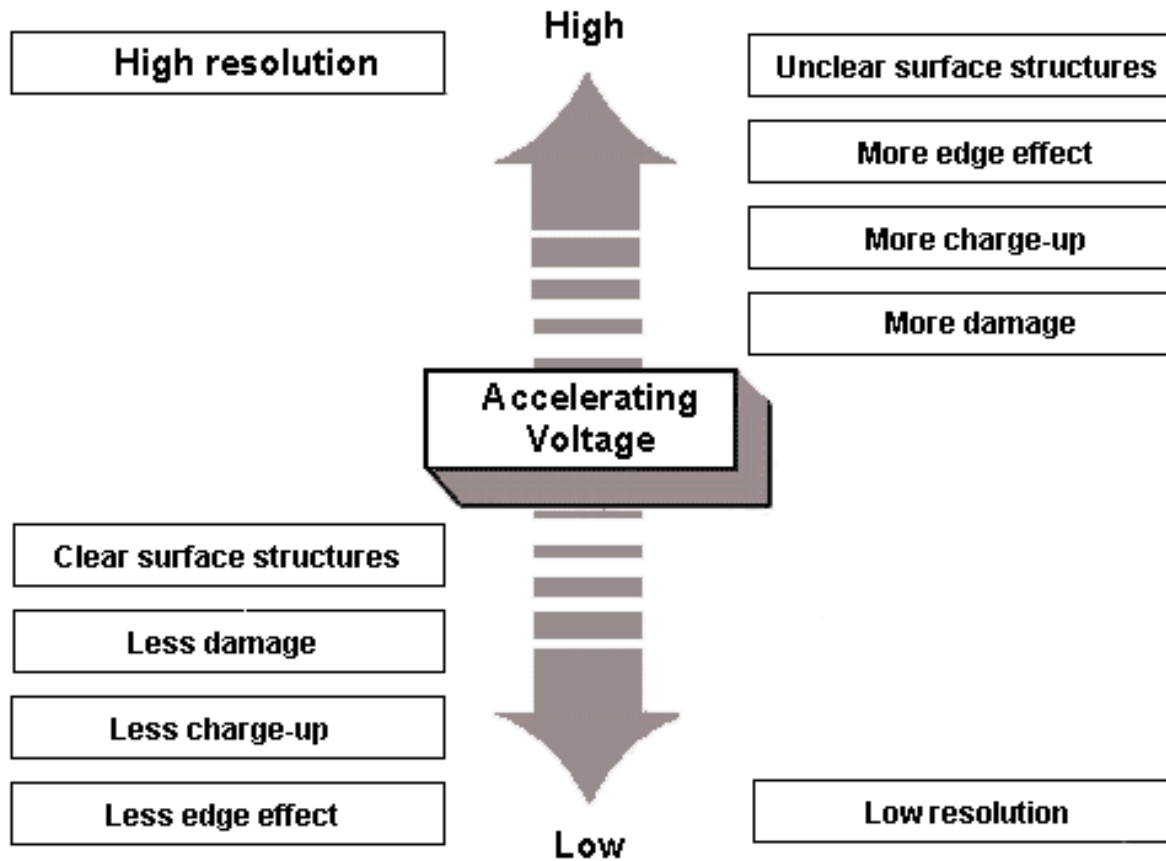
http://www.charfac.umn.edu/sem_primer.pdf

Resolution of Images

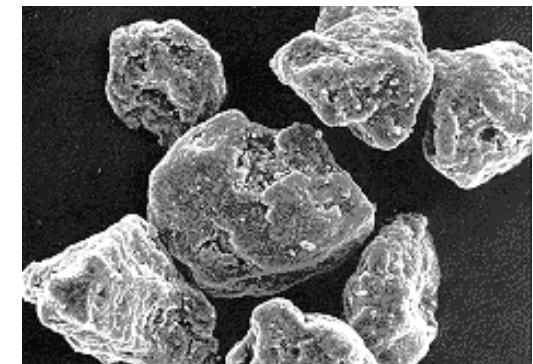
In an extremely good SEM, **resolution** can be **a few nm**. The limit is set by the **electron probe size**, which in turn depends on the quality of the **objective lens** and **electron gun**.

Magnification	Pixel diameter on Specimen	
	μm	nm
10	10	10000
100	1	1000
1000	0.1	100
10000	0.01	10
100000	0.001	1

Effect of acceleration voltage



30 kV 2500 X

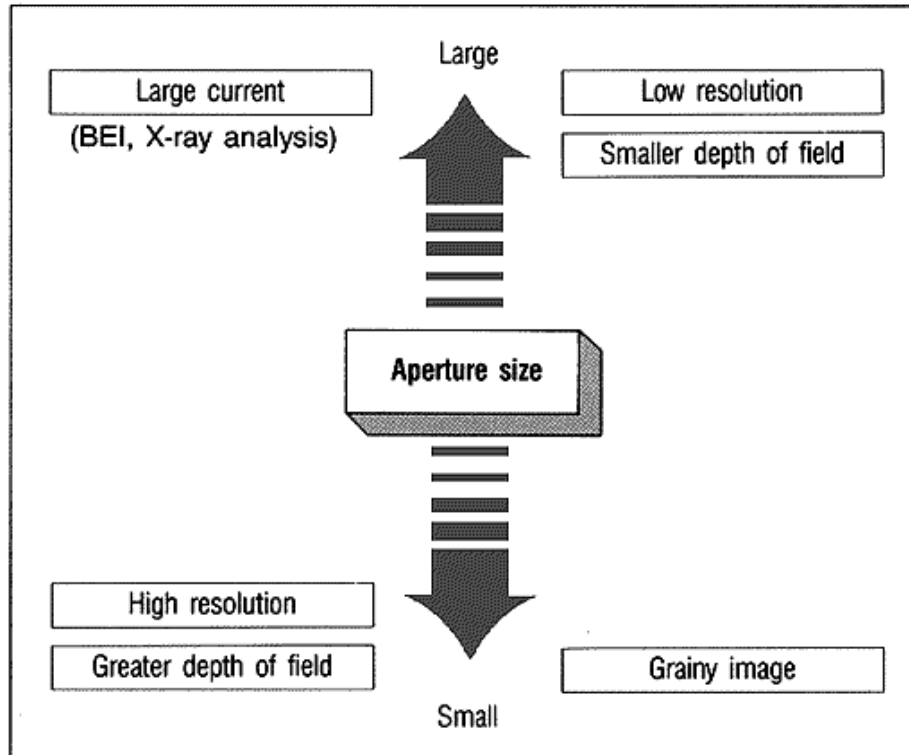


5.0 kV 2500 X

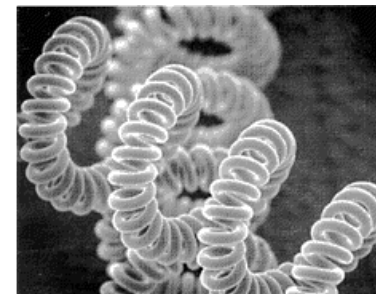
The image sharpness and resolution are better at the higher accelerating voltage. However, using high accelerating voltage cannot reveal the contrast of the specimen surface structure.

Effect of objective lens aperture

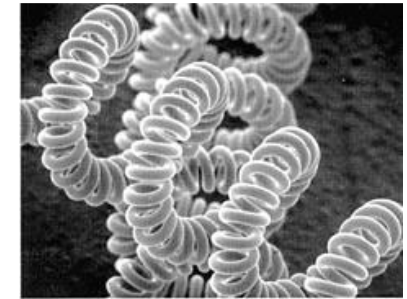
SEM images require not only a fine electron probe, but also a sufficient amount of signal for forming an image. The objective lens (OL) cannot be reduced unnecessarily. The OL aperture must be selected with consideration given to the effect on the image.



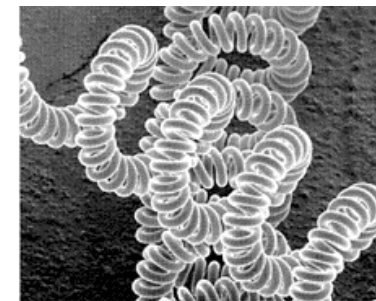
Electric light bulb filament



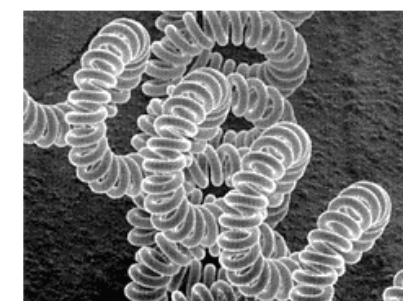
Aperture diameter = 600 μm
Working distance = 10 mm



Aperture diameter = 200 μm
Working distance = 10 mm



Aperture diameter = 200 μm
Working distance = 38 mm

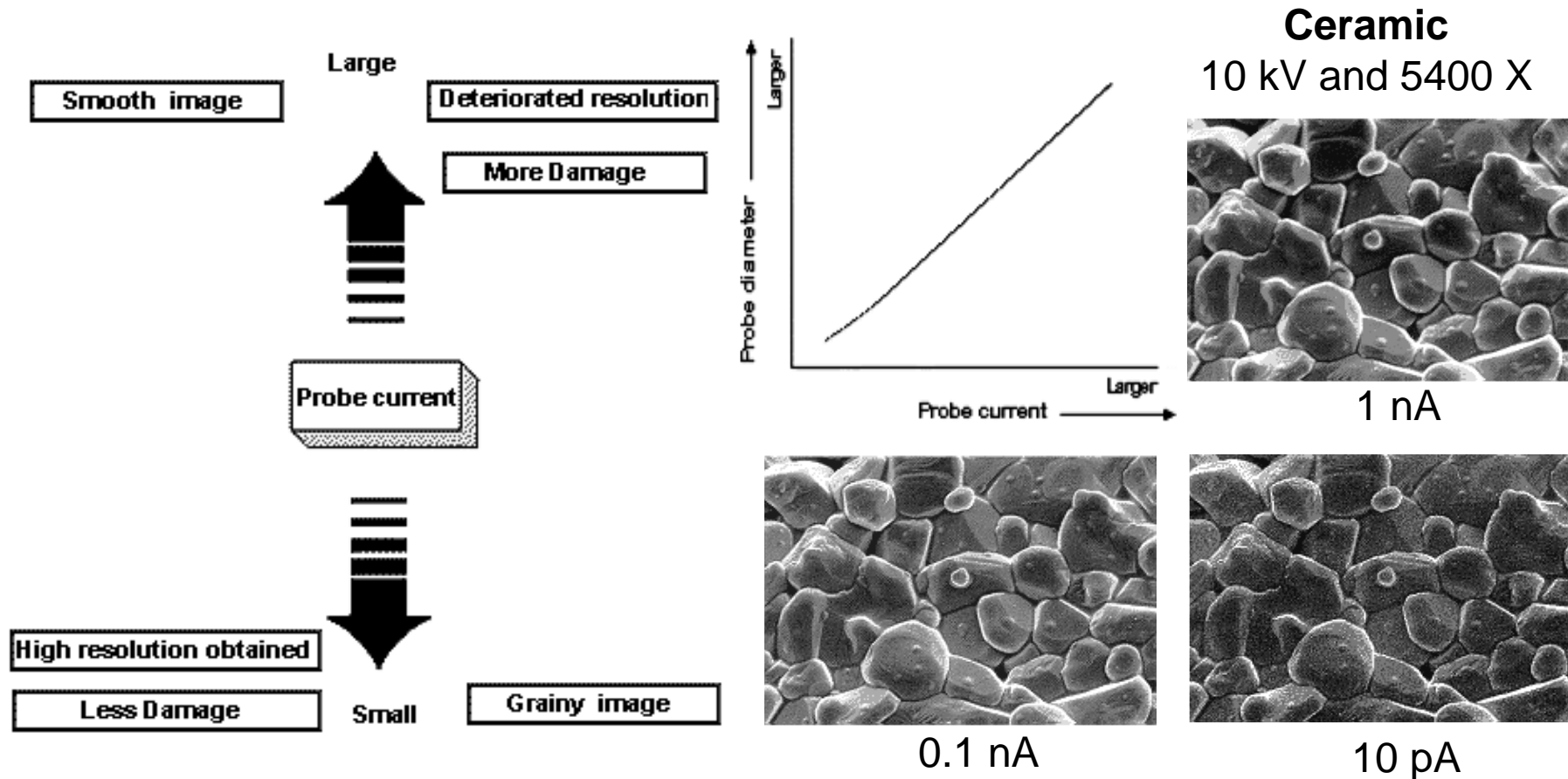


Aperture diameter = 100 μm
Working distance = 38 mm

The smaller the objective lens aperture diameter and the longer the working distance (WD), the greater will be the depth of field

Effect of electron probe current

In the SEM, the smaller the electron probe diameter, the higher the magnification and resolution. However, as the probe diameter is reduced, the probe current is reduced, affecting image smoothness.

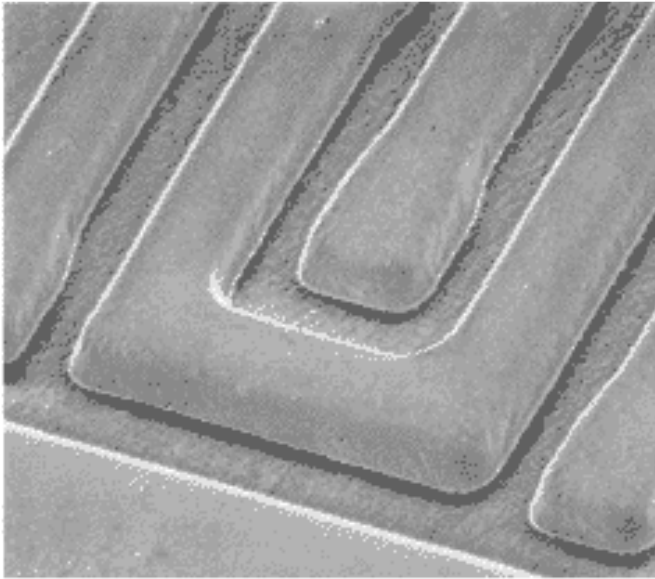


The smaller the probe current, the sharper is the image, but the image becomes grainy.

Effect of sample charging

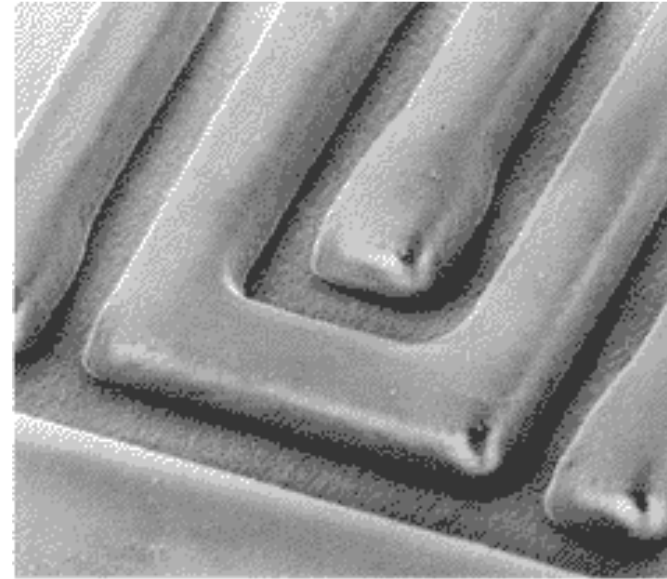
Charging is the condition when a material cannot effectively conduct the beam energy imparted to it. Non-conducting specimen will accumulate charge and the ensuing image will glow. Typically non-conducting samples are coated with thin conductive films (Carbon, Gold-Palladium, Platinum etc) to facilitate electron flow.

Image with No Surface Charging



1.0 kV 3200 X

Image with Surface Charging



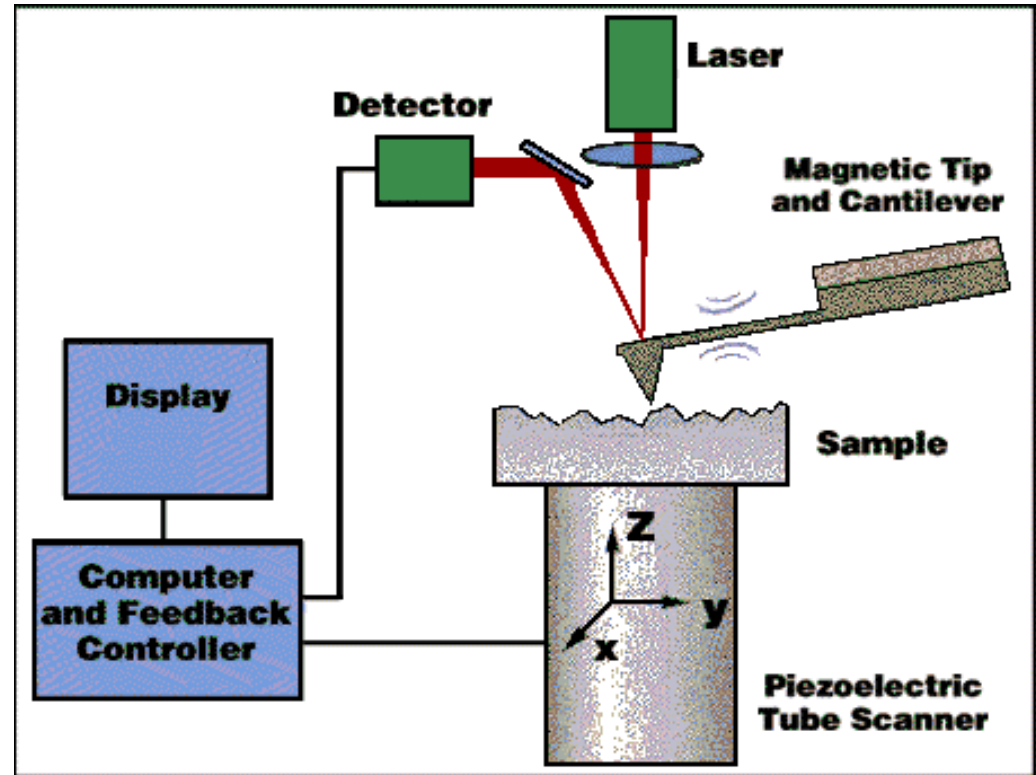
1.3 kV 3200 X

Scanning probe microscopy (SPM)

Scanning probe microscopy (SPM)

Scanning probe microscopy (SPM) is a branch of microscopy that forms images of surfaces using a **physical probe** that scans the specimen.

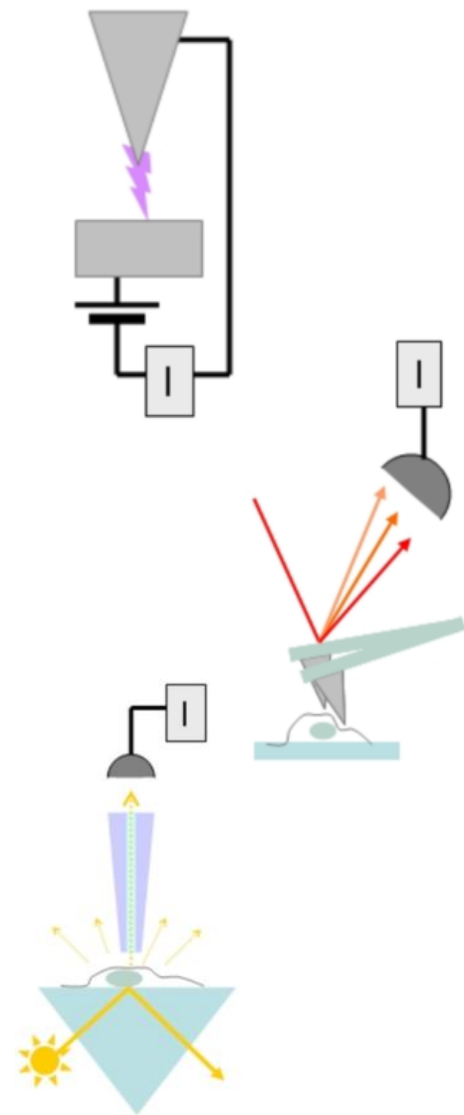
- SPM was founded in 1981, with the invention of the **scanning tunneling microscope** by Binnig and Rohrer. (the Nobel Prize in Physics in 1986)
- To form images, scanning probe microscopes **raster scan the tip over the surface**.
- At discrete points in the raster scan a value is recorded (depends on the type of SPM), displayed as a heat map to produce the final STM images



Different types of SPM

Broadly speaking, there are three main categories of SPM

- **Scanning tunneling microscopy (STM)**: uses an atomically sharp metallic tip and records the minute **tunneling current** (I) between the tip and the surface, when the tip is hovering so close to the surface that electrons can move between the surface and the tip. It maps out the sample topography and electrical properties.
- **Atomic force microscopy (AFM)**: a cantilever with a sharp tip - somewhat like the needle of an old record player - is scanned over the surface. Using the **van der Waals** forces or contact forces between a tip and the sample, the sample topography or mechanical properties can be measured.
- **Scanning near-field optical microscopy (SNOM)**: a probe with a **small aperture** is scanned over the surface collecting the **scattered light** coming from regions much smaller than the wavelength of the light used.



SPM: advantages and disadvantages

Advantages:

- The **resolution** of the microscopes is not limited by diffraction, only by the **size of the probe-sample interaction volume** (as small as a few pm).
- Able to measure small local differences in object height (like that of 135 pm steps on <100> silicon).
- Laterally the probe-sample interaction extends only across the tip atom or atoms involved in the interaction.
- The interaction can be used to modify the sample to create small structures
- Can be measured in any ambient: air or liquid

Disadvantages:

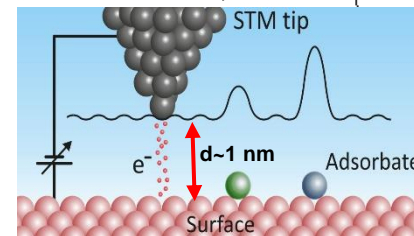
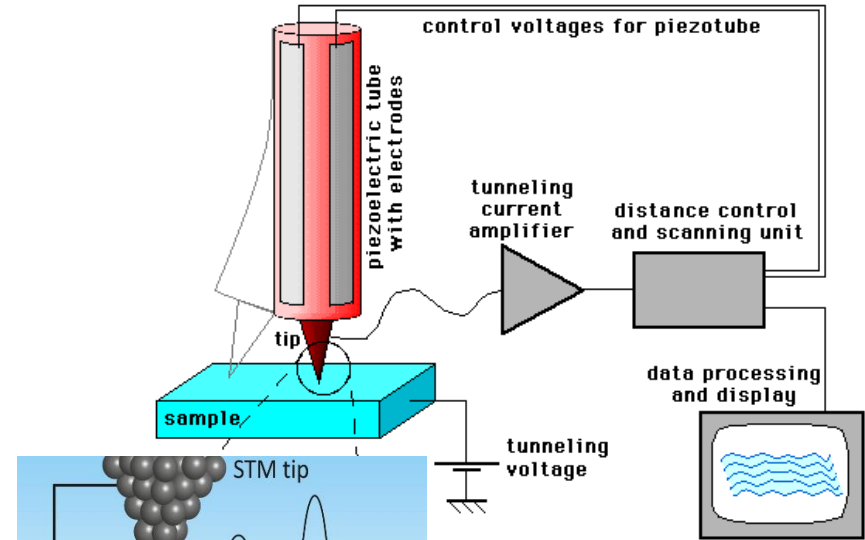
- The detailed shape of the scanning tip is sometimes difficult to determine, affecting the resulting data especially when the specimen varies in height.
- The scanning techniques are generally **slower** in acquiring images.
- Result greatly affected by time-domain effects like specimen drift, feedback loop oscillation, and mechanical vibration.
- The maximum image size is generally small
- Not useful for examining buried solid-solid or liquid-liquid interfaces

Scanning Tunneling Microscopy (STM)

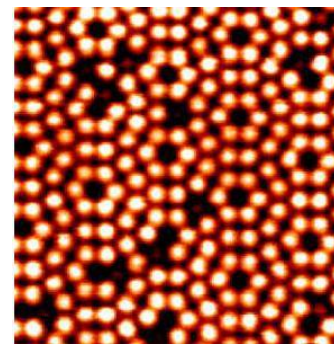
- STMs use a sharpened, **conducting tip** with a bias voltage applied between the tip and the sample.
- When the tip is brought within about 10Å of the sample, electrons from the sample begin to "tunnel" through the gap into the tip or vice versa, depending upon the sign of the bias voltage.
- The resulting tunneling current varies with tip-to-sample spacing, and it is the signal used to create an STM image.

$$I = Ve^{-Cd}$$

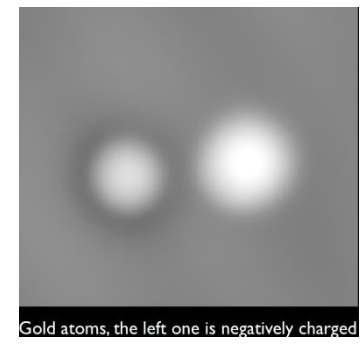
- For tunneling to take place, both the sample and the tip **must be conductors or semiconductors**. STMs cannot image insulating materials.



TU WIEN Institut für Allgemeine Physik www.iap.tuwien.ac.at

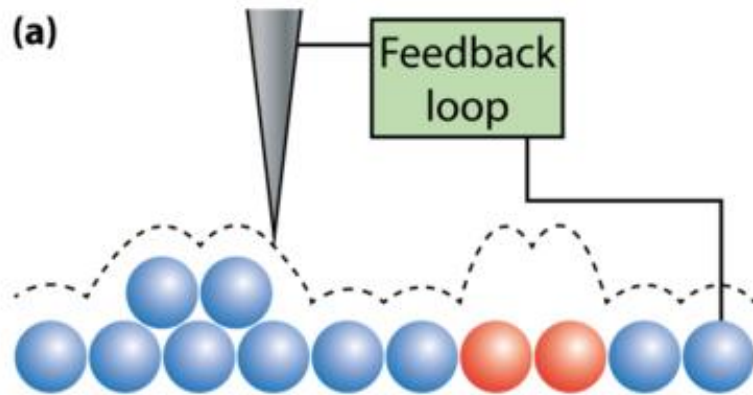


Silicon atoms on a surface via STM



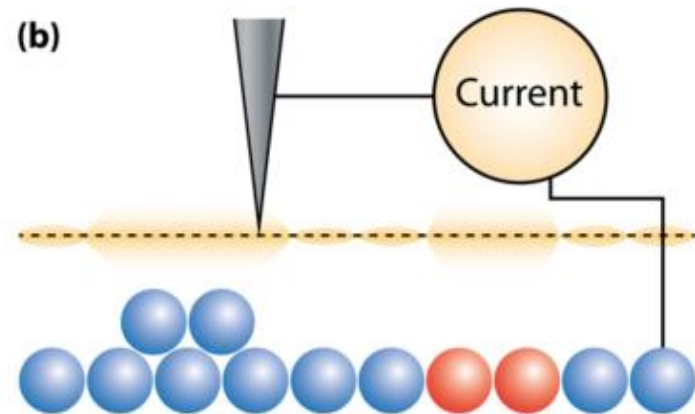
Gold atoms, the left one is negatively charged

STM: modes of operation



constant current mode

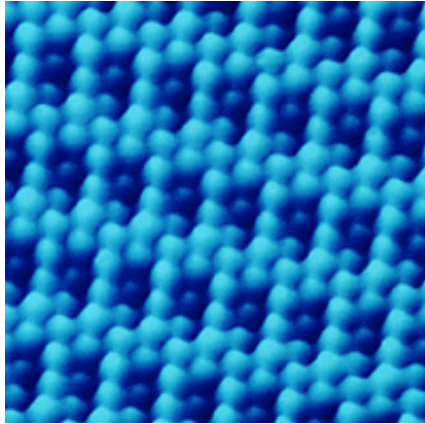
- STMs use feedback to keep the tunneling current constant by adjusting the height of the scanner at each measurement point
- the voltage applied to the piezoelectric scanner is adjusted to increase/decrease the distance between the tip and the sample
- The image is then formed by plotting the **tip height vs. the lateral tip position**.



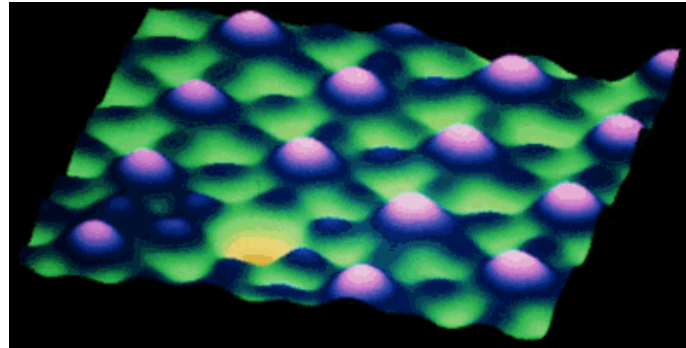
constant height mode

- Tunneling current is monitored as the tip is scanned parallel to the surface.
- There is a periodic variation in the separation distance between the tip and surface atoms.
- A plot of the **tunneling current vs. tip position** shows a periodic variation which matches that of the surface structure—a direct "image" of the surface.

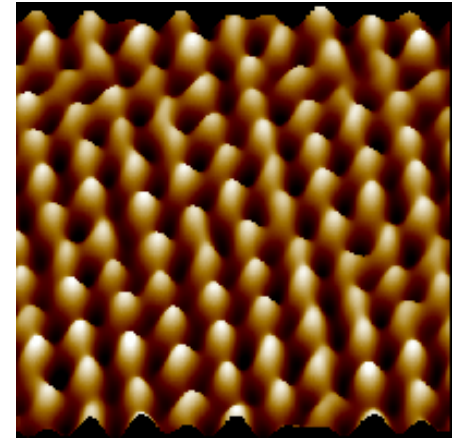
STM images



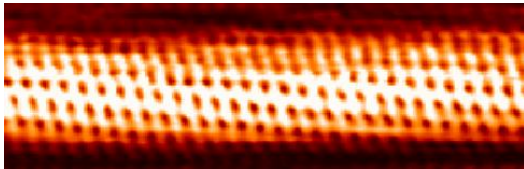
Oxygen Atom Lattice on Rhodium Single Crystal. 4nm Scan



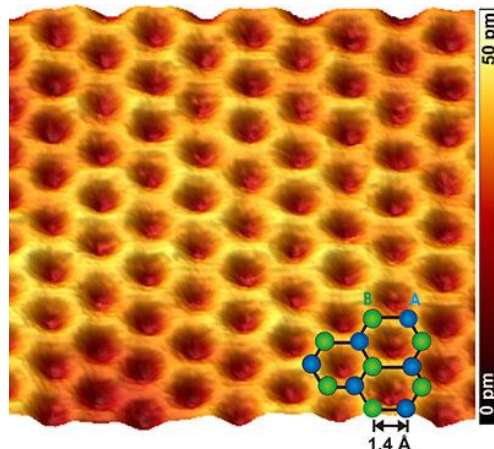
Single-atom Defect in Iodine Adsorbate Lattice on Platinum. 2.5nm Scan



Mica Surface Atoms, 5nm Scan



An STM image of a single-walled carbon nanotube



Atomically resolved graphene lattice. The blue and green spheres indicate two carbon atoms of the unit cell labeled by A and B.

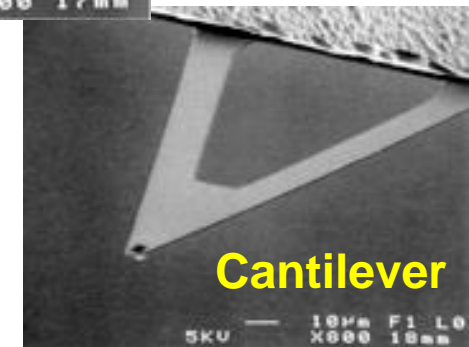
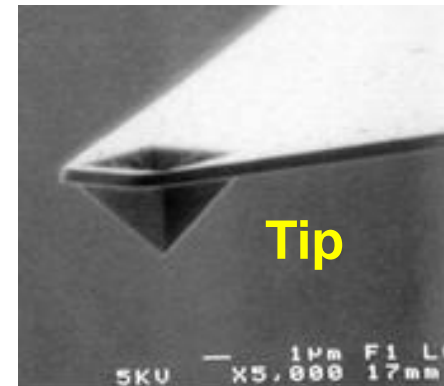
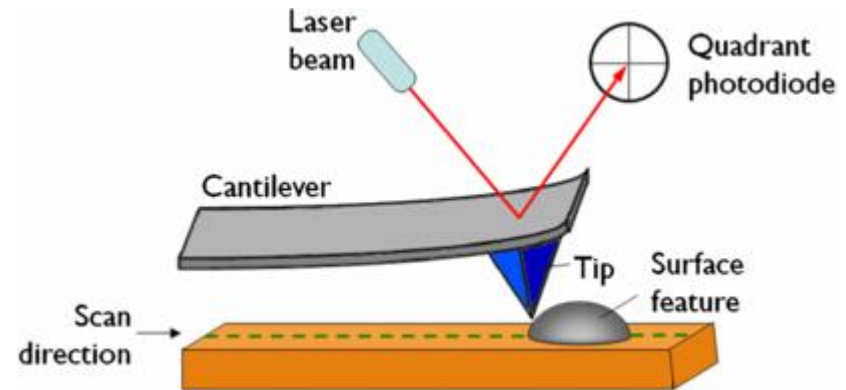
<http://www.nanoscience.de/HTML/research/graphene.html>



Cobalt atoms arranged with a scanning tunneling microscope

Atomic force microscopy (AFM)

- The atomic force microscope (AFM) probes the surface of a sample with a **sharp tip**, a couple of microns long and often **less than 100Å in diameter**. The tip is located at the free end of a cantilever that is 100 to 200μm long.
- Forces between the tip and the sample surface cause the cantilever to **bend**, or **deflect**.
- A detector measures the **cantilever deflection** as the tip is scanned over the sample, or the sample is scanned under the tip.
- The measured cantilever deflections allow a computer to generate a map of surface topography.
- AFMs can be used to study **insulators and semiconductors as well as electrical conductors**.

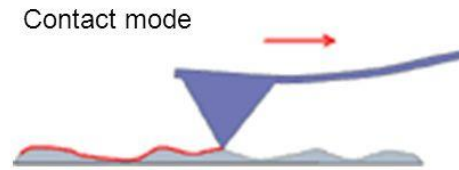


AFM: modes of operation

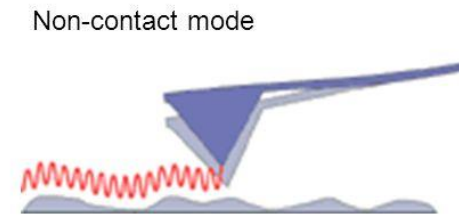
AFM operation is usually described as one of three modes, according to the nature of the tip motion: **contact mode**, also called static mode (as opposed to the other two modes, which are called dynamic modes); **tapping mode**, also called intermittent contact, AC mode, or vibrating mode; and **non-contact mode**

Modes of operation. There are 3 modes of AFM operation

1. Contact mode



2. Non-contact mode



3. Tapping mode

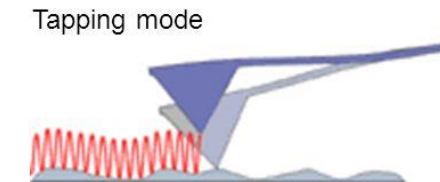
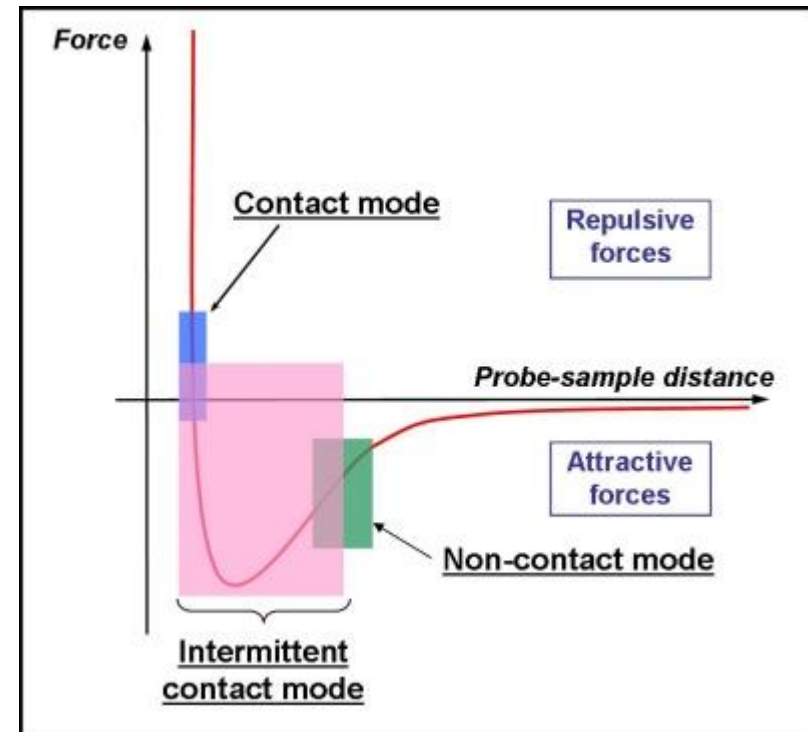
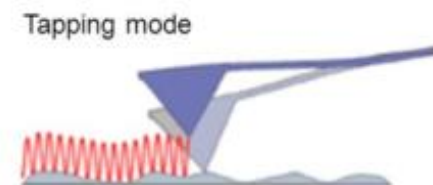
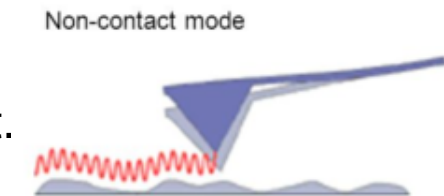
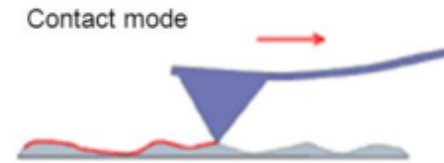


Fig. 7.3 Modes

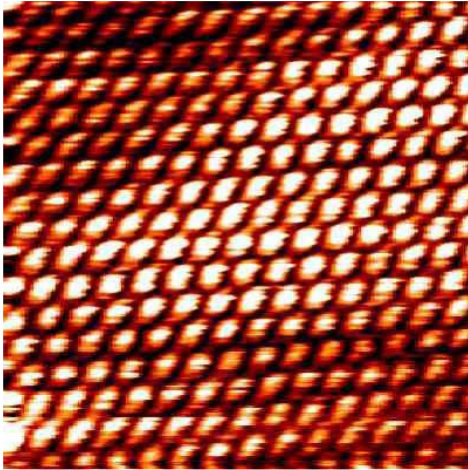


AFM: modes of operation

- **Contact:** the tip is "dragged" across the surface of the sample and the contours of the surface are measured either using the deflection of the cantilever directly or the feedback signal required to keep the cantilever at a constant position.
 - High speed but image **heavily influenced by frictional and adhesive forces** which can **damage** samples and distort image.
- **Non-contact:** the tip does not contact the sample surface. The cantilever is **oscillated with typically a few nm-pm**. A topographic image of the sample surface is constructed by measuring the tip-to-sample distance at each (x,y) data point.
 - No tip or sample degradation effects, hence it is preferred for **soft samples**.
 - Provides **lower resolution** and can be hampered by the contaminant layer which can interfere with oscillation
- **Tapping:** the cantilever is driven to oscillate up and down at or near its resonance frequency with amplitude up to 200 nm. An image is produced by imaging the force of the intermittent contacts of the tip with the sample surface.
 - eliminates frictional forces and prevent the tip from being trapped by adhesive meniscus forces from contaminants.



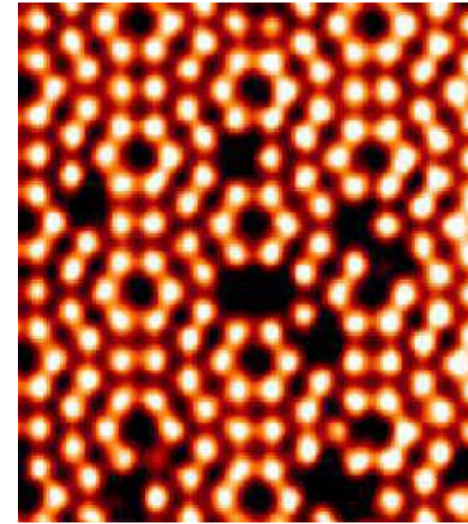
AFM applications



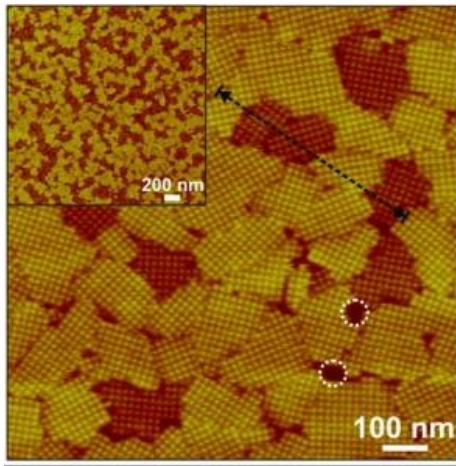
Contact Mode: Au(111) polycrystalline film on glass.



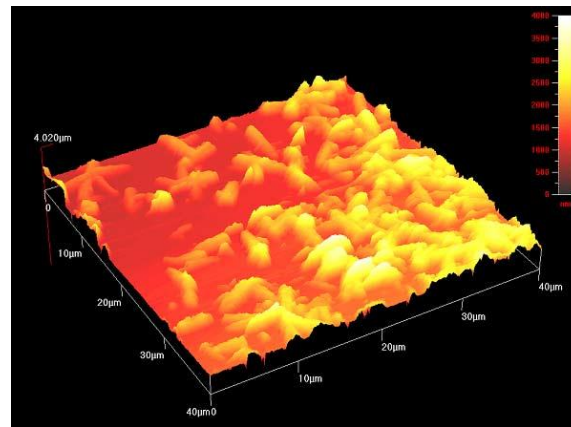
Tapping Mode: Arene on graphite



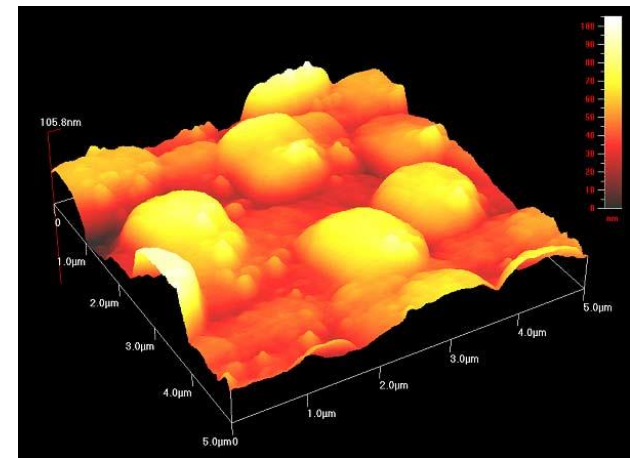
Non-contact Mode: Si(111) 7x7



2D S-layers assembled on mica
(Image from Molecular Foundry, LBNL)



3D image of DNA crystals



3D image of Si wafer coated with Ta layer

STM vs. AFM

STM	AFM
Measures local electron density of states , not nuclear positions—not true topographic imaging	Real topographic imaging
High lateral and vertical resolution –because of the exponential dependence of the tunneling current on distance	Lower lateral resolution
Exponential dependence between tunneling current and distance	The force-distance dependence in AFM is much more complex
Probe electronic properties (LDOS – including spin states)	Probe various physical properties: magnetic, electrostatic, hydrophobicity, friction, elastic modulus, etc
Generally applicable only to conducting (and semiconducting) samples	Applied to both conductors and insulators
Writing voltage and tip-to-substrate spacing are integrally linked	Writing voltage and tip-to-substrate spacing can be controlled independently

Advanced SPM Techniques

- **Lateral Force Microscopy (LFM)**
measures frictional forces between the probe tip and the sample surface
- **Magnetic Force Microscopy (MFM)**
measures magnetic gradient and distribution above the sample surface
- **Electric Force Microscopy (EFM)**
measures electric field gradient and distribution above the sample surface
- **Scanning Thermal Microscopy (SThM)**
measures temperature distribution on the sample surface
- **Scanning Capacitance Microscopy (SCM)**
measures carrier (dopant) concentration profiles on semiconductor surfaces
- **Nanoindenting/Scratching**
measures hardness, and scratch or wear testing to investigate film adhesion and durability
- **Phase Imaging**
measures variations in surface properties (stiffness, adhesion, etc.) as the phase lag of the cantilever oscillation relative to the piezo drive and provides nano-scale information about surface structure



**JEOL 6700F Ultra High Resolution
Scanning Electron Microscope**



A 3D variable temperature STM



**HS-AFM*1.0 - a High-Speed Atomic Force
Microscope**

SEM/SPM: Internet resources

SEM:

<http://www.mse.iastate.edu/microscopy>

http://www.charfac.umn.edu/sem_primer.pdf

<http://cfamm.ucr.edu/documents/sem-intro.pdf>

http://virtual.itg.uiuc.edu/training/EM_tutorial

<http://science.howstuffworks.com/scanning-electron-microscope.htm/printable>

http://www.matter.org.uk/tem/electron_gun/electron_sources.htm

http://www.matter.org.uk/tem/lenses/simulation_of_condenser_system.htm

<http://micro.magnet.fsu.edu/primer/java/lenses/converginglenses/index.html>

SPM:

http://www.doitpoms.ac.uk/tlplib/afm/tip_surface_interaction.php

<http://physics.usask.ca/~chang/course/phys404/STMandAFM.pdf>

http://www.eng.utah.edu/~ljang/images/Lecture_4_SPM.pdf

http://leung.uwaterloo.ca/MNS/102/Lect_2013/Lect_26A.pdf

<http://www.asylumresearch.com/Gallery/Gallery.shtml>