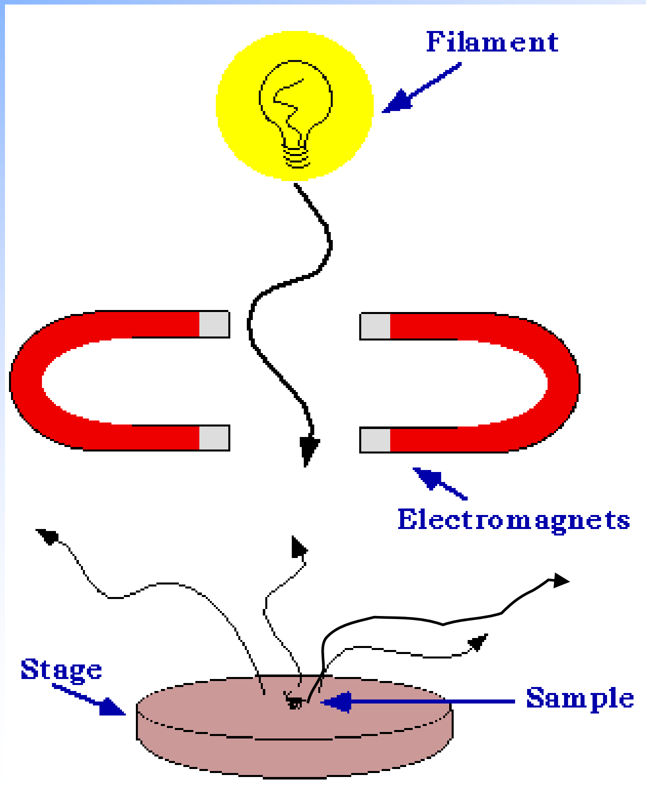
29 Jan. 2010

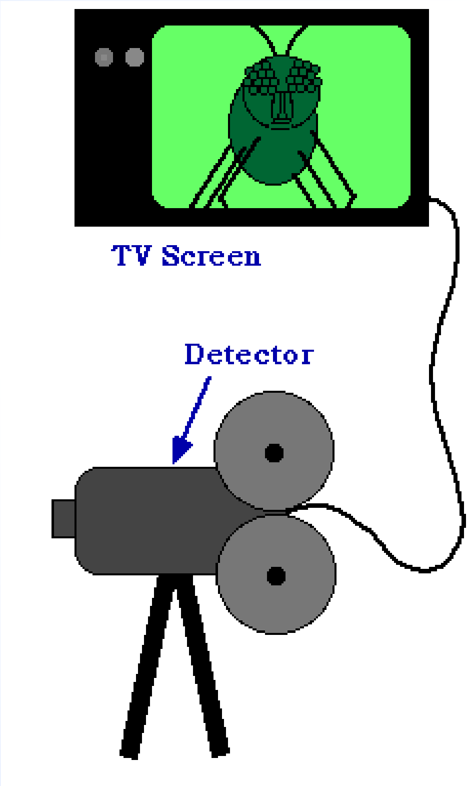
Scanning electron microscopy (SEM)

D.S. Su

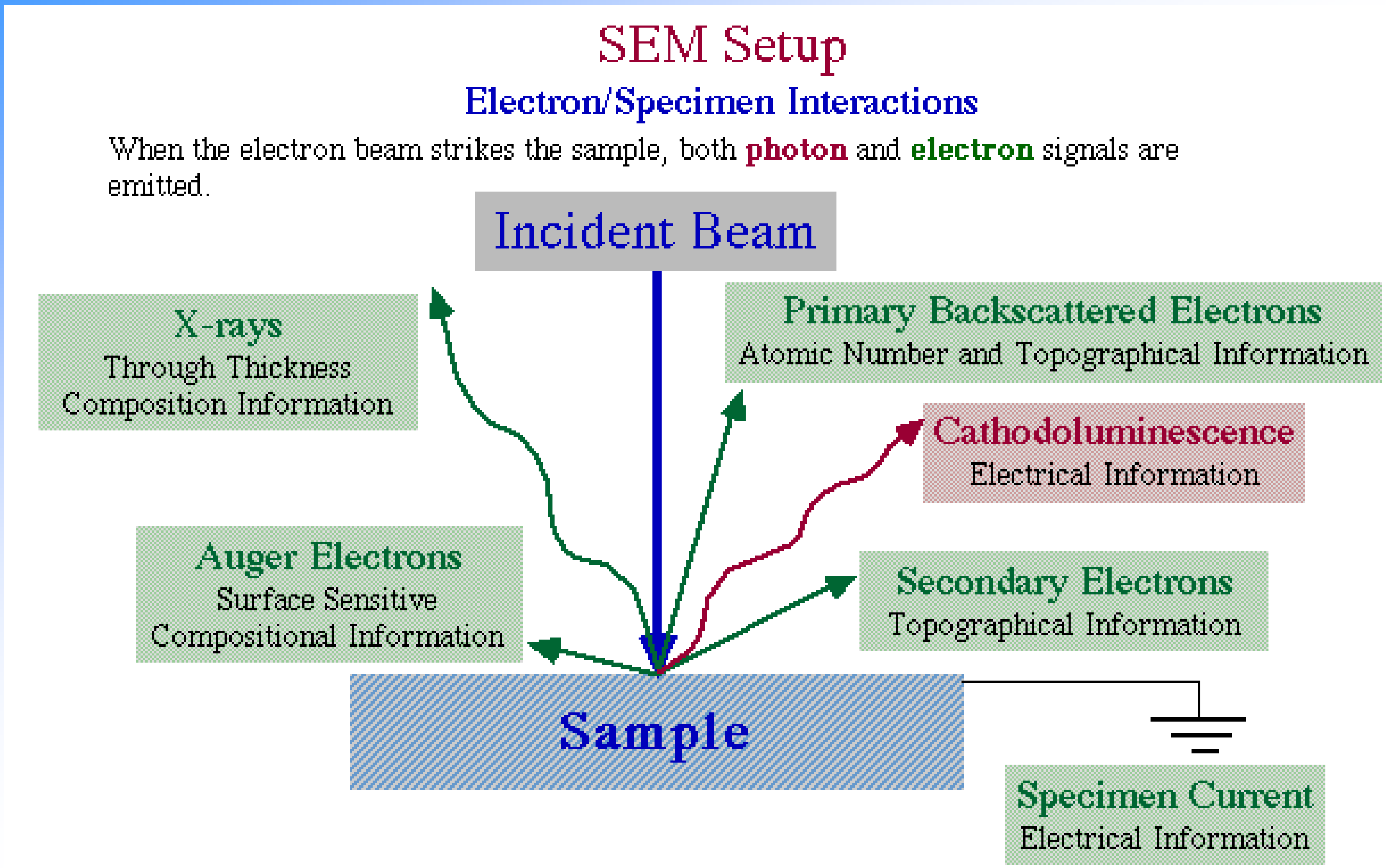
(SEM: Sächsisches Eisenbahnmuseum)

Principle of SEM





SEM: electron beam/specimen interactions



Inelastic scattering of electrons

Secondary Electron Emission

Secondary electrons are electrons in the specimen that are ejected by the beam electron

- **If the electrons are in the conduction or valence bands then it doesn´t take much energy to eject them. They are called slow SEs with energies typically below about 50 eV.**

- **If the electrons are strongly bound inner-shell electrons they are less readily ejected, but when they are thrown out of their shells they can have a significant fraction of the beam energy (fast SEs)**

- **If the electrons are ejected from an inner shell by the energy released when an ionized atom returns to the ground state, then these secondary electrons are called Auger electrons.**

Inelastic scattering of electrons: Secondary electrons

Slow Secondary Electrons

ejected from the conduction or valence bands of the specimen (usually has energy < 50 eV)

SEs can only escape if they are near the specimen surface.

Used to form images of the specimen surface.

Fast Secondary Electrons

High-energy electrons which are generated in the specimen.

In a TEM, fast SEs can have energies of ~ 50 – 200 keV

FSEs are generally both unavoidable and undesirable. They are not used to form images or give spectroscopic data. And they may low the quality of the latter.

Inelastic scattering of electrons: Secondary electrons

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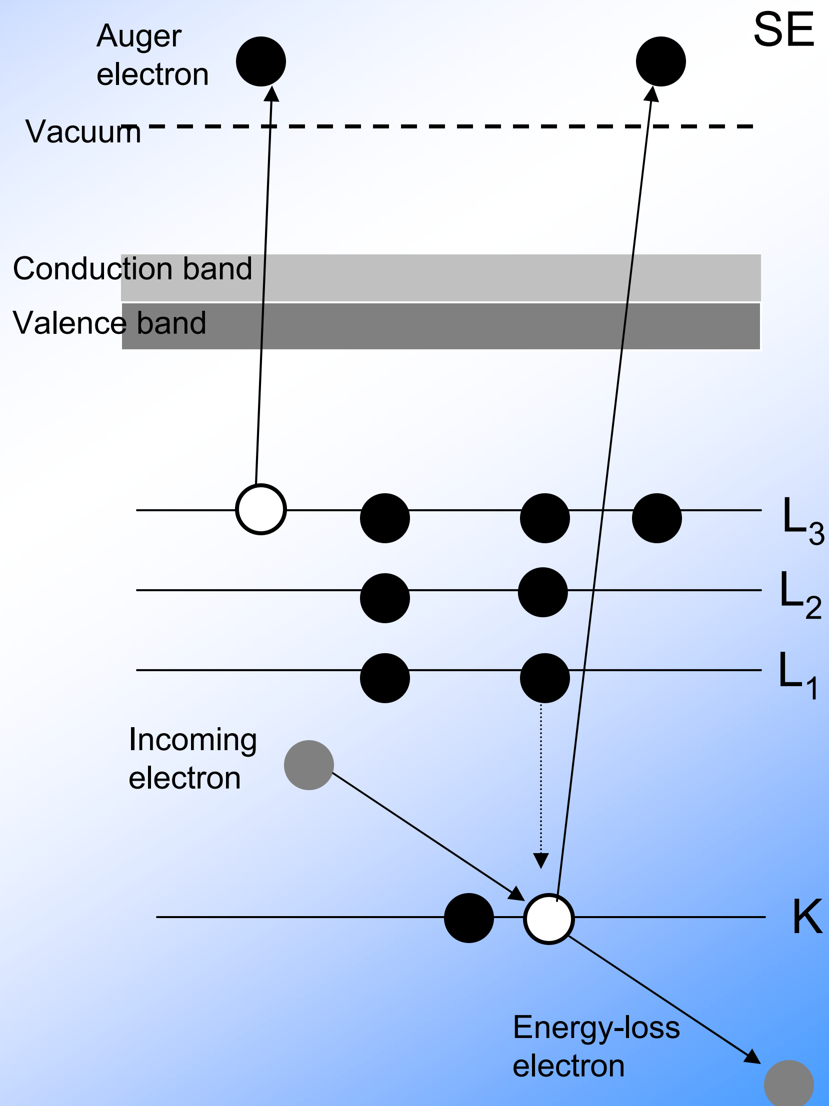
Auger Electrons

The energy of Auger electrons is given by the difference between the original excitation energy and the binding energy of the outer shell from which electron was ejected.

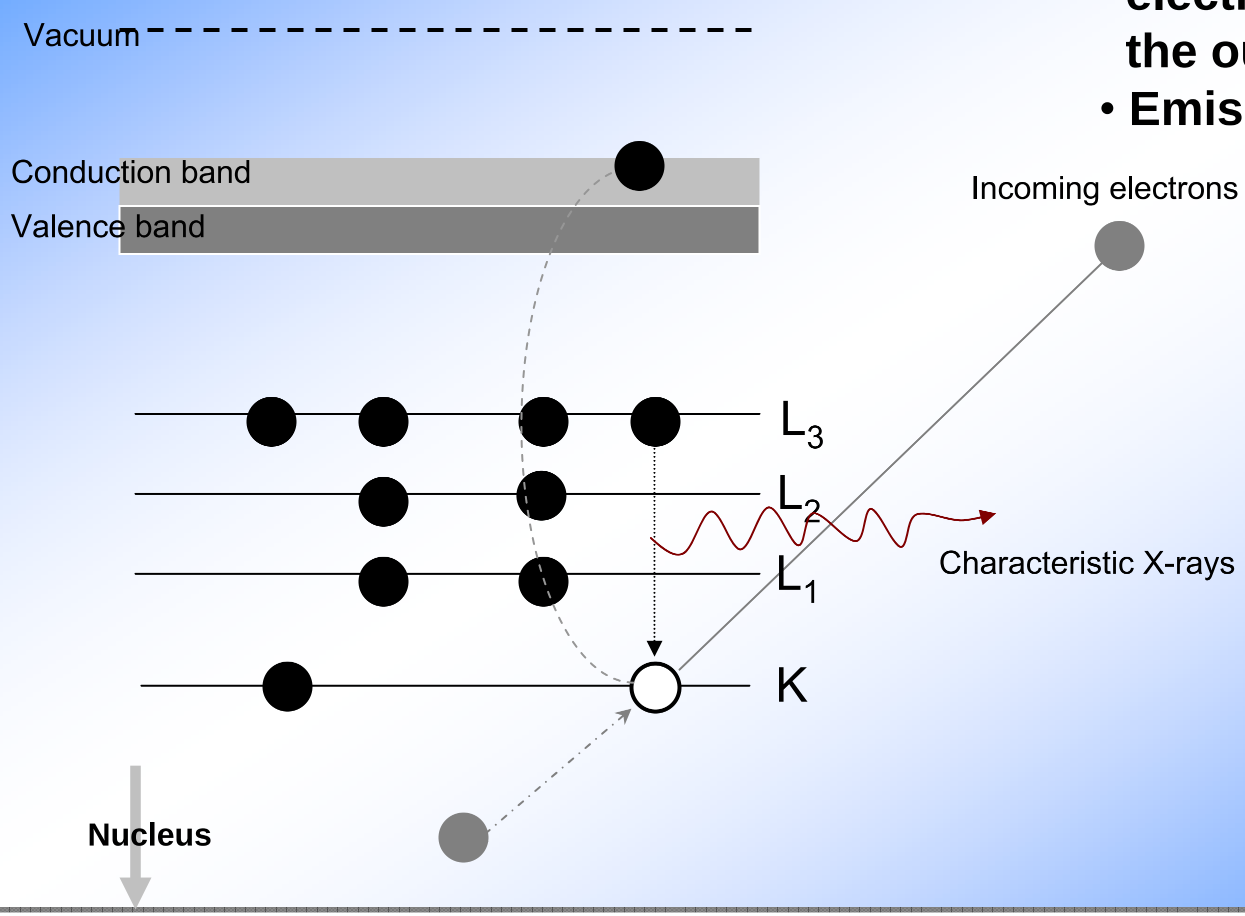
Typical Auger electron energies are in the range of a few hundred eV to a few keV and are strongly absorbed within the specimen

An alternative to X-ray emission as an Ionized atom returns to ground state.



X-ray emission of atomic electrons

Characteristic X-rays



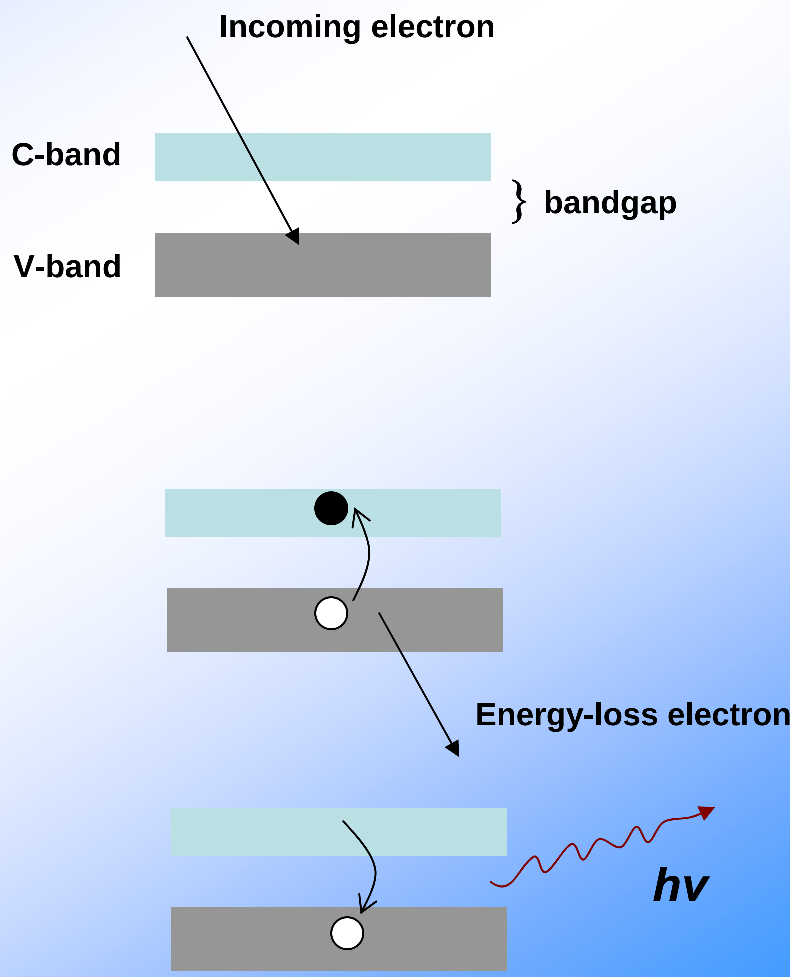
- Ionization
- Filling in the missing electron with one from the outer shells.
- Emission of X-rays or Auger e^-

Inelastic scattering of electrons

Electron-hole pairs and Cathodoluminescence (CL)

A semiconductor creates electron-hole pairs when hit by high-energy electron

If the electrons and holes recombine, light will be emitted (cathodoluminescence)



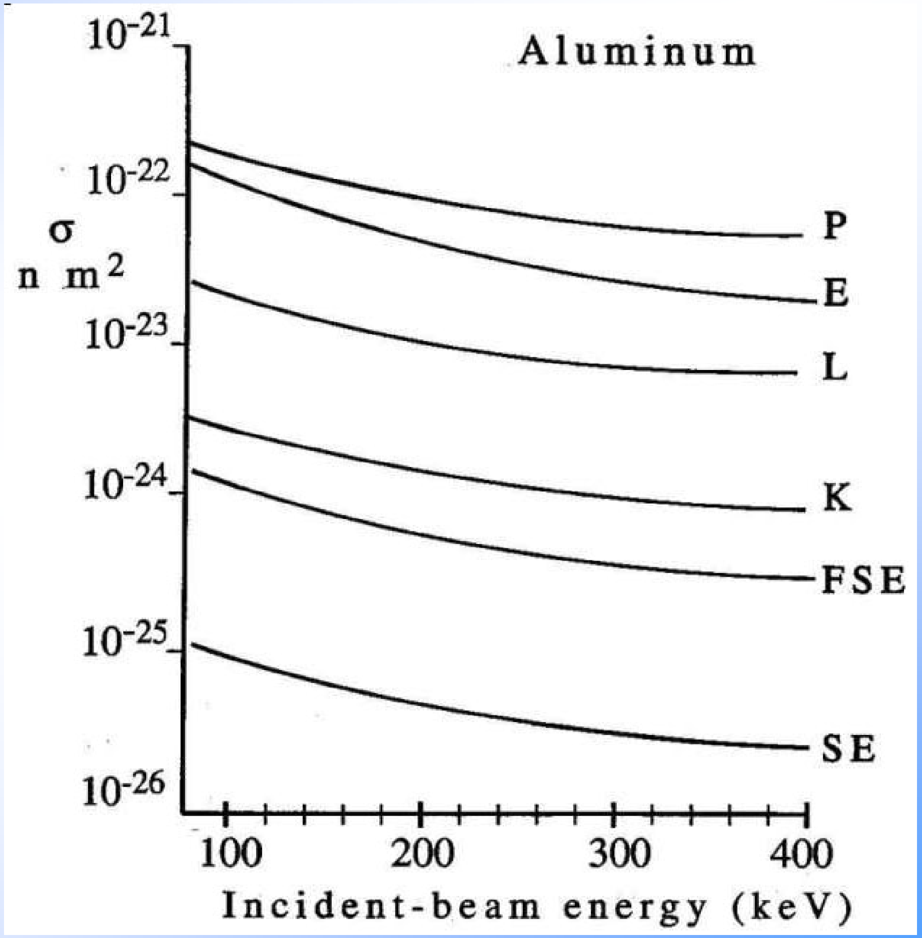
If a bias is applied (or if it happens to be a *p-n* junction), the electrons and holes can be separated.

The current detected is called the *electron beam induced current* or EBIC signal

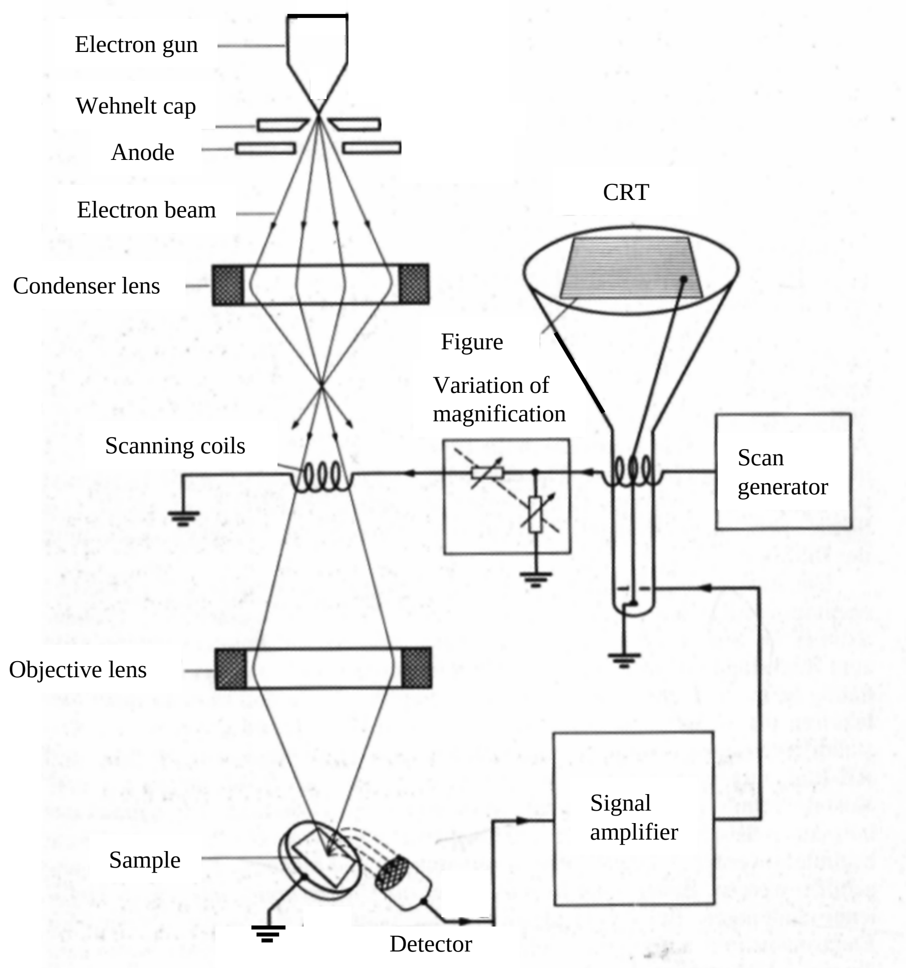
***Charge-collection microscopy* (CCM)**

Inelastic scattering of electrons

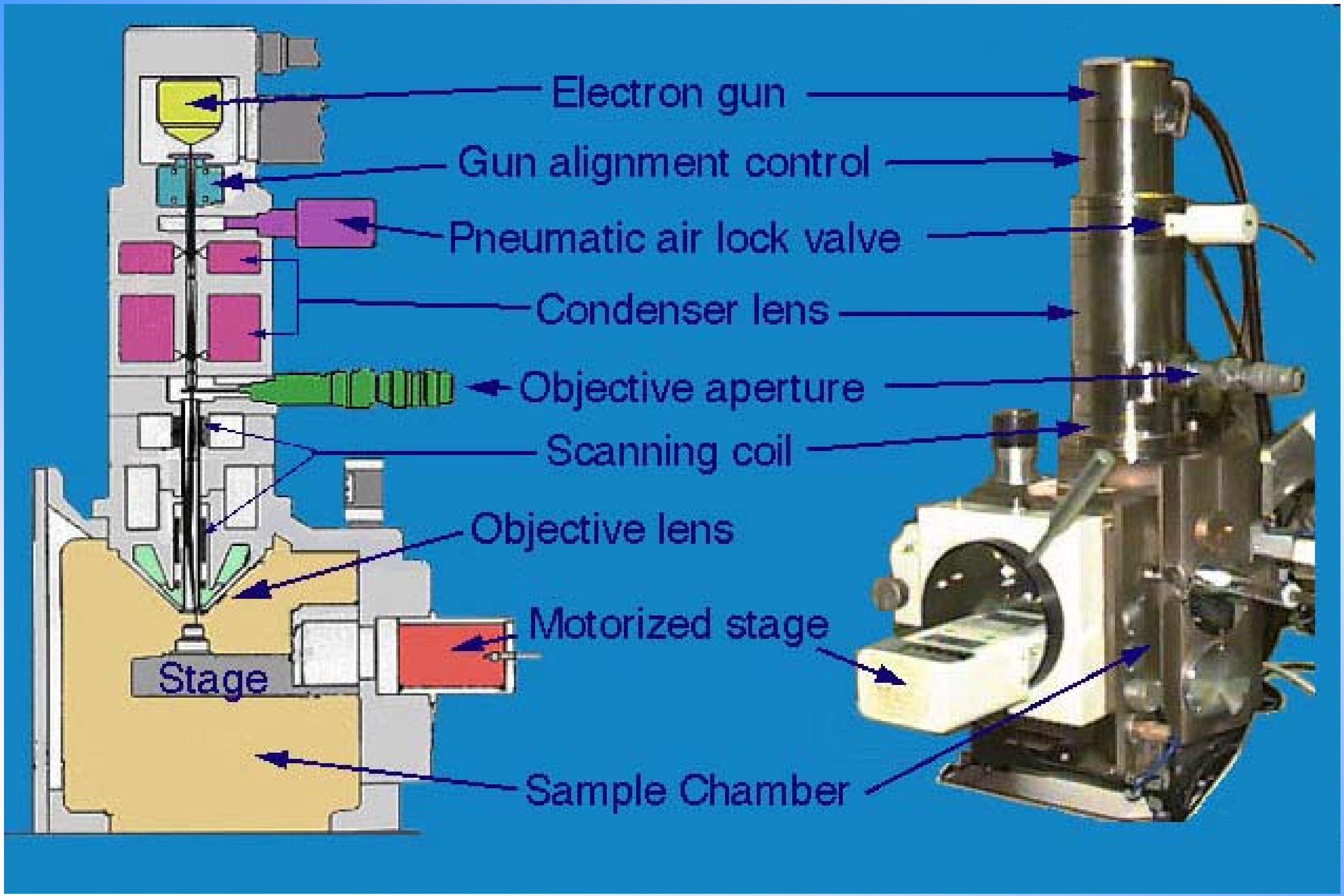
Cross sections for the various inelastic scattering processes in Al as function of the incident electron energy, assuming a small angle of scatter; plasmon (P), K and L-Shell ionization (K,L), fast and slow secondary electron generation (FSE, SE). For comparison purposes the elastic cross section (E) is also included.



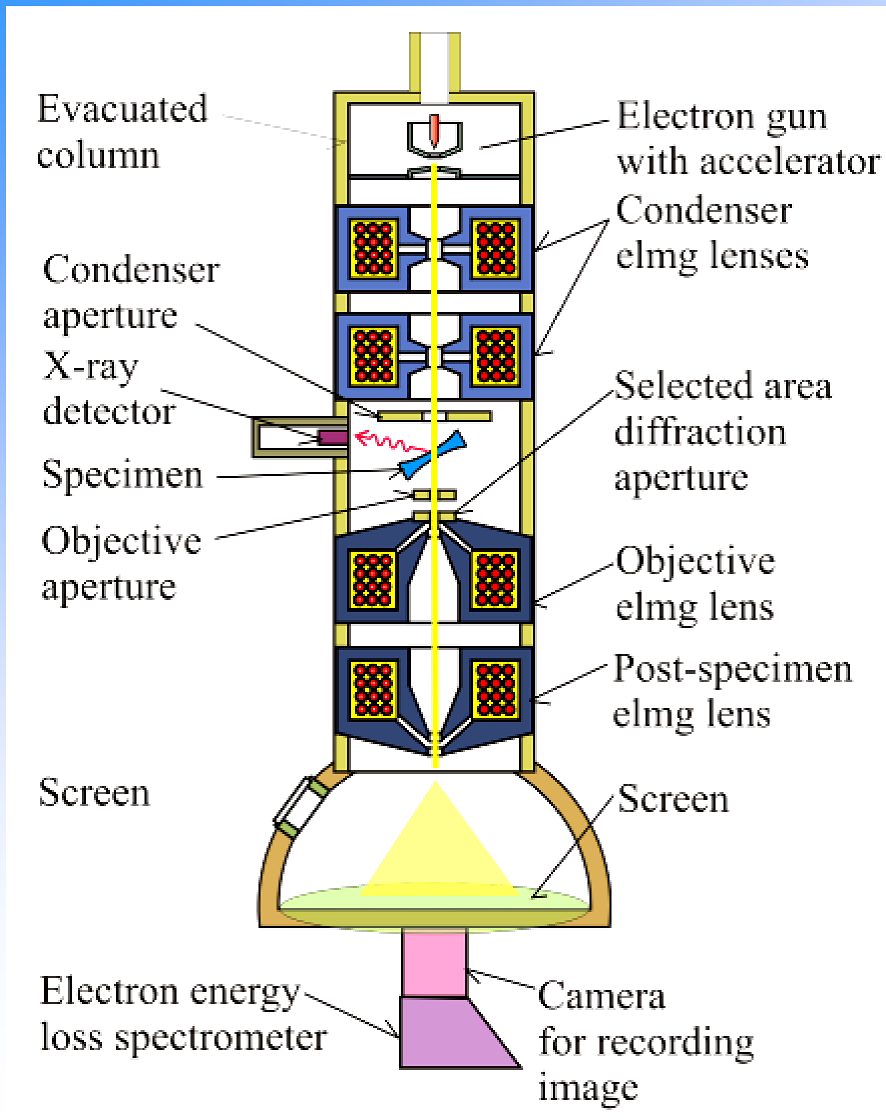
Scheme of Scanning Electron Microscope



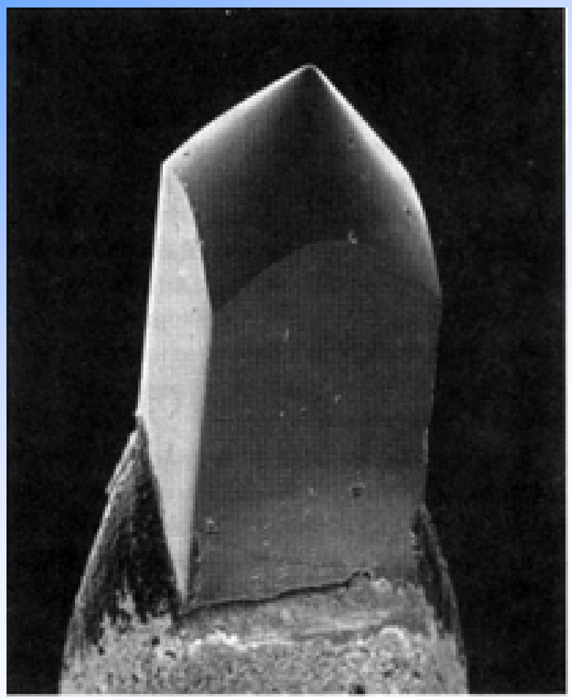
SEM: electron optical column



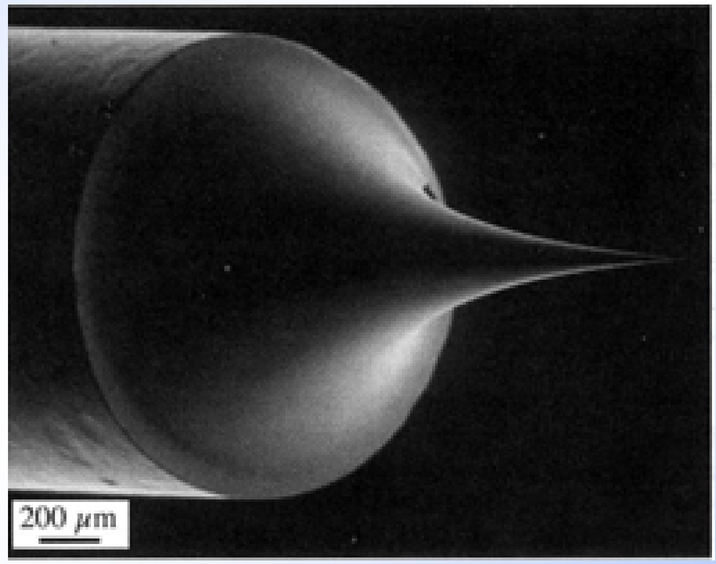
TEM is more complicated



Electron Sources



An LaB_6 crystal



An FEG tip, showing the extraordinarily fine W needle

LaB_6 Gun

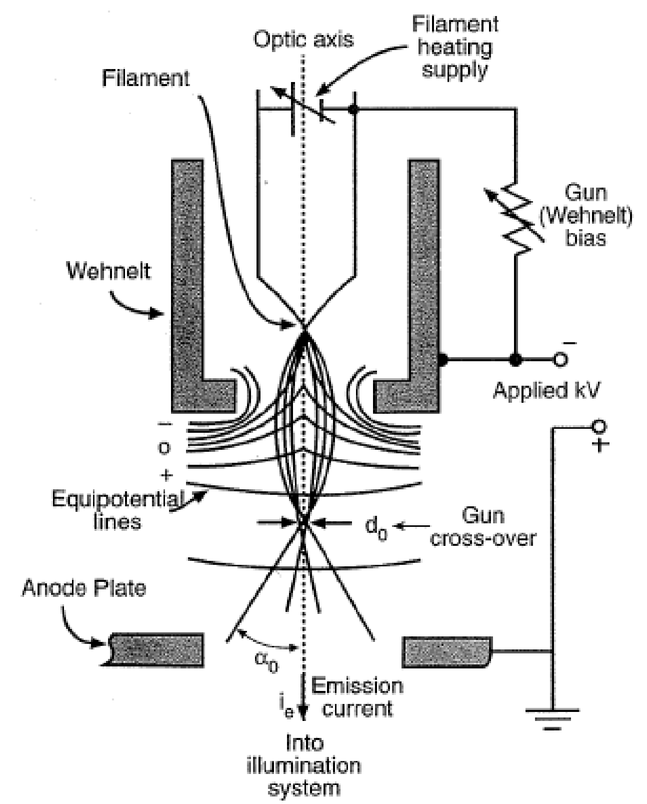
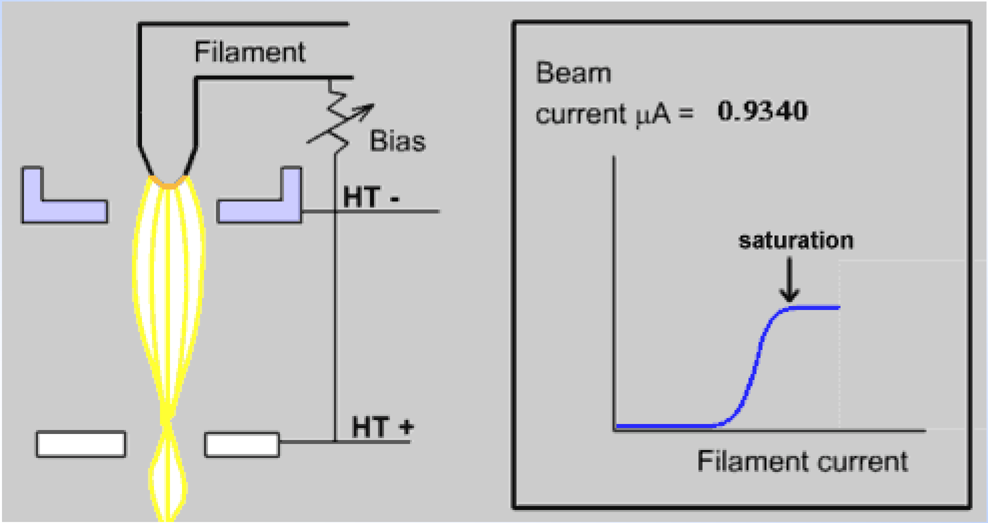


Figure 5.1. Schematic diagram of a thermionic electron gun. A high voltage is placed between the filament and the anode, modified by a potential on the Wehnelt which acts to focus the electrons into a crossover, with diameter d_0 and convergence/divergence angle α_0.

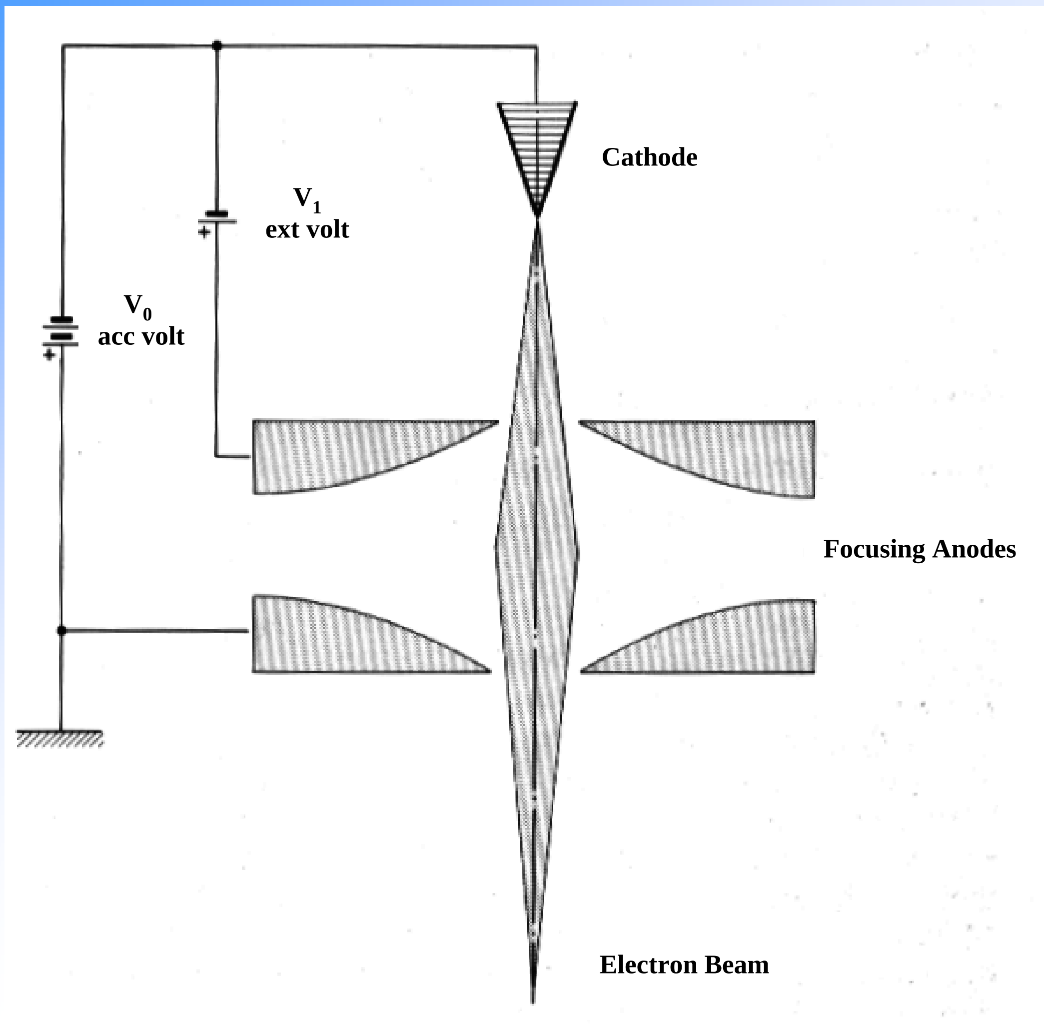


Probe diameter > 5 nm

Brightness 10^9 A/m^{2}ster

Vacuum required 10^{-4} Pa

Field Emission Gun



Probe diameter 1-2 nm

Brightness 10^{13} A/m^{2}ster

Vacuum required 10^{-8} Pa

SEM: General Information

Accelerating voltage 100 V – 30000 V

Resolution 0.7 nm – 5 nm (depends on the kind of electron gun)

Samples should be electrically conductive and

also be mounted electrically conductive on the holder

Size of sample is limited by the specimen chamber

Kind of Information

Topography

Morphology

Composition

Crystallographic structure

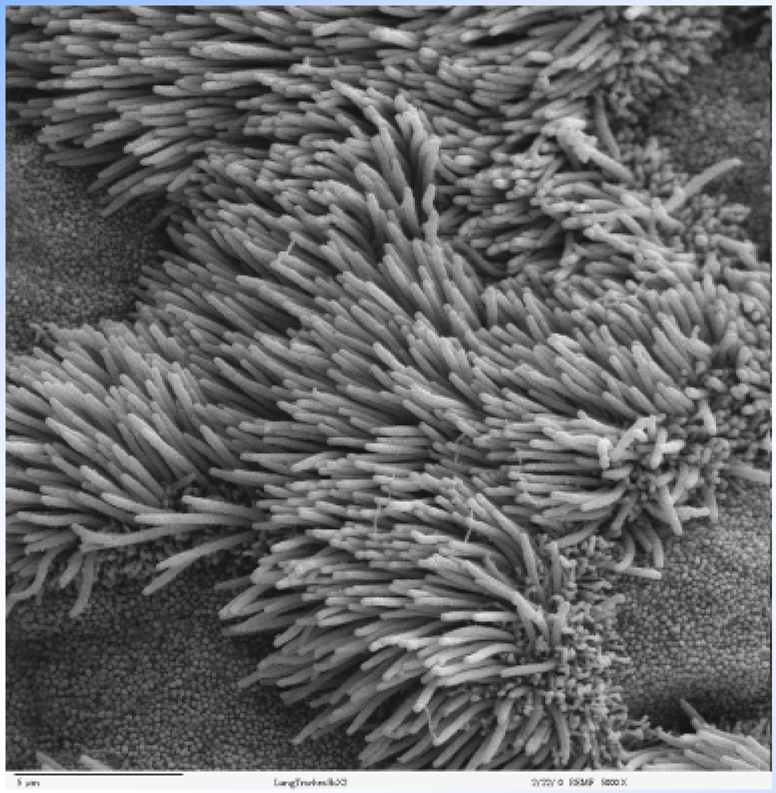
SEM: sample preparation

1) Remove all water, solvents, or other materials that could vaporize while in the vacuum.
2) Firmly mountall the samples.
3) Non-metallic samples, such as plants, fingernails, and ceramics, should be coated so they are electrically conductive.

An insect coated in gold, prepared for viewing with a scanning electron microscope.

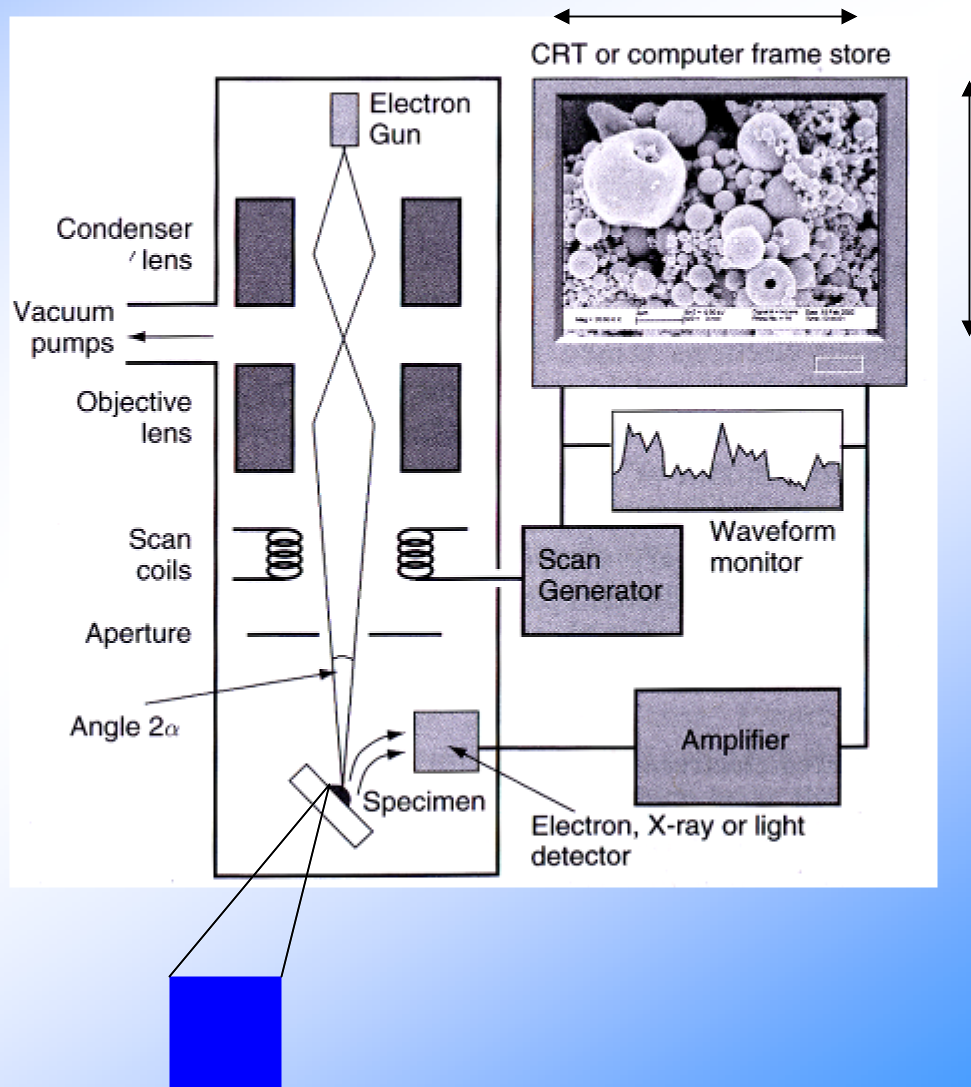
Secondary Electron Imaging (SEI)

- Secondary electrons are detected
- The mostly used operation mode of a SEM



SEM: Magnification

In a SEM, magnification results from the ratio of the dimensions of the raster on the specimen and the raster on the display device. Assuming that the display screen has a fixed size, higher magnification results from reducing the size of the raster on the specimen, and vice versa. Magnification is therefore controlled by the current supplied to the x, y scanning coils,, and not by objective lens power.



SEM: Resolution

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- 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 which produces the scanning beam.
- The resolution is also limited by the size of the interaction volume, or the extent to which the material interacts with the electron beam.
- The spot size and the interaction volume are both large compared to the distances between atoms, so the resolution of the SEM is not high enough to image individual atoms

Resolution of a TEM

$$q_{max} = \frac{1}{0.67(C_s\lambda^3)^{\frac{1}{4}}}.$$

SEM: Information depth

Information

of

surface

and

bulk

Penetration depth of PE

and

size of X-ray resolution

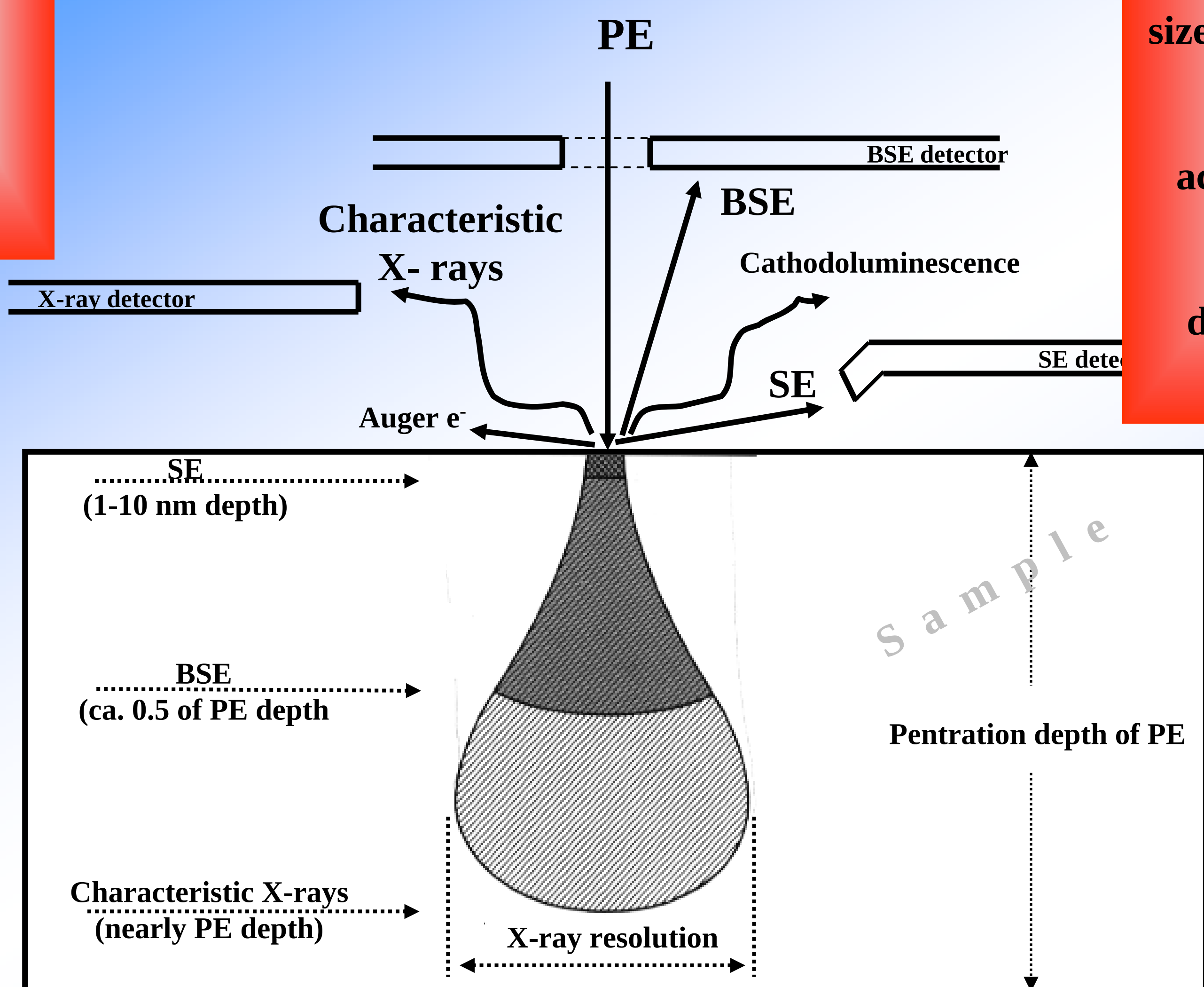
depend on

accelarating voltage

and

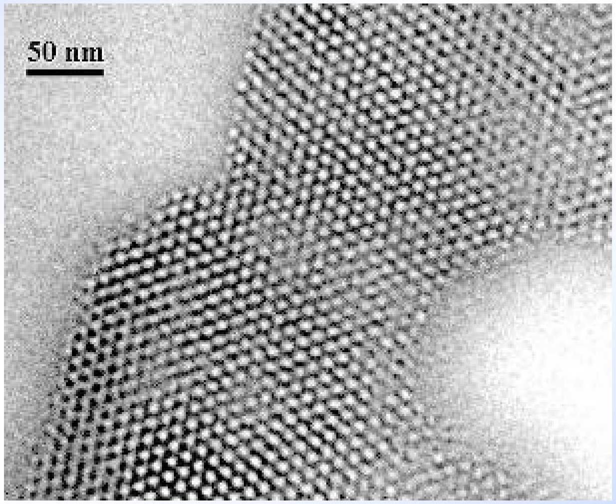
density of material

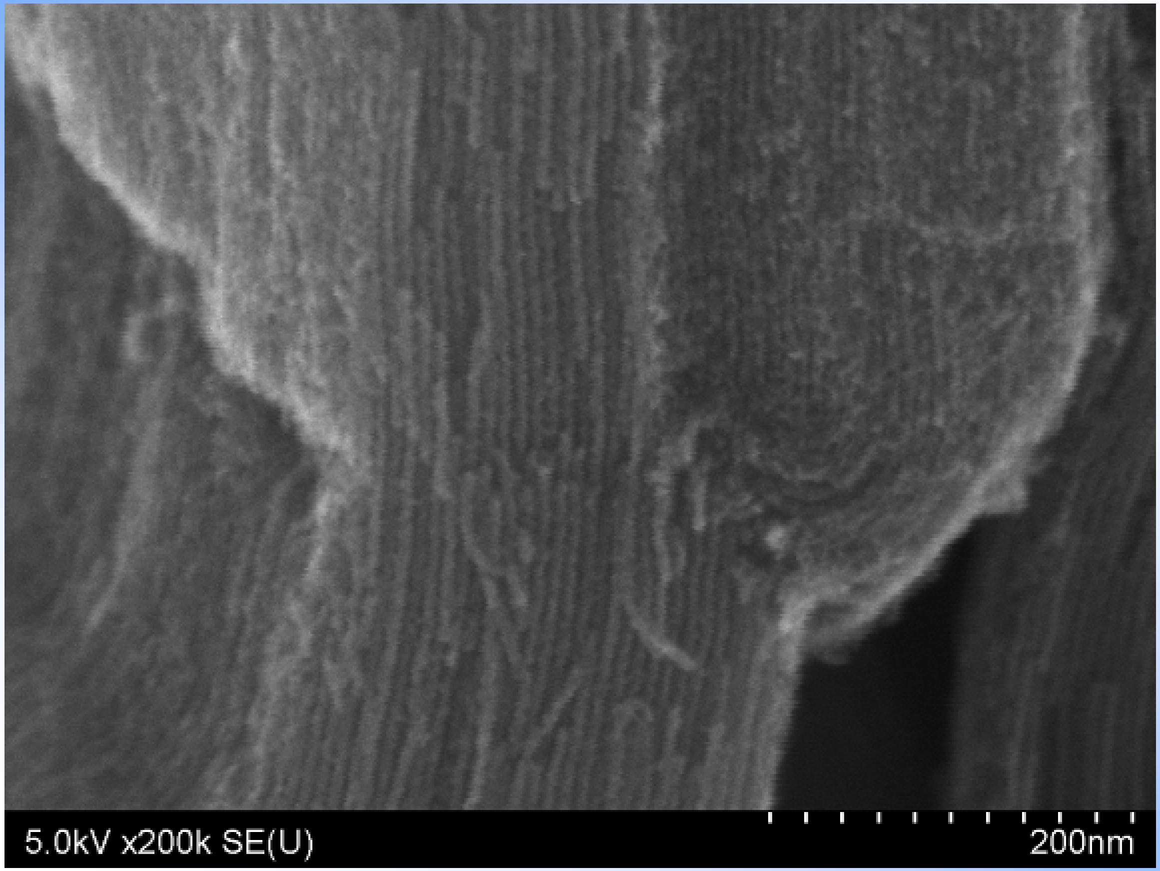
$R=100 \cdot E^{1.43}/\rho$



Secondary Electron Imaging (SEI)

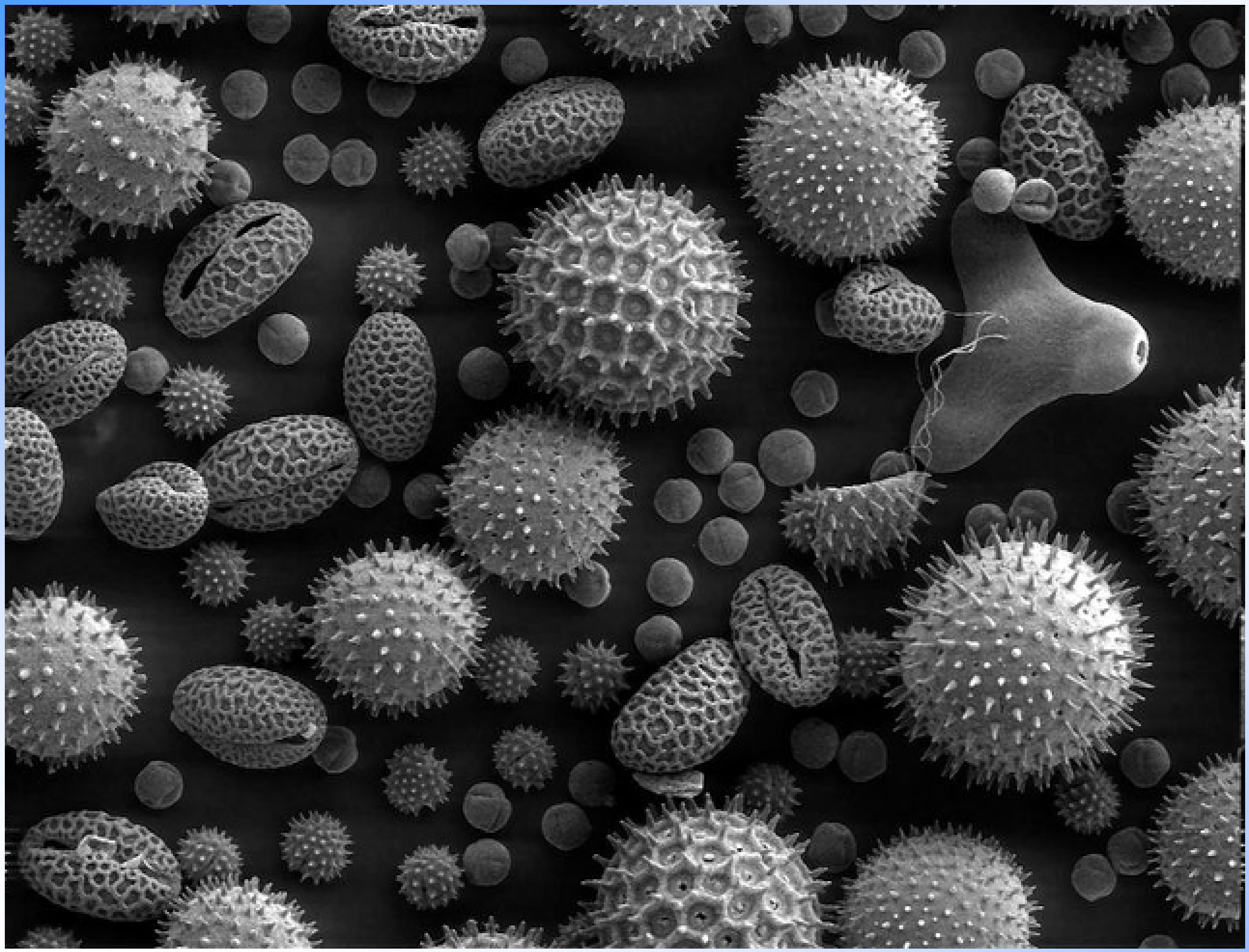
- **Resolution**





Secondary Electron Imaging (SEI)

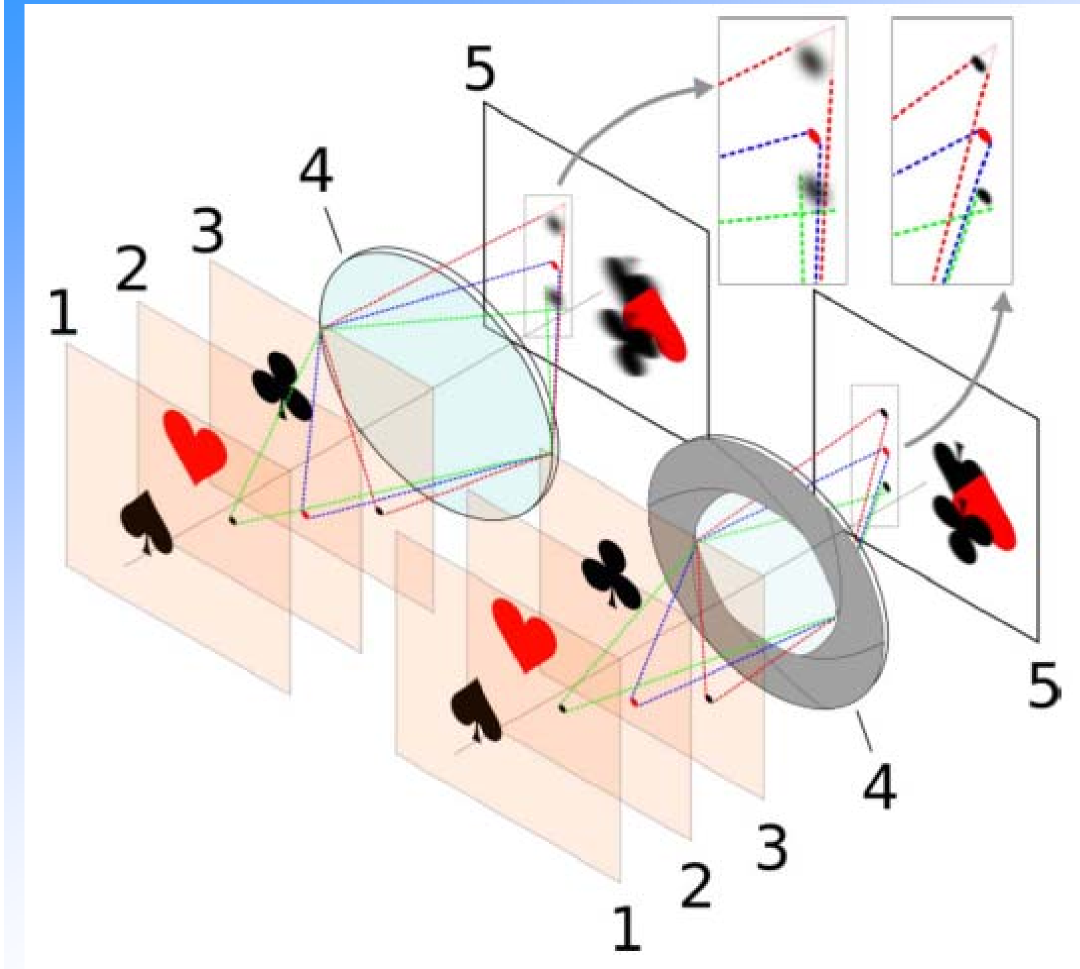
- **Why 3 D appearance ?**



Due to the very narrow electron beam, SEM micrographs have a large depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample.

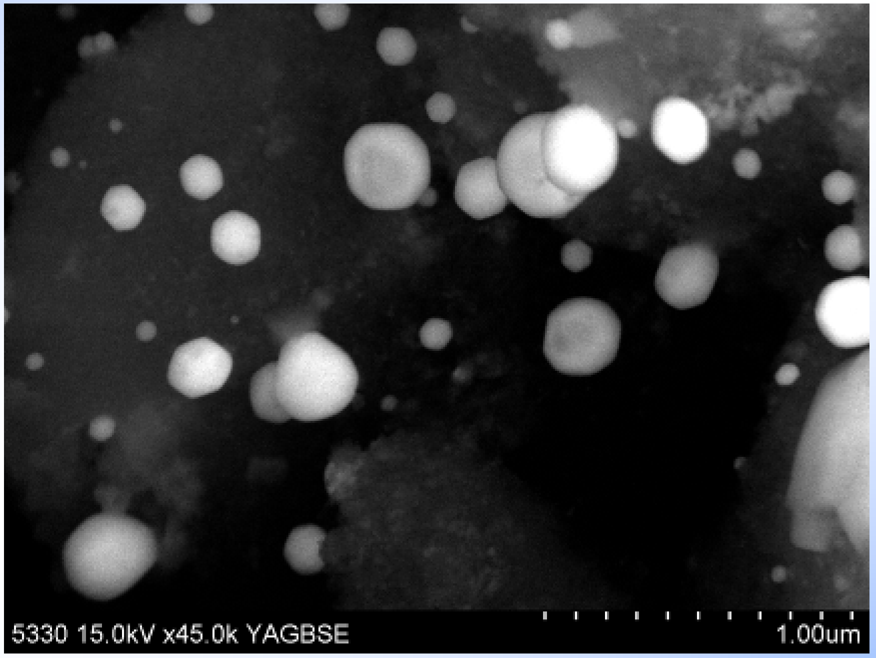
Pollen grains taken on an SEM show the characteristic depth of field of SEM micrographs

Depth of field



Imaging using Back Scattered Electrons (BSE)

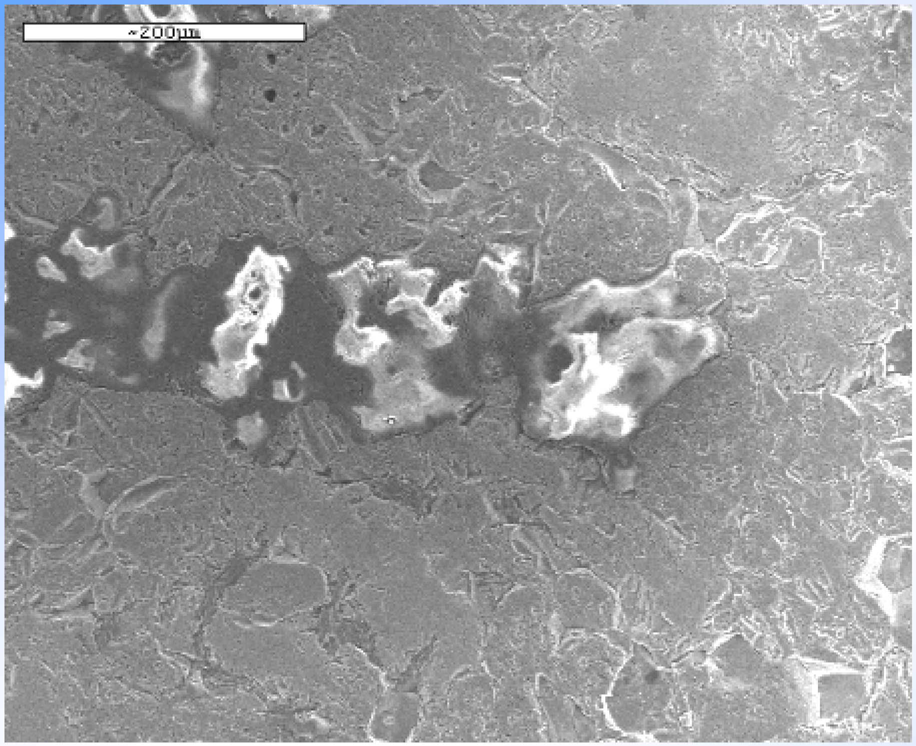
$$\sigma(\theta) \sim Z^2$$



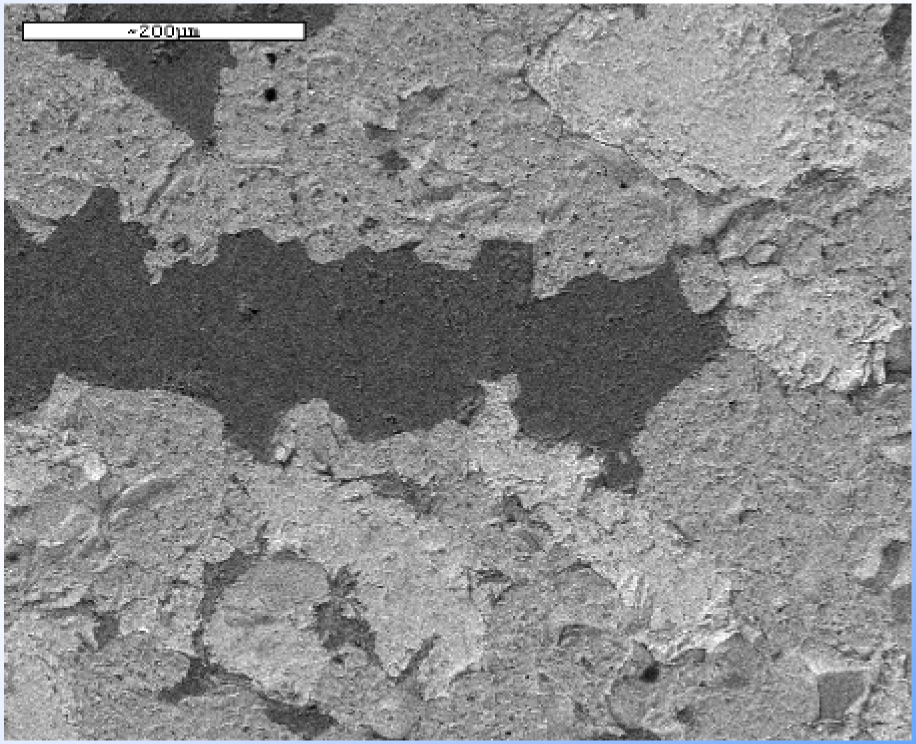
BSE image of BMW catalyst, bright particle being Pd.

SE image – BSE image

Mineral

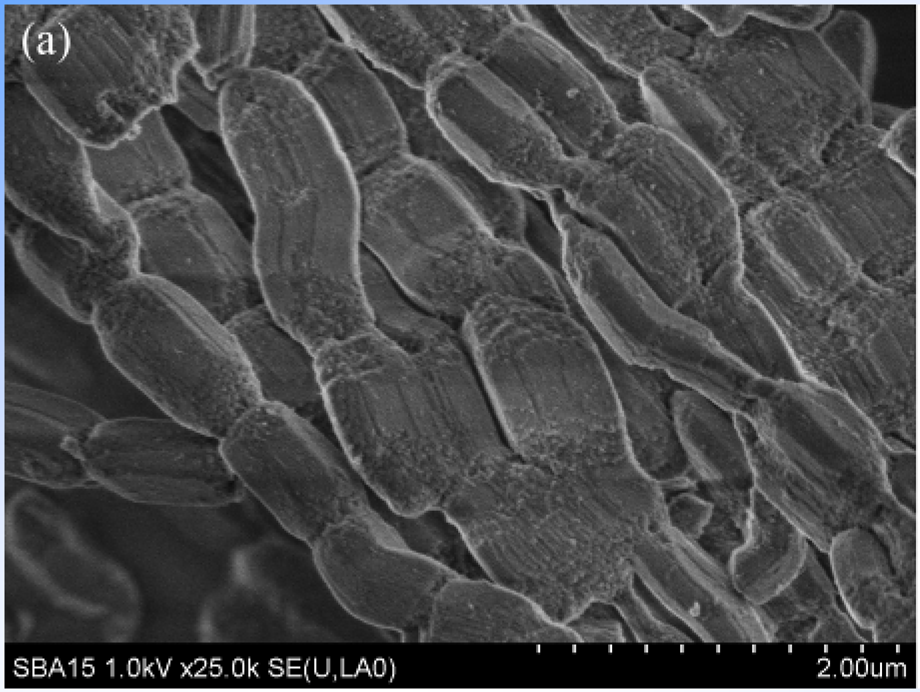


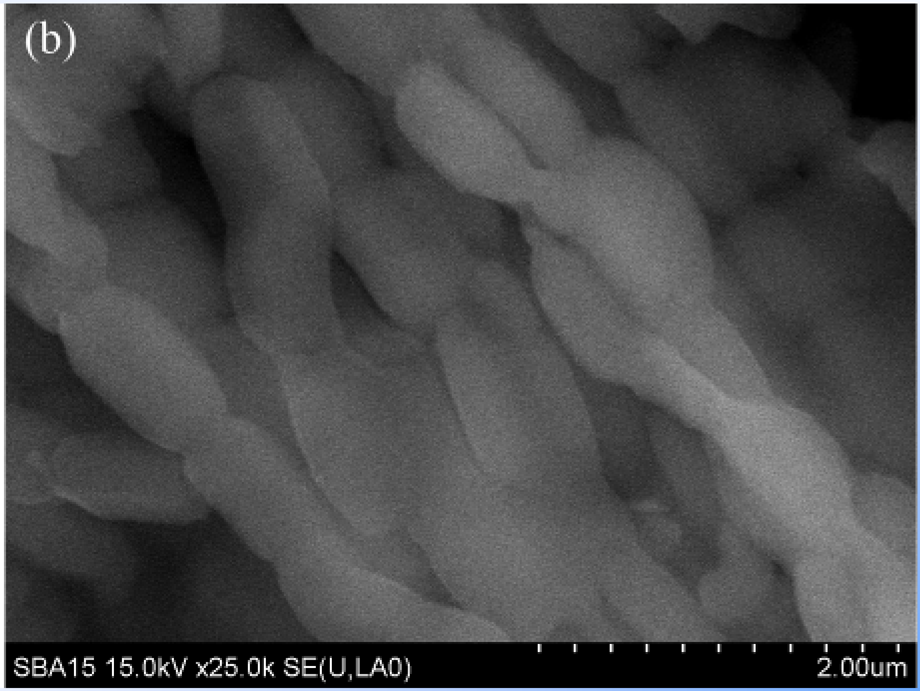
020823 **SE** **HV15kV**



020824 **BSE** **HV15kV**

• SBA-15



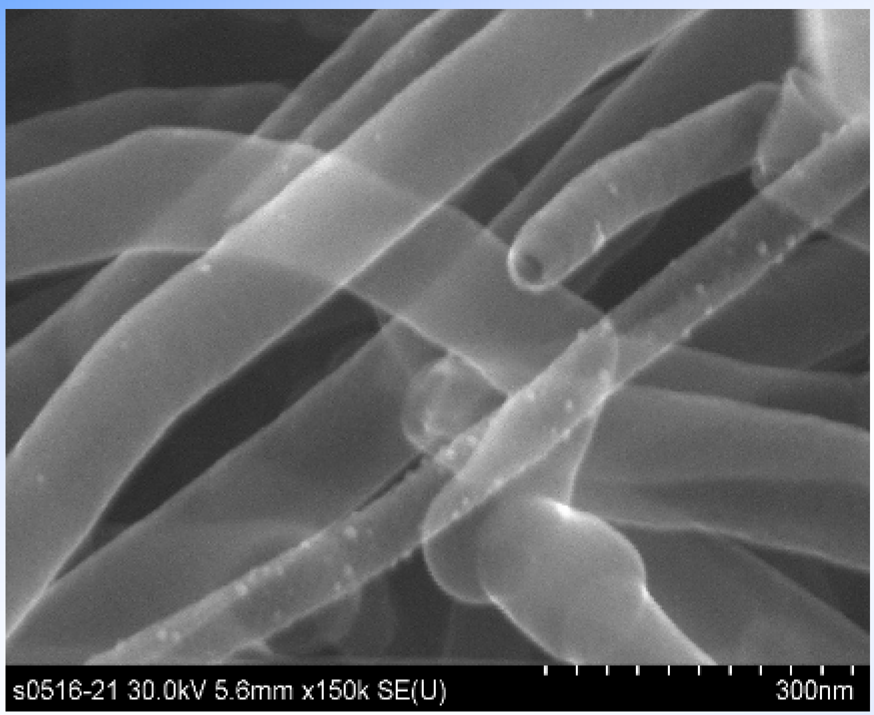


1 kV

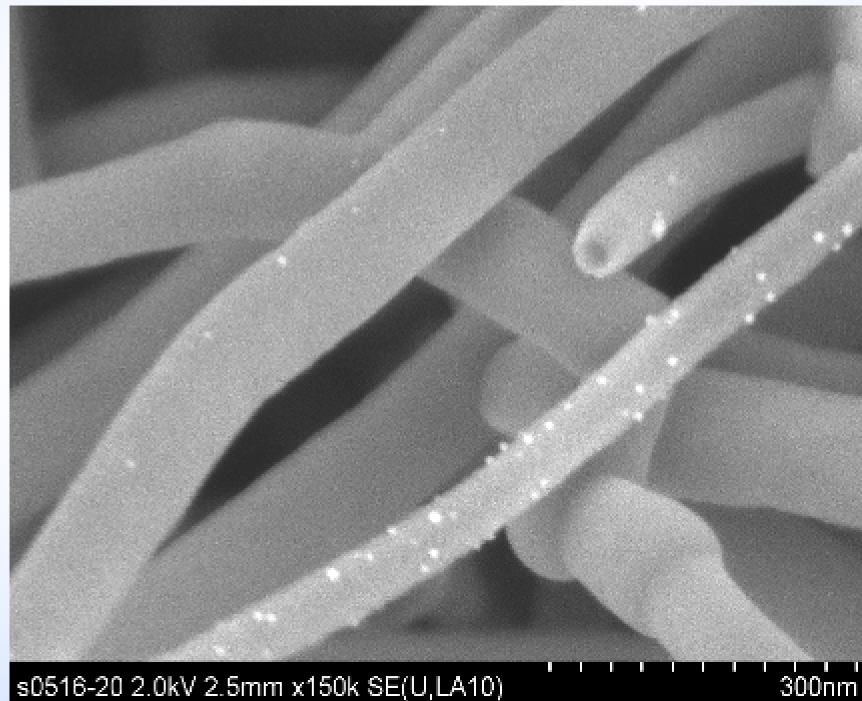
15 kV

Changing the high voltage of the SEM....

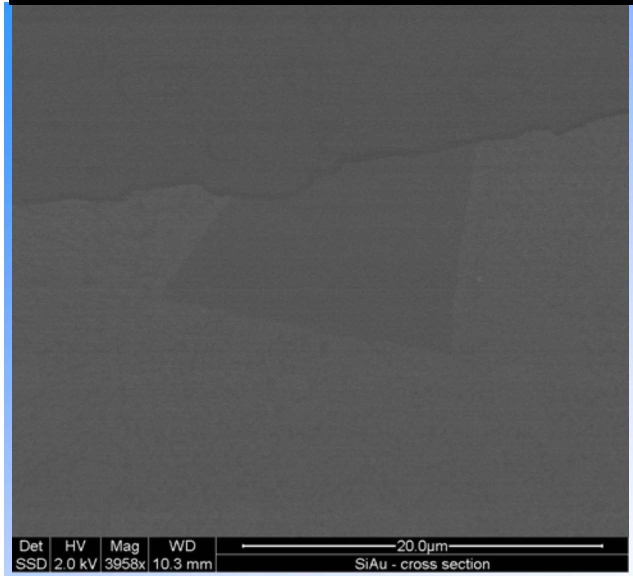
SE, HV 30 kV

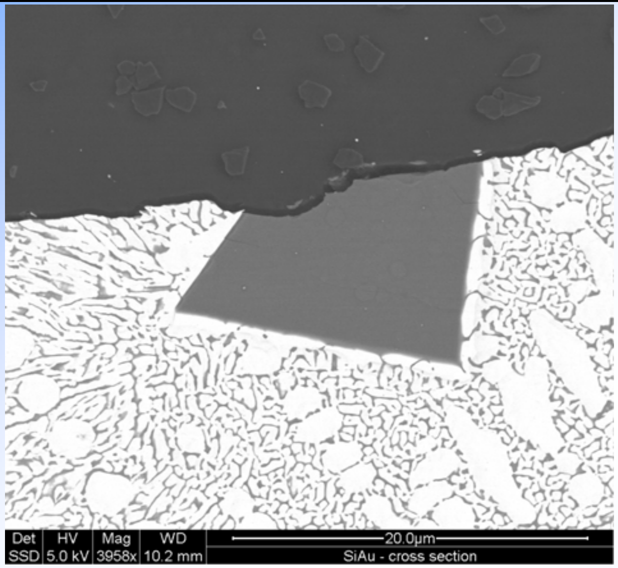


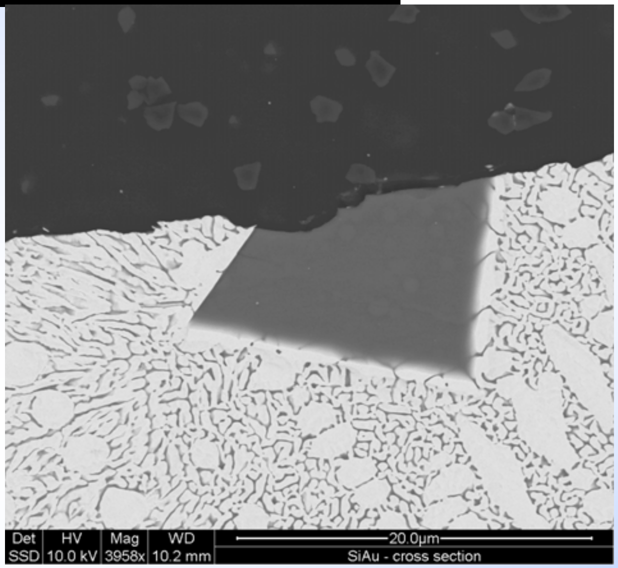
SE, HV 2 kV

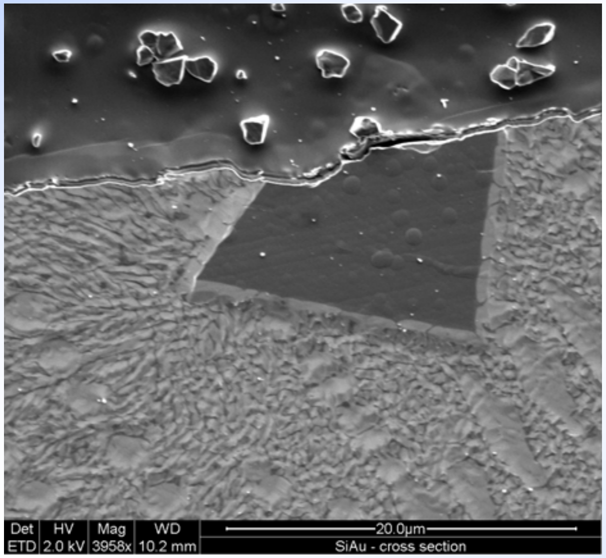


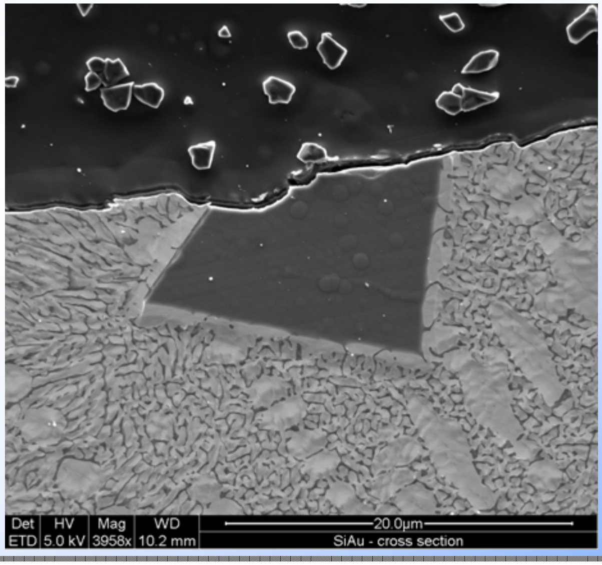
Influence of acc voltage

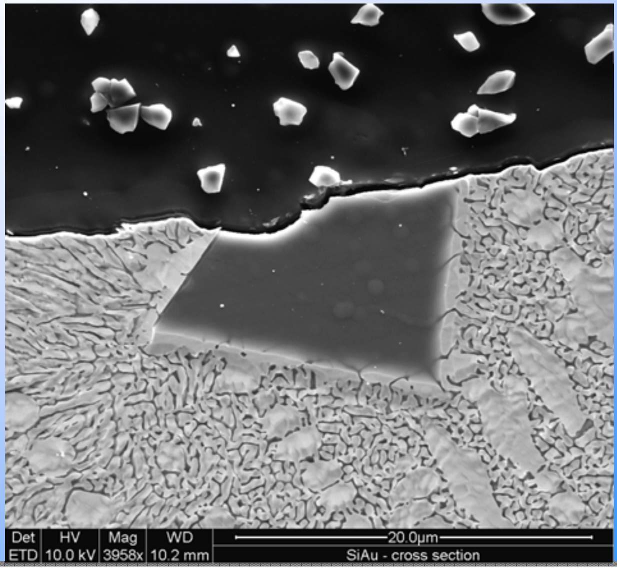












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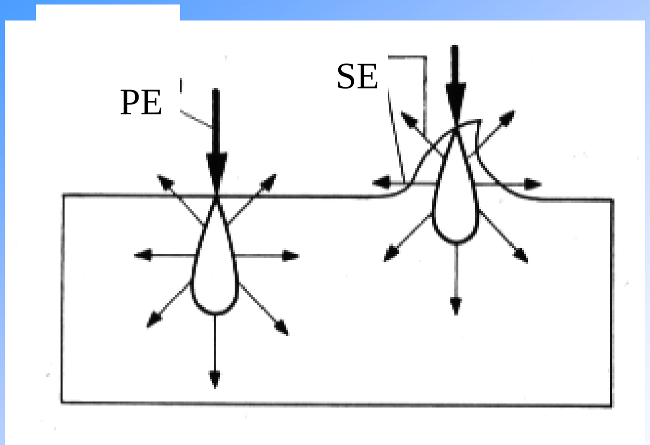
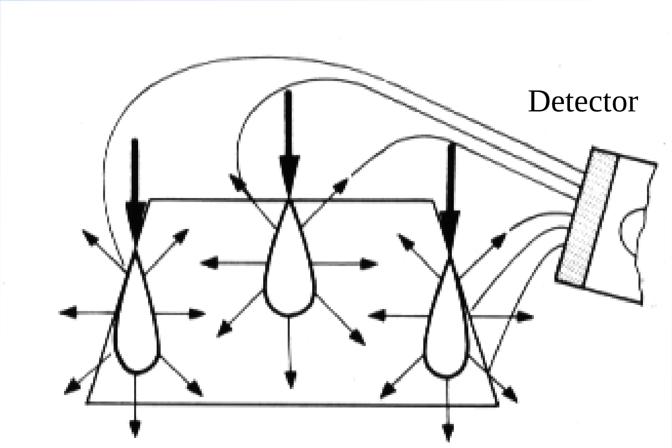
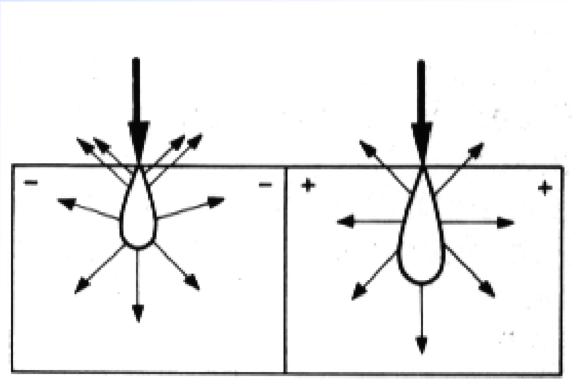
SEM: Contrast Mechnisms



Effect of topography
spikes and edges are brighter than plane surfaces

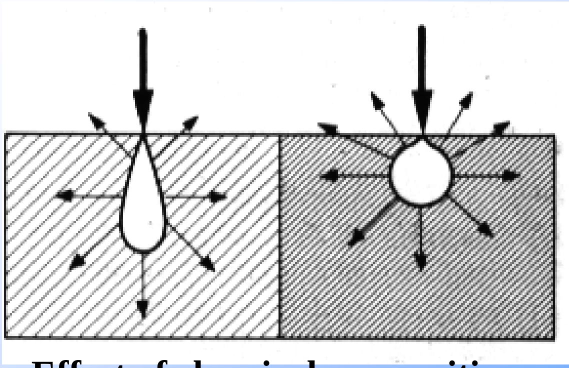


Effect of position relative to the detetector
a surface turned towards the detector is brighter than one turned away from it



Effect of electrical charge

negatively charged points are brighter than positively charged ones

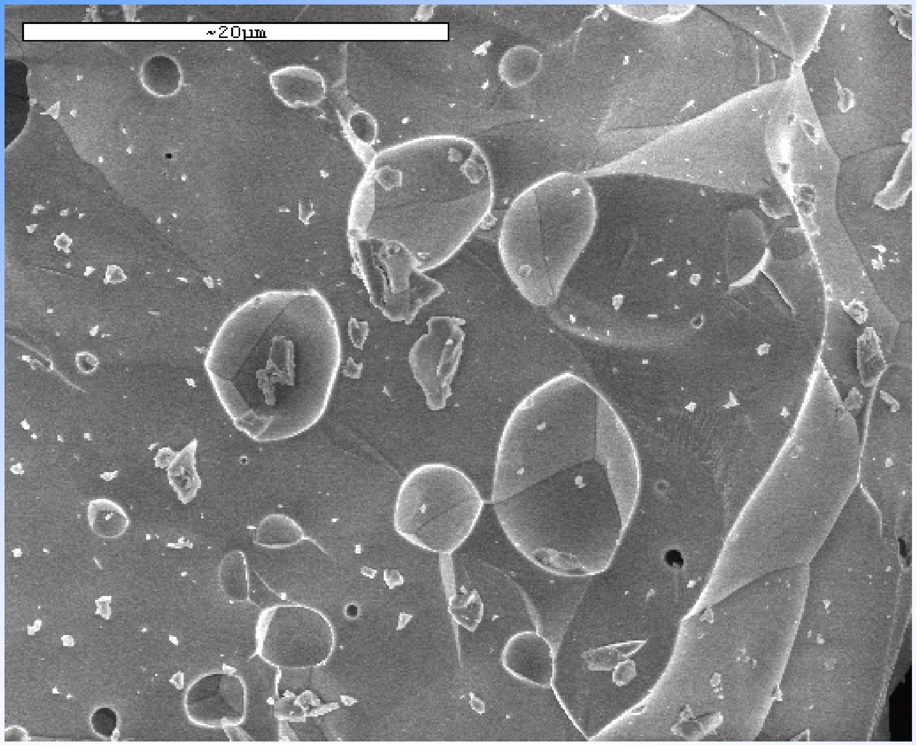


Effect of chemical composition

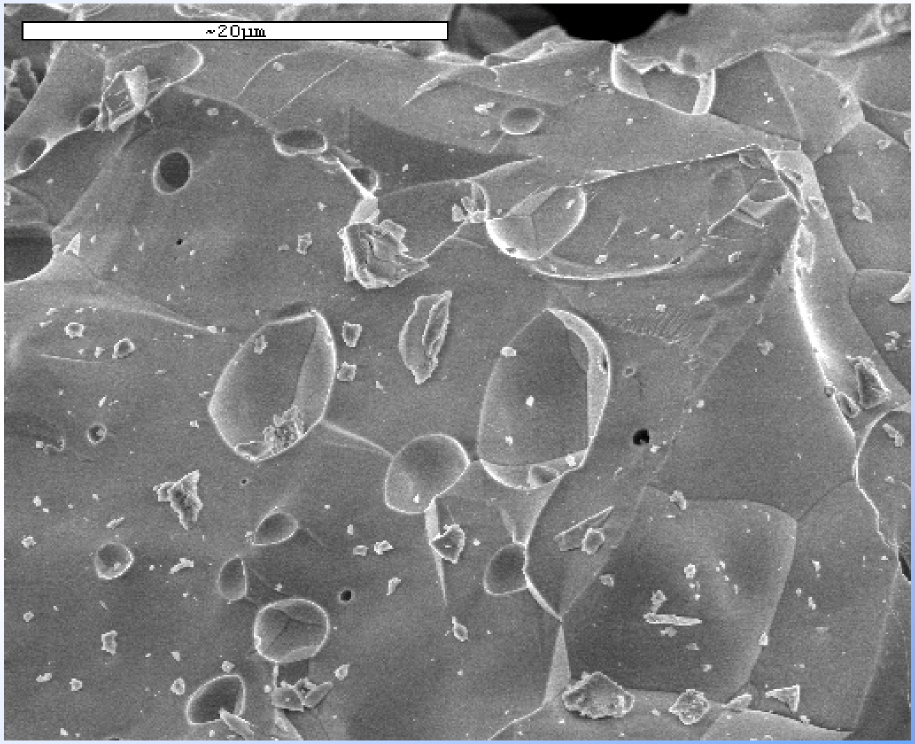
regions of heavy elements are brighter than those of light ones

SEM: Effect of topography

$La_{0.8}Sr_{0.2}Ga_{0.85}Mg_{0.15}O_{2.825}$



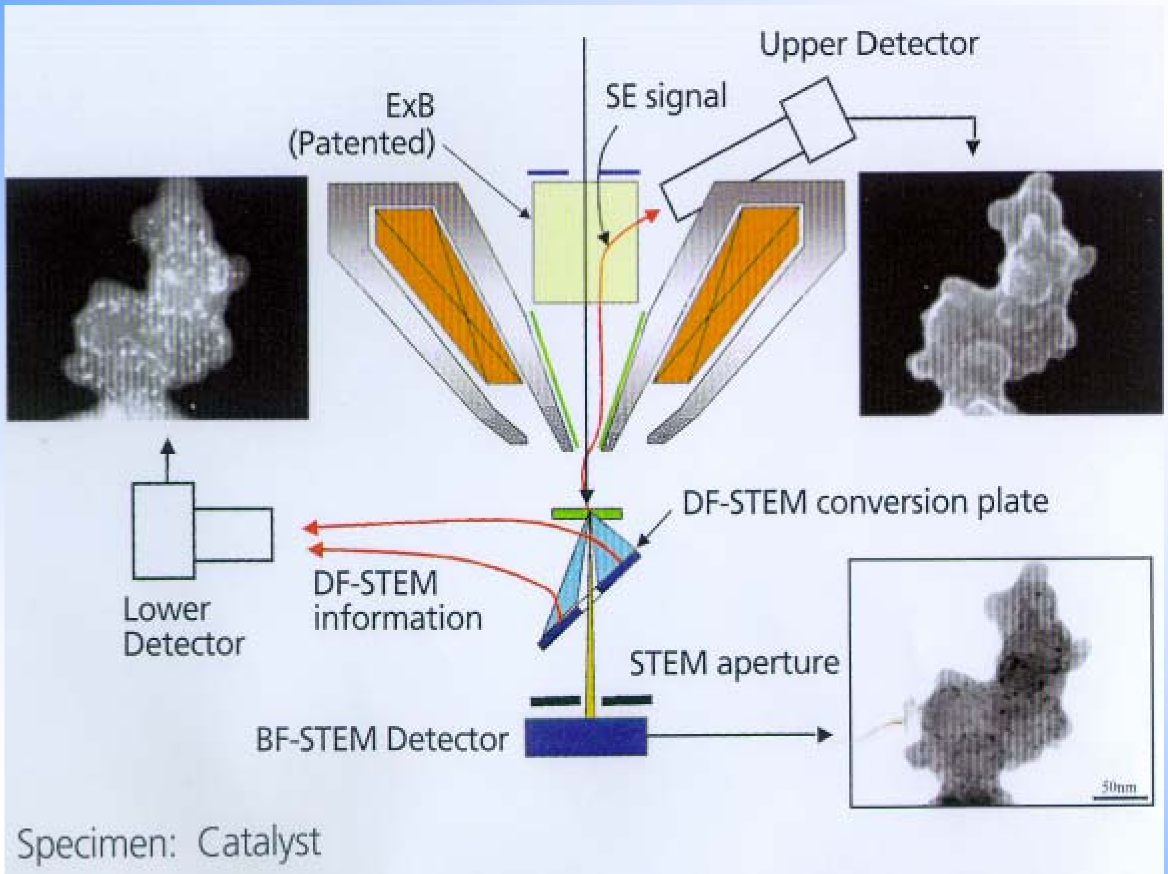
020293 **<0°** **HV 10kV**



020294 **<45°** **HV 10kV**

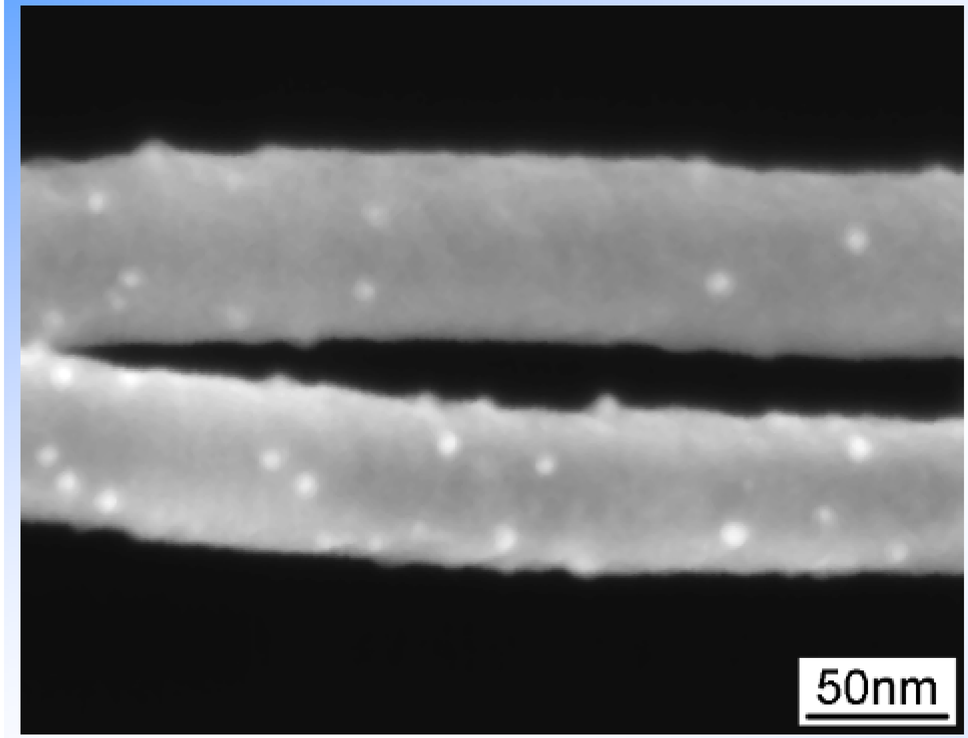
Tilting

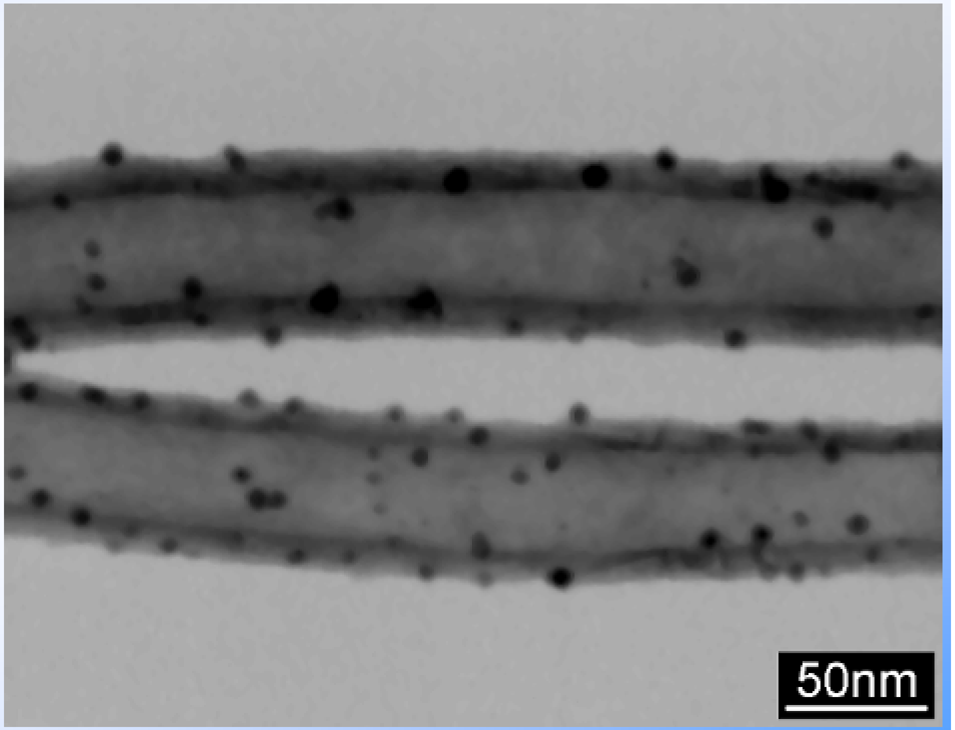
New SEM: scanning and transmission



New SEM: scanning and transmission

- Ni/CNTs





Chemical Composition

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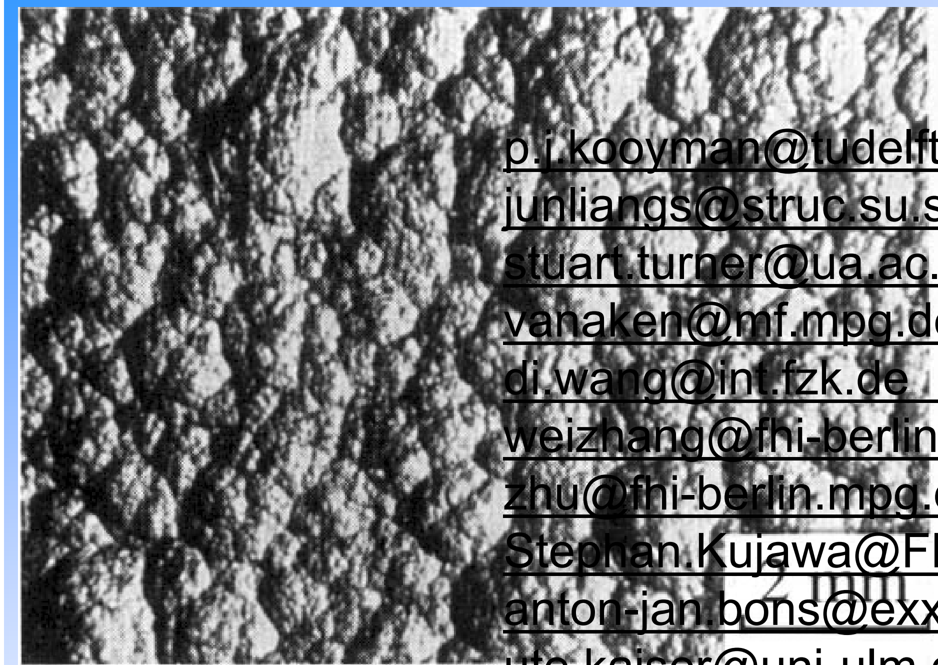

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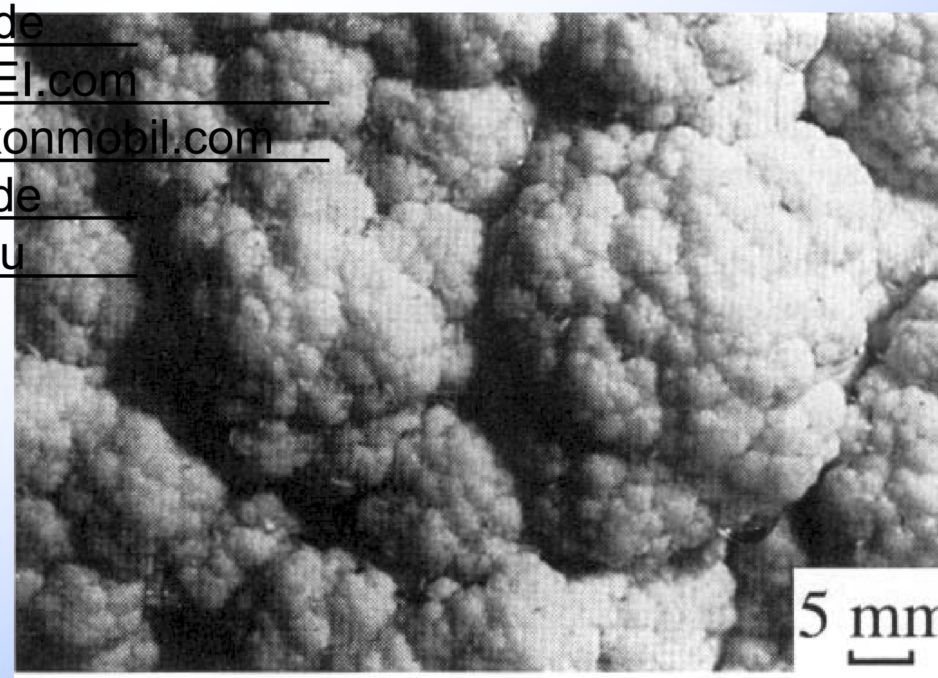
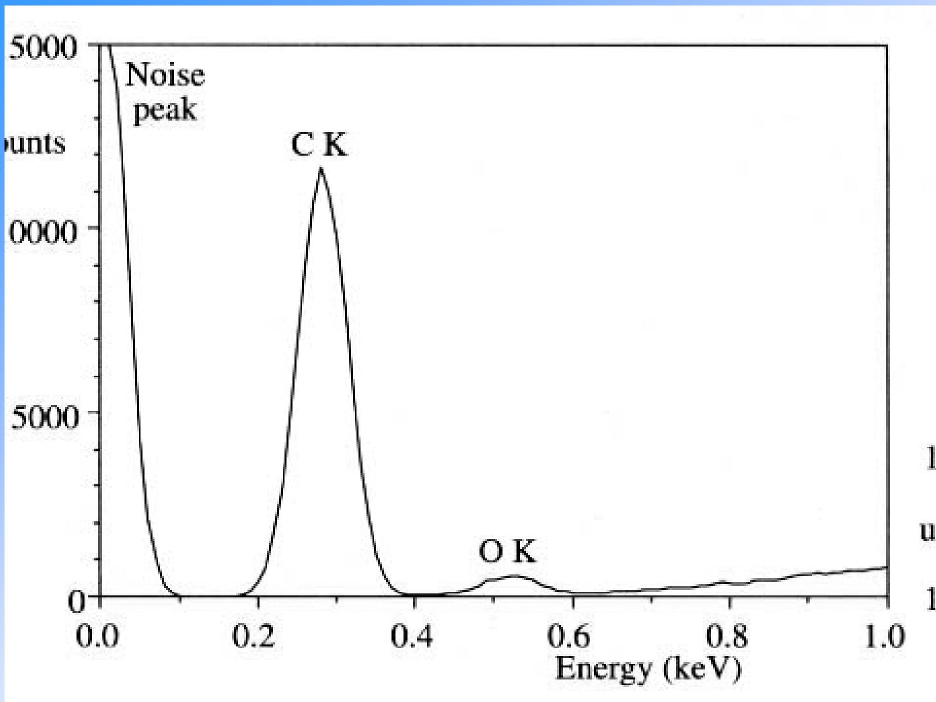
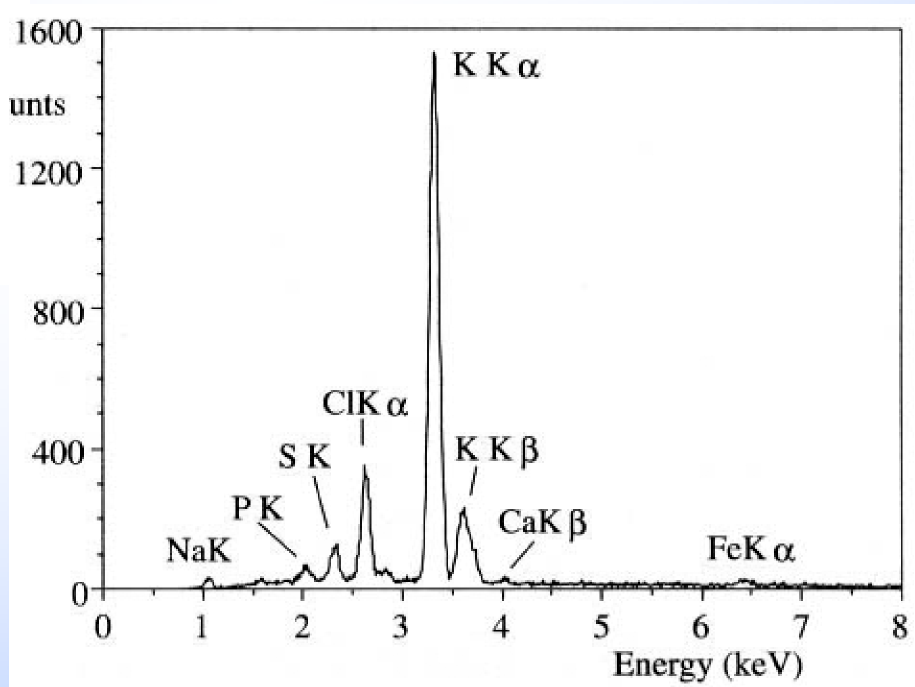
specht@berkeley.edu



Chemical Composition: EDX

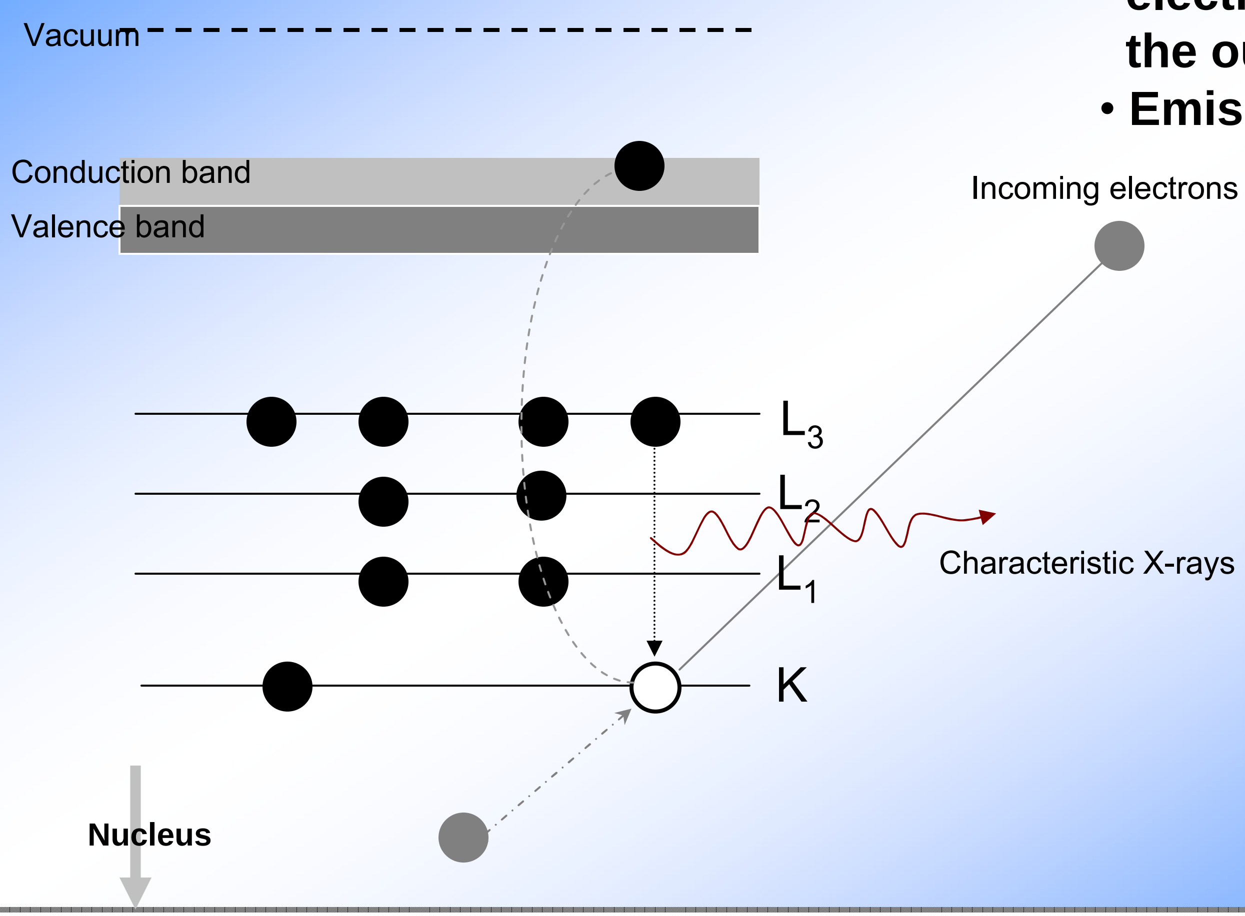




X-ray emission of atomic electrons

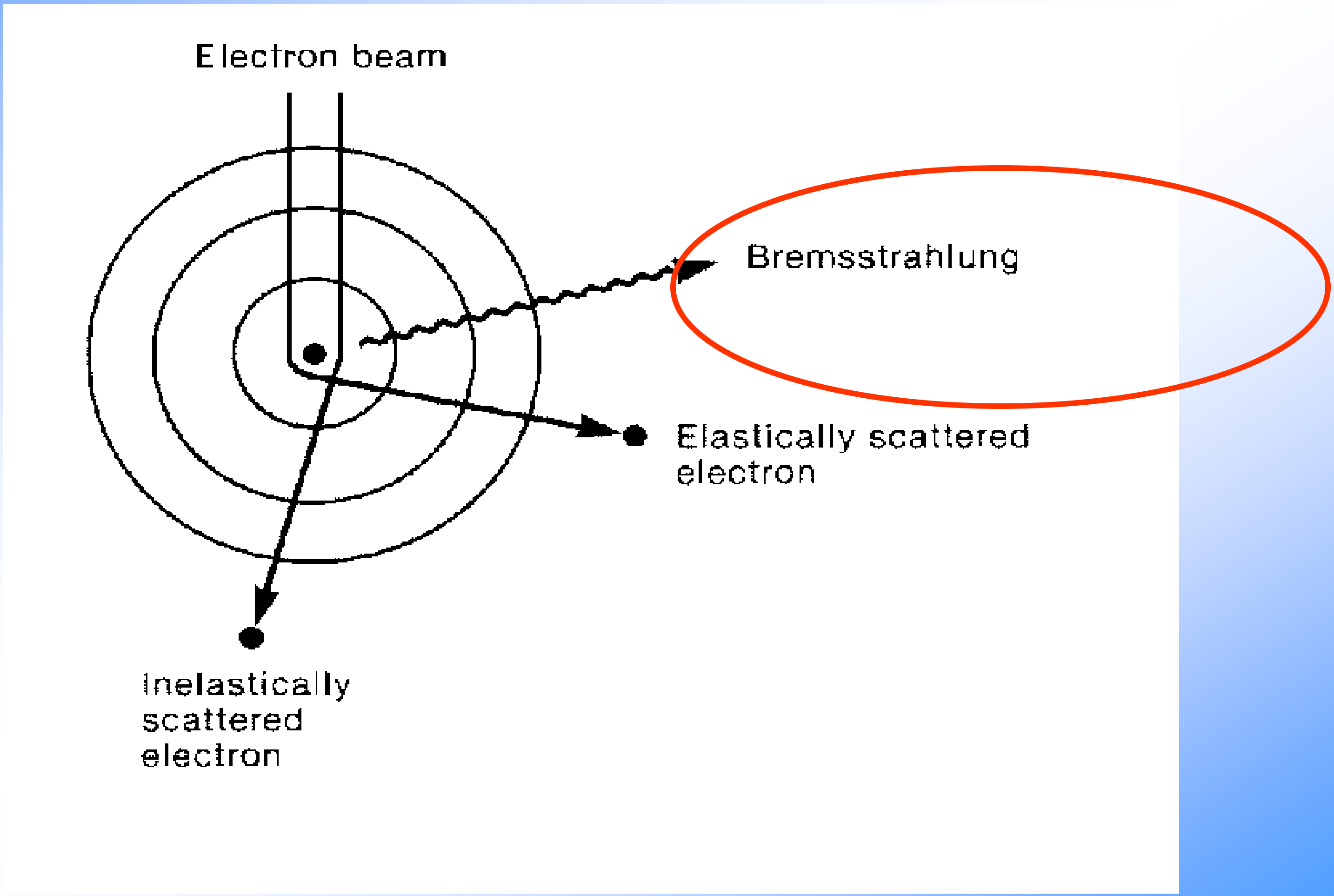
Characteristic X-rays

- Ionization
- Filling in the missing electron with one from the outer shells.
- Emission of X-rays or Auger e$^-$



Inelastic scattering of electrons

Bremsstrahlung

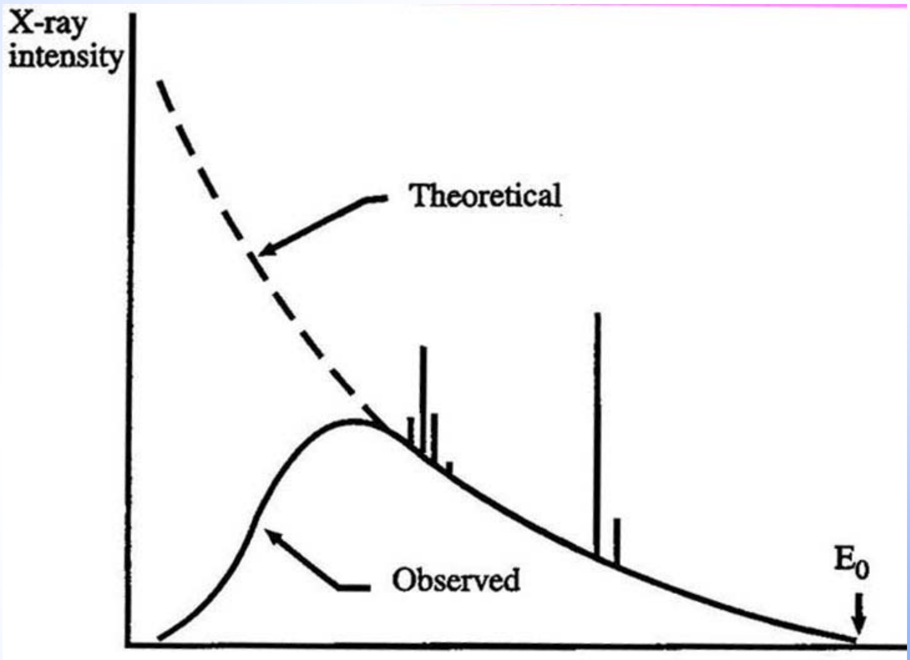


Bremsstrahlung X-rays

If an electron is decelerated by the Coulomb field of the nucleus while penetrating completely through the electron shell, it emit an X-ray.

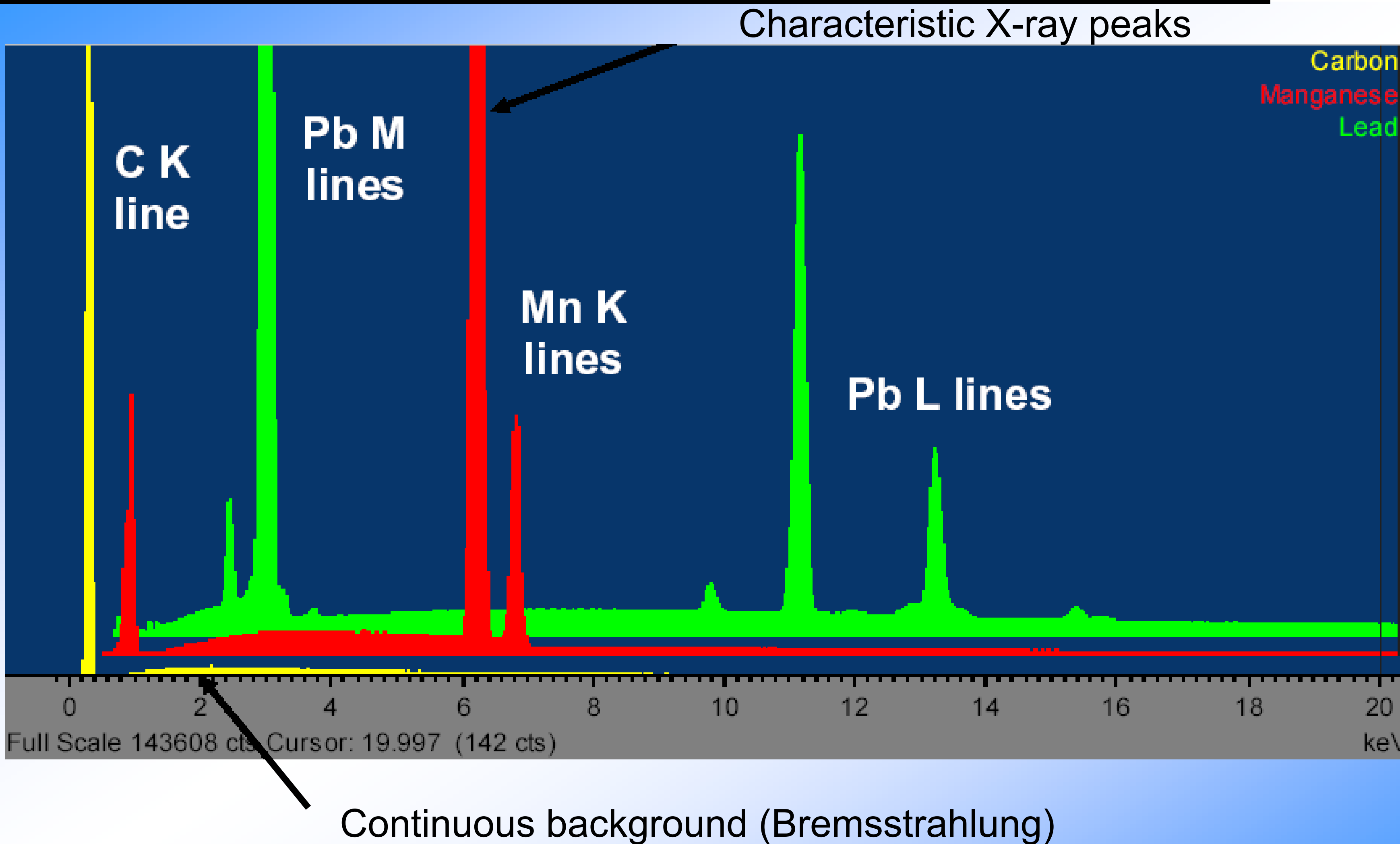
$$N(E) = \frac{KZ(E_0 - E)}{E}$$

N(E): number of bremsstrahlung photon of energy E, produced by electrons of Energy E_0.

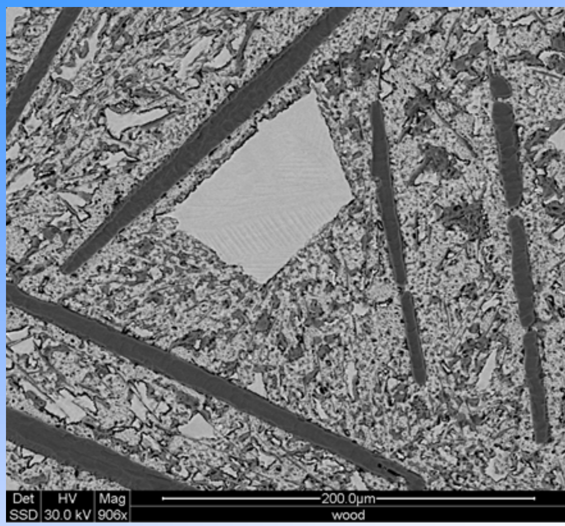


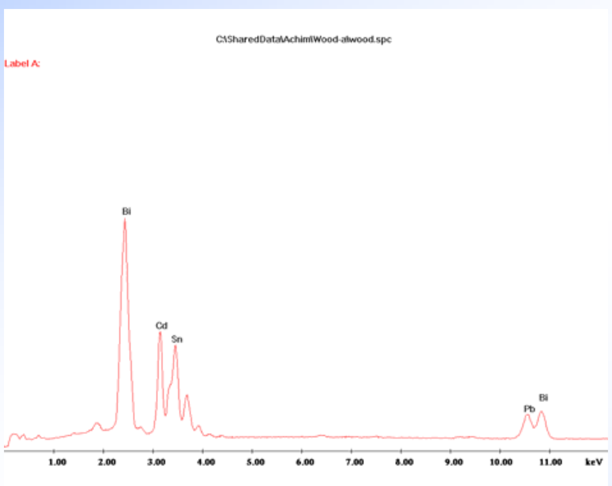
The bremsstrahlung X-ray intensity as a function of energy. The generated intensity increases rapidly at low energies but Very low energy bresstrahlung is absorbed in the specimen And the detector, so the observed intensity drops to zero. E_0 is the energy of the electrons that cause the X-ray emission.

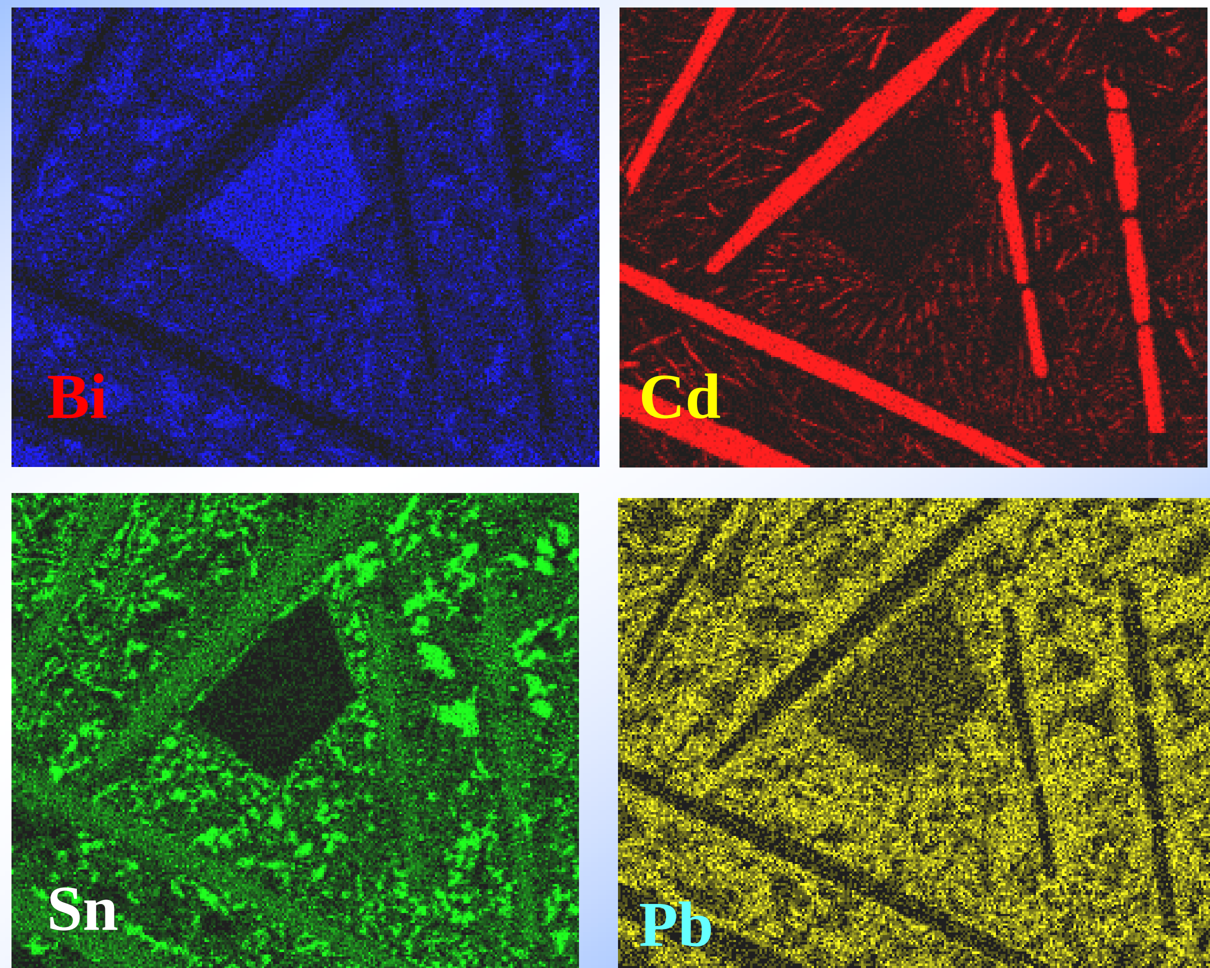
The EDX Spectrum



Localization of the element distribution

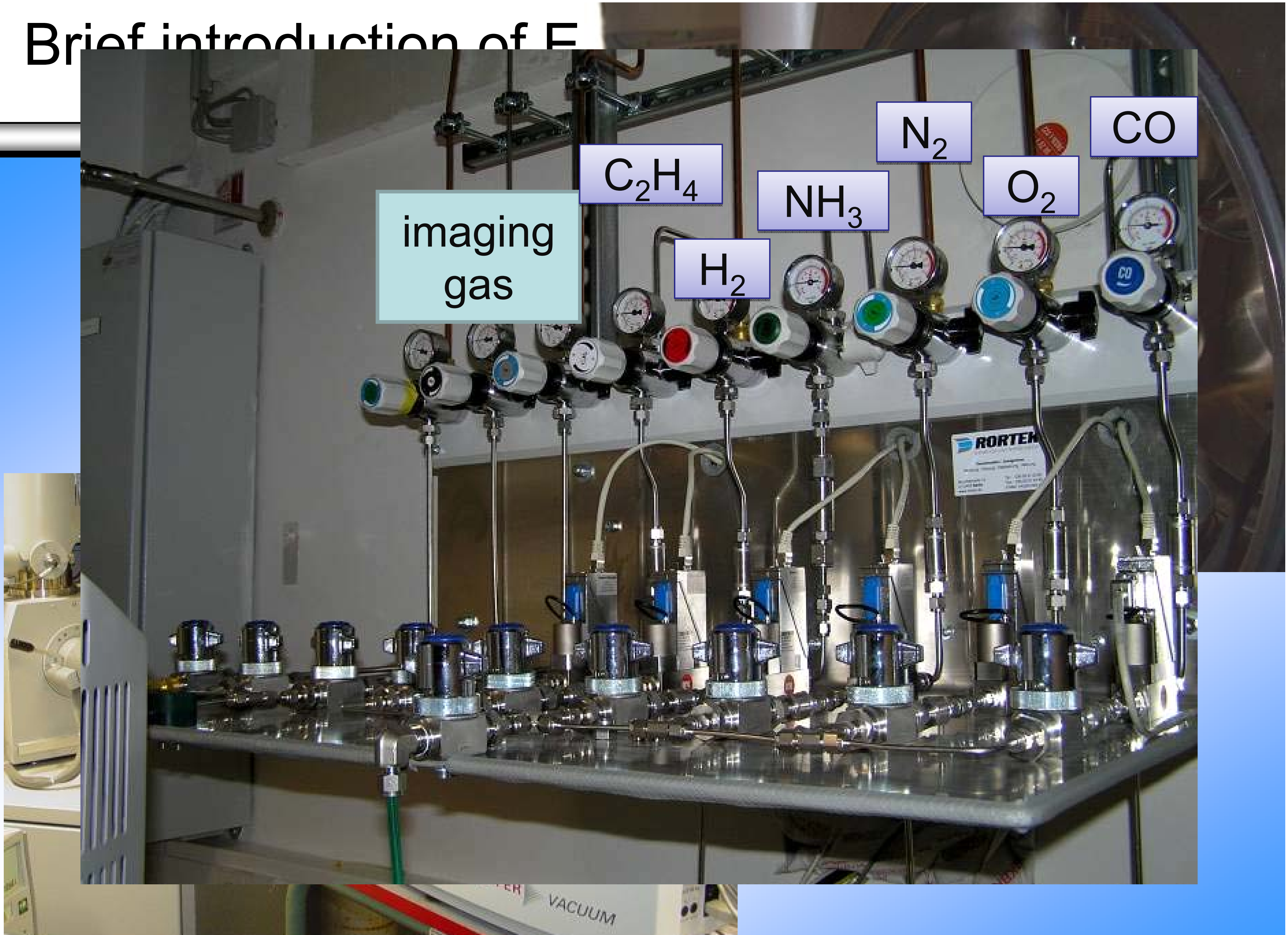




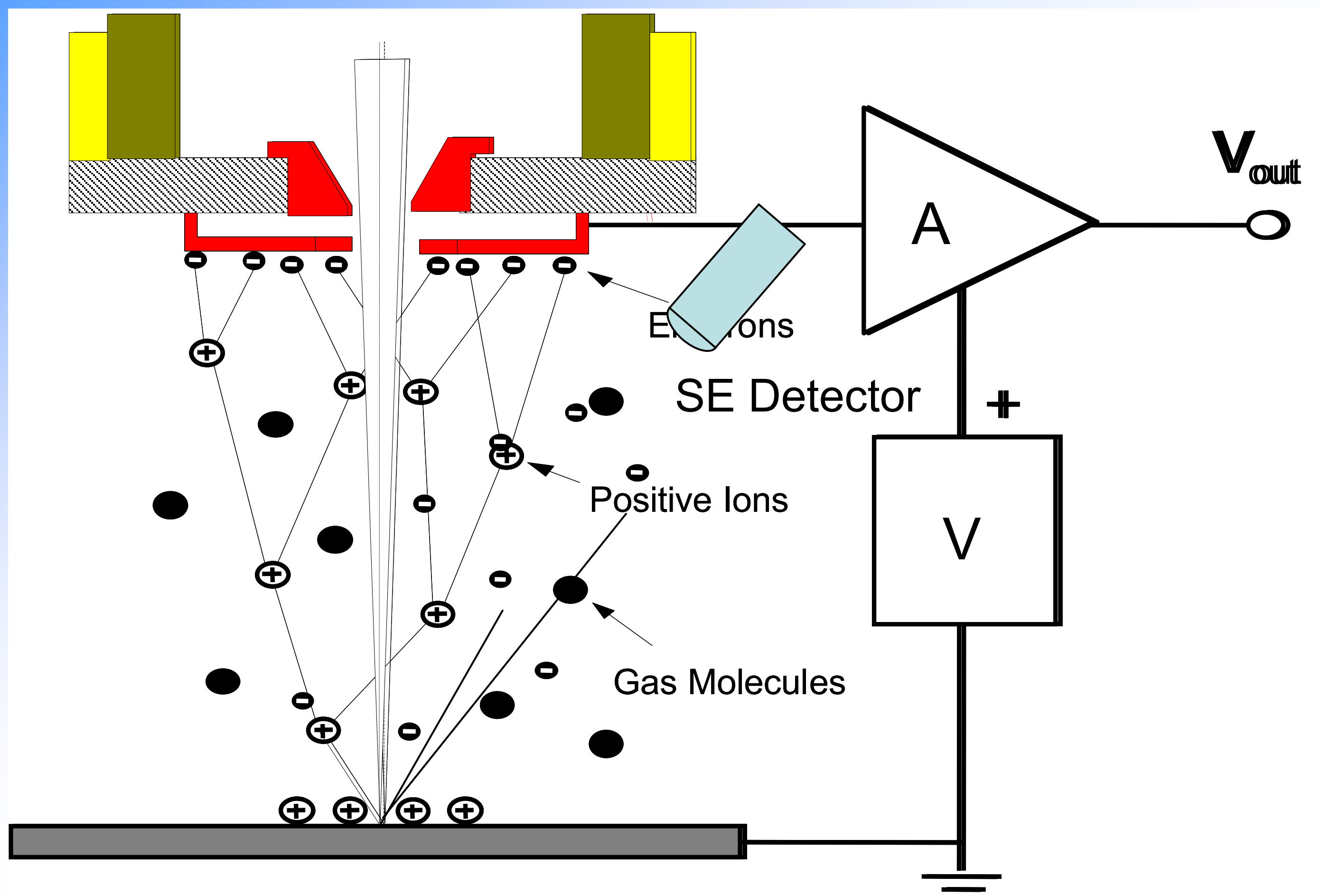


Wood Alloy - Mapping

Brief introduction of E

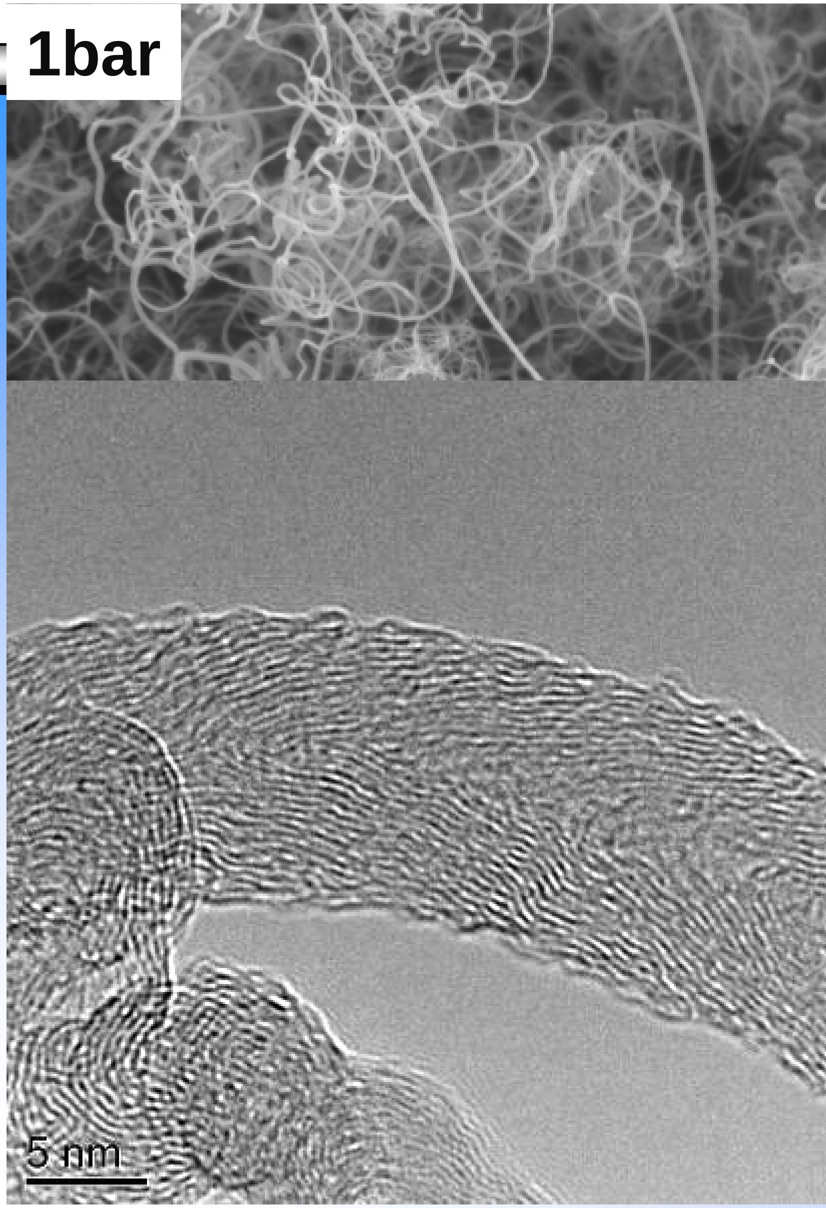


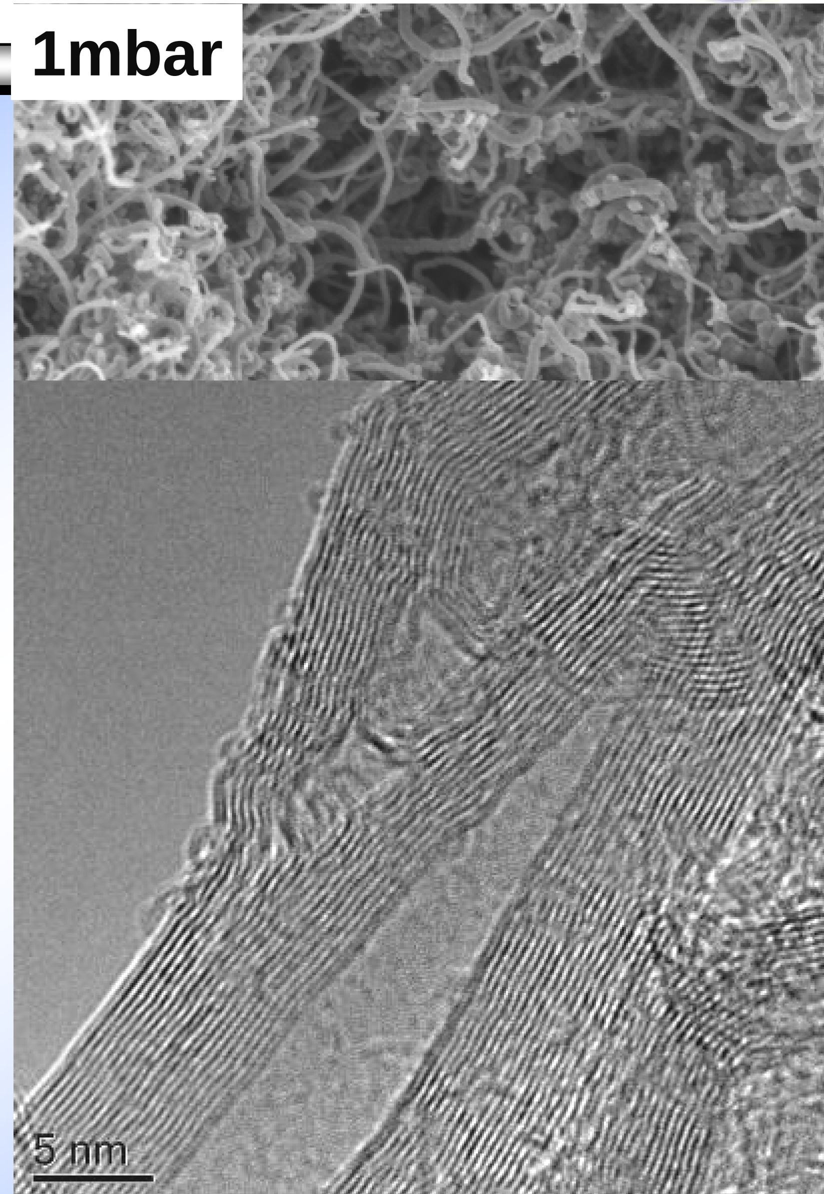
Brief introduction of image detector

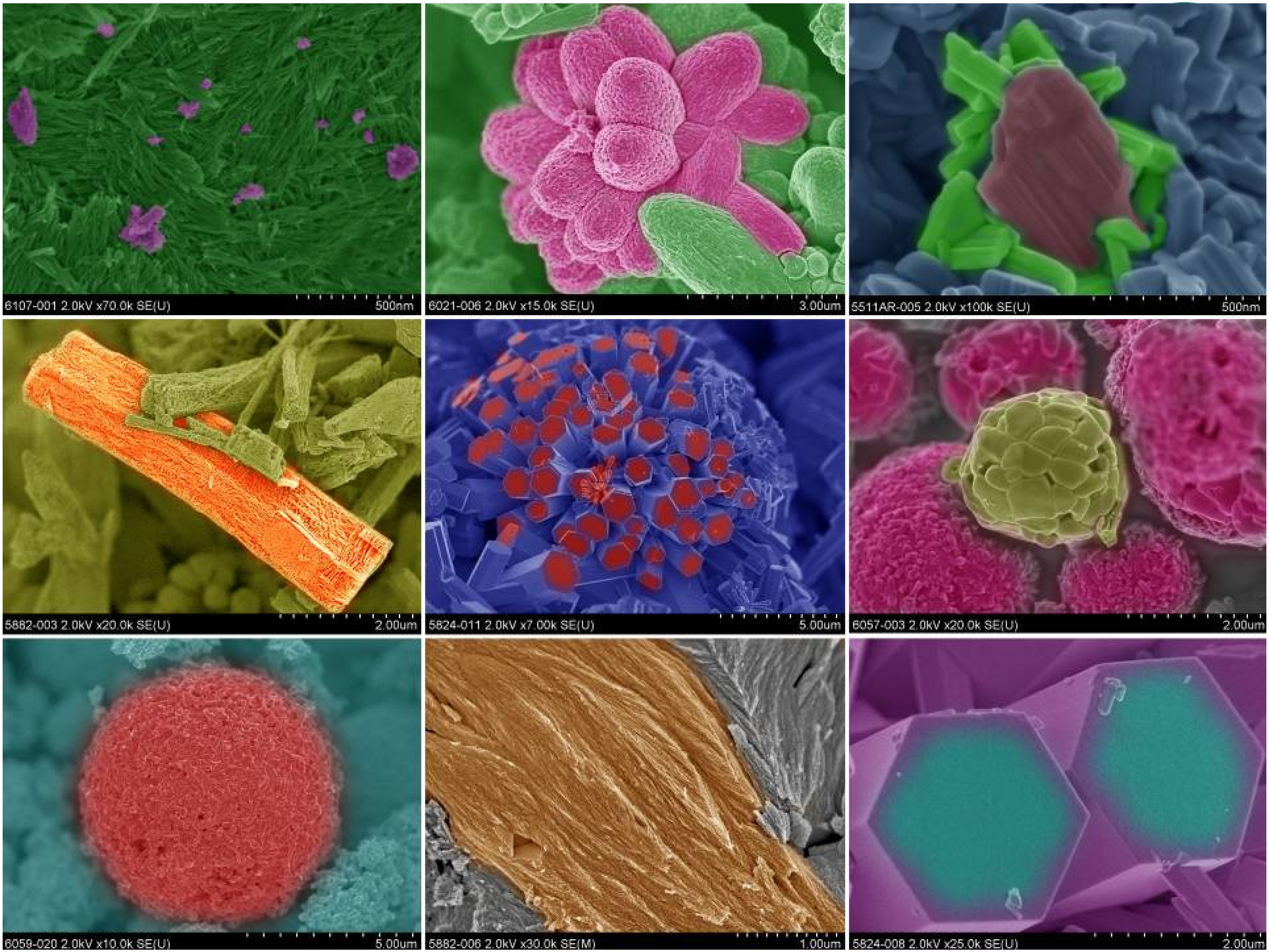


Schematic diagram of SE Detector
Schematic diagram of Gas Ionization Detector

SEM and TEM results






6107-001 2.0kV x70.0k SE(U)
500nm
6021-006 2.0kV x15.0k SE(U)
3.00um
5511AR-005 2.0kV x100k SE(U)
500nm
5882-003 2.0kV x20.0k SE(U)
2.00um
5824-011 2.0kV x7.00k SE(U)
5.00um
6057-003 2.0kV x20.0k SE(U)
2.00um
6059-020 2.0kV x10.0k SE(U)
5.00um
5882-006 2.0kV x30.0k SE(M)
1.00um
5824-008 2.0kV x25.0k SE(U)
2.00um

MAX-PLANCK-GESELLSCHAFT

Scheme of Scanning Electron Microscope