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Scanning Electron Microscopy

Nicola Pinna

Department of Chemistry, CICECO, University of Aveiro, 3810-193 Aveiro, Portugal.

School of Chemical and Biological Engineering, College of Engineering, Seoul National University (SNU), Seoul 151-744, Korea

E-mail: pinna@ua.pt - pinna@snu.ac.kr

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The scanning electron microscope (SEM) provides images that closely approximate the physiology of the eye and brain expect

The flatness of the topological and morphological details that is observed in the light optical microscope and the transmission electron microscope is replaced by an image that appears very similar to what we would expect to see with our naked eyes

The main difference between optical and SEM images is the lack of color and depth information (such as in a photograph).

The latter can be corrected by acquiring two images at different angle of tilt

The visual impact of SEM images and the ability to reveal details that are displaced along the optical axis, in addition to those resolved in the two dimensional field of view of the image plane, has led to the application of SEM to all branches of science

Comparison of an optical photograph and SEM image of a wasp

http://commons.wikimedia.org/wiki/Vespula_germanica

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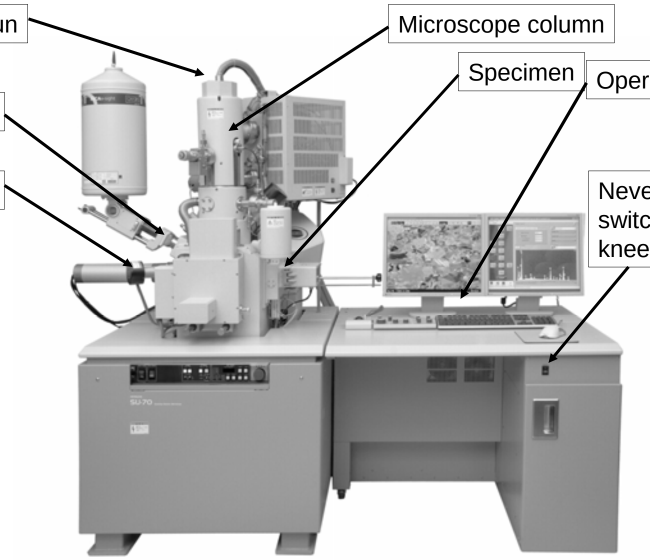
Components of the SEM



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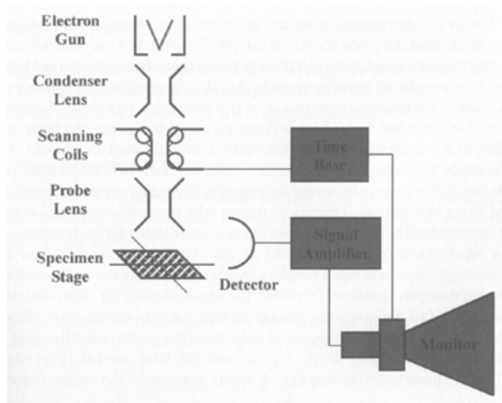
In the SEM a fine probe of electrons is focused onto the sample surface and then scanned across the surface in a television raster

The signal generated by the interaction of the probe with the specimen is collected in order to generate the image

As we already introduced the kind of interactions between an electron beam and a specimen can take place, you should be able to discuss them…

Illumination system of a SEM



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Beam focusing conditions

The probe lens is used to focus the electron beam onto the specimen surface in the SEM

The SEM condenser system reduces the size of the source

The scanning coils are used to scan the beam across the surface

The best resolution obtainable cannot be better than the focused probe size on the sample surface!

In practice there are three limitations on the minimum diameter that can be achieved for the probe beam in the plane of the specimen

1 – Spherical and chromatic aberrations of the probe lens

2 – The maximum beam current that can be focused into a probe of a given diameter

3 – The need to allow sufficient working space beneath the probe lens pole-pieces in order to accommodate large and rough specimens

Modern microscopes, equipped with a field emission gun as electron source, which drastically reduces the size of the primary electron source and increases the current density, permits to achieve resolutions of the order of 1 nm even at low voltages

The power of the SEM derives from the wide range of signals that may result from the interaction of the electron probe with the sample surface

These include: …

Characteristic X-rays, Cathodoluminescence, Backscattered electrons, secondary electrons, …

Inelastic scattering and energy losses

The electrons from the incident beam propagating through the crystal follow an irregular scattering path, losing energy as their integrated path length in the crystal increases

The inelastic scattering path for electron can be simulated quantitatively by Monte Carlo methods based on random scattering events

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Monte Carlo simulations of electron beam–sample interactions in a scanning electron microscope (SEM).

The program used is CASINO: "monte CArlo SImulation of electroN trajectory in sOlids"

This program is a Monte Carlo simulation of electron trajectory in solid specially designed for low beam interaction in a bulk and thin foil. This complex single scattering Monte Carlo program is specifically designed for low energy beam interaction and can be used to generate many of the recorded signals (X-rays and backscattered electrons) in a scanning electron microscope. This program can also be efficiently used for all of the accelerated voltage found on a field emission scanning electron microscope(0.1 to 30 KeV).

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CASINO V2.42—A Fast and Easy-to-use Modeling Tool for Scanning Electron Microscopy and Microanalysis Users

DOMINIQUE DROUIN[1], ALEXANDRE RÉAL COUTURE[1], DANY JOLY[1], XAVIER TASTET[1], VINCENT AIMEZ[1], RAYNALD GAUVIN[2]

[1]Electrical Engineering Department, Universite de Sherbrooke, Sherbrooke, Québec, J1K 2R1, Canada
[2]Department Mining, Metals and Materials Engineering, McGill University, Montreal, H3A 2B2 , Canada

http://www.gel.usherbrooke.ca/casino/index.html

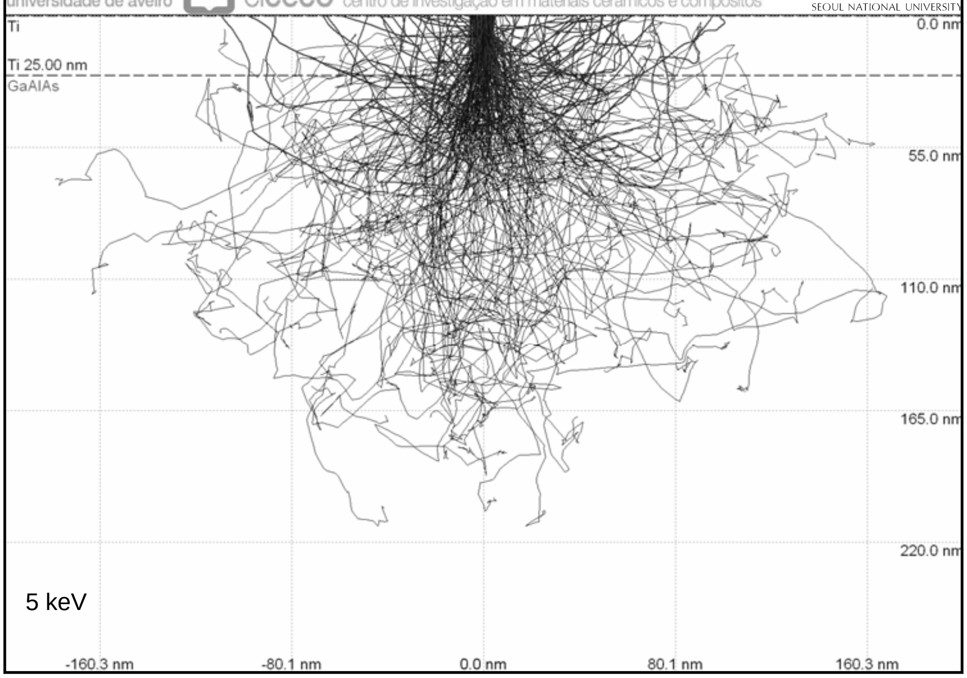
Simulate the electron of electron trajectories in a solid constituted of 25 nm thick Ti film on a GaAlAs substrate

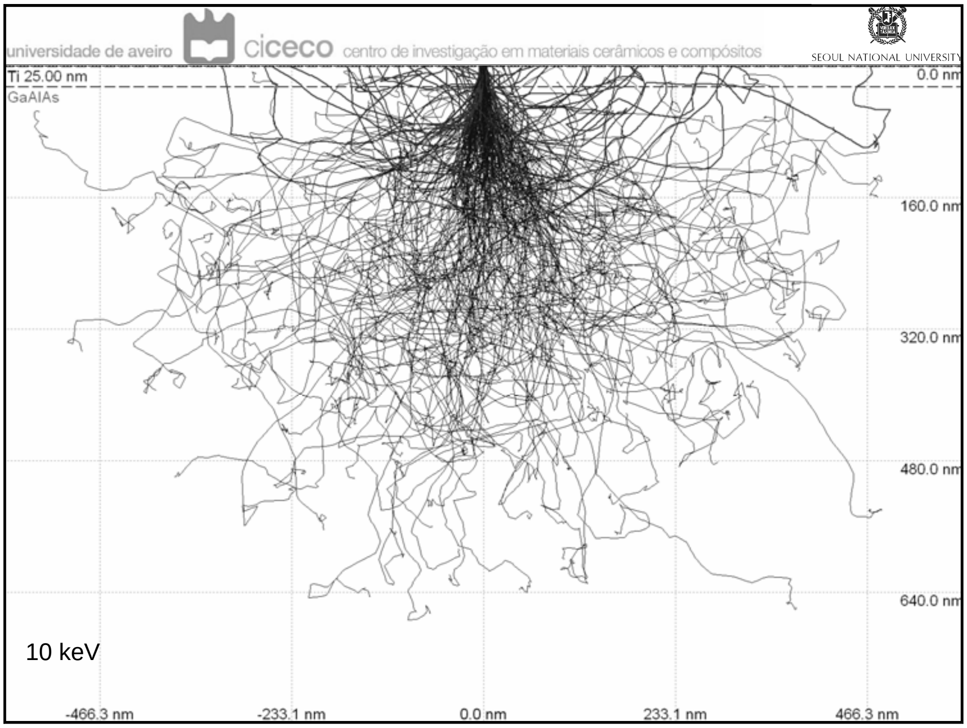
- 200 electron trajectories are displayed
- Incident energy varies from 5 to 30 keV (step 5 keV)
- Red trajectories represent electrons that are backscattered
- Blue trajectories represent electrons that are absorbed

The generation of secondary electrons is not taken into account !

The evaluation of the electron trajectories is primordial for scanning electron microscope users in interpretation of imaging and microanalysis and also with more advanced procedures including electron-beam lithography.

- Volume probed by EDX measurements
- Electron-beam lithography
- Etc.

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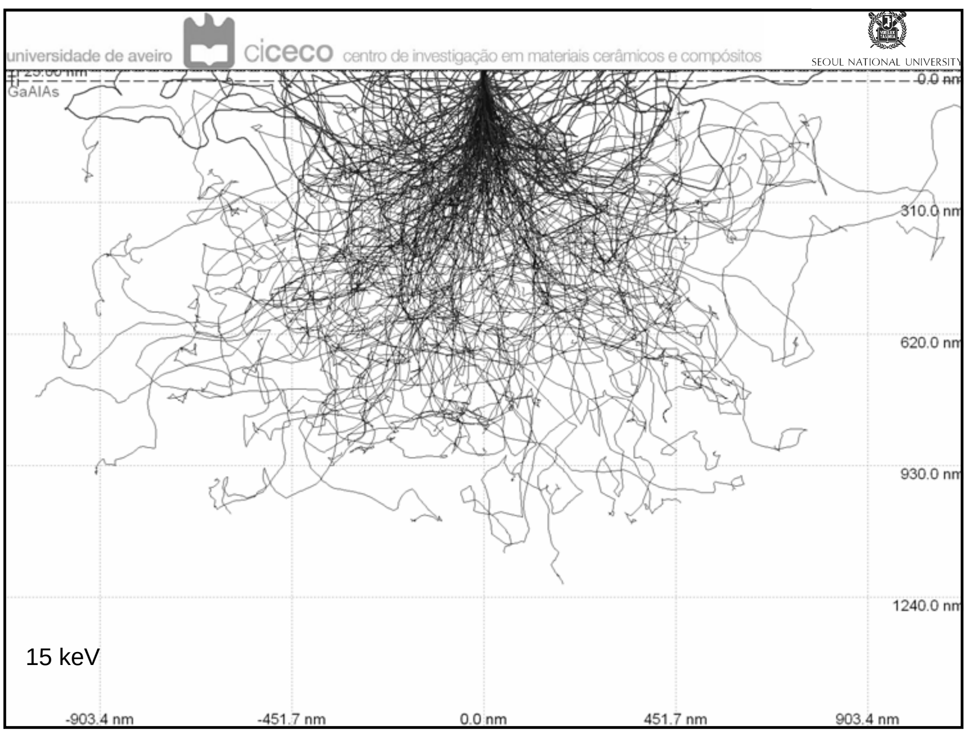
Ti 25.00 nm
GaAlAs
0.0 nm
160.0 nm
320.0 nm
480.0 nm
640.0 nm
10 keV
-466.3 nm
-233.1 nm
0.0 nm
233.1 nm
466.3 nm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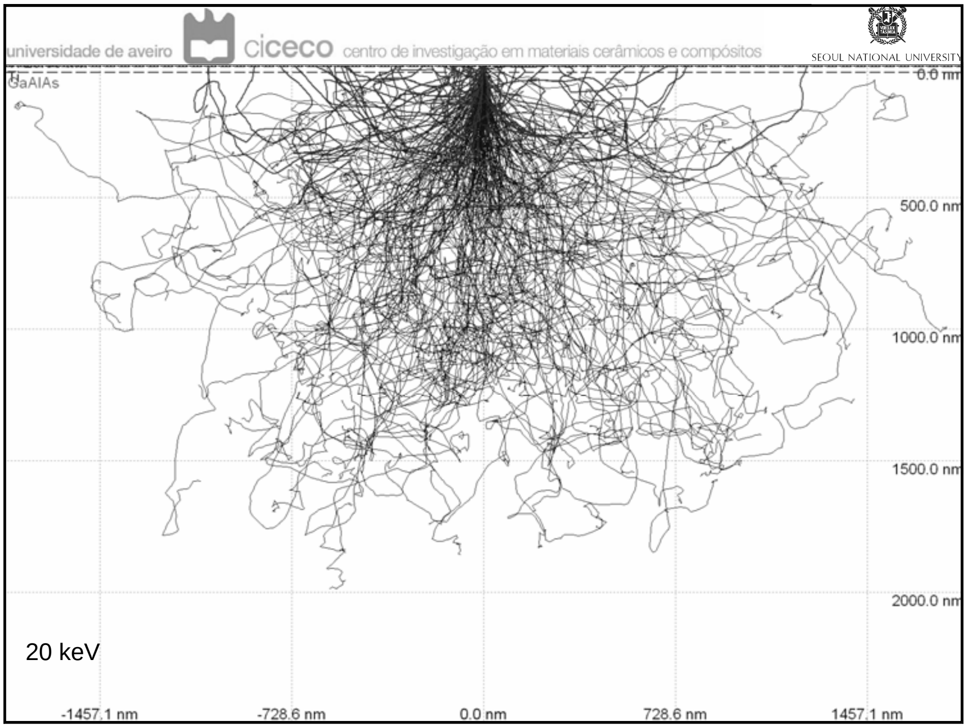
GaAlAs
0.0 nm
310.0 nm
620.0 nm
930.0 nm
1240.0 nm
15 keV
-903.4 nm
-451.7 nm
0.0 nm
451.7 nm
903.4 nm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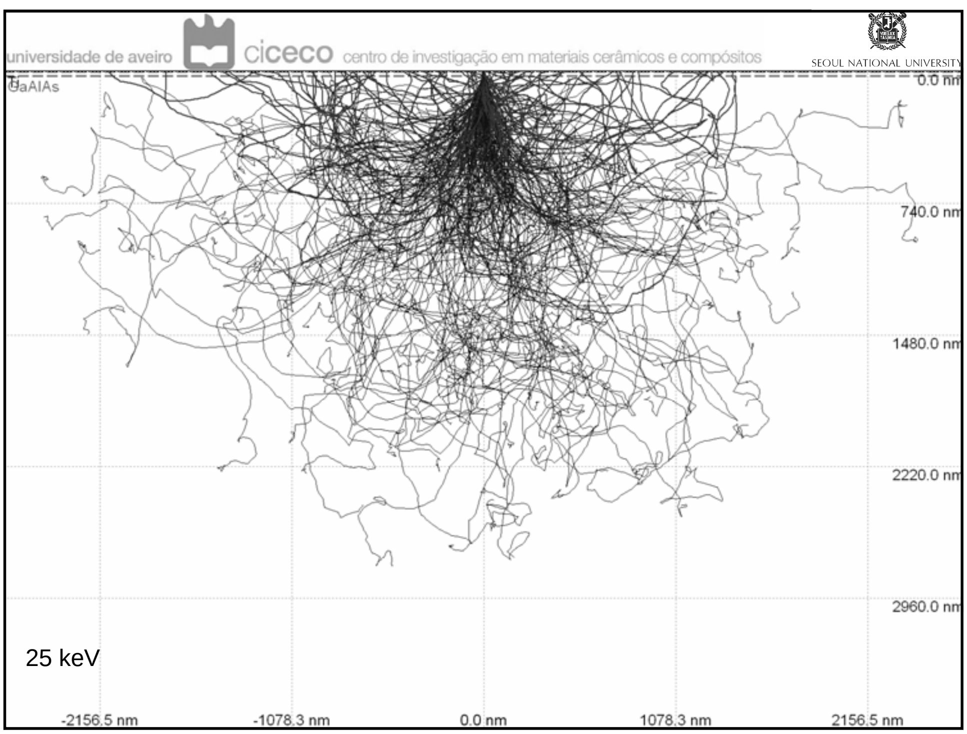
GaAlAs
0.0 nm
500.0 nm
1000.0 nm
1500.0 nm
2000.0 nm
20 keV
-1457.1 nm
-728.6 nm
0.0 nm
728.6 nm
1457.1 nm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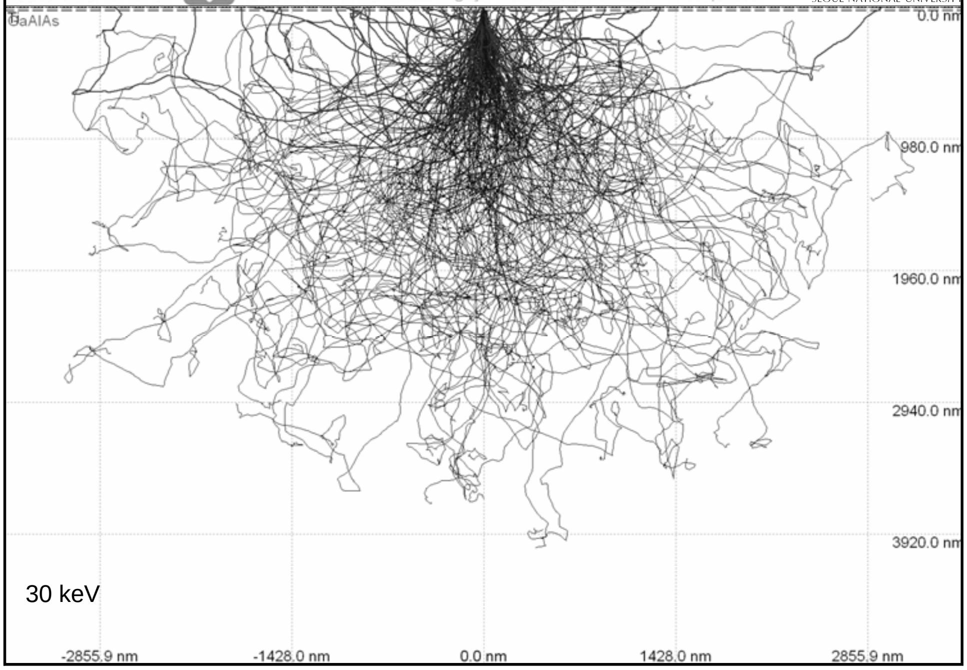
GaAlAs
0.0 nm
740.0 nm
1480.0 nm
2220.0 nm
2960.0 nm
25 keV
-2156.5 nm
-1078.3 nm
0.0 nm
1078.3 nm
2156.5 nm



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Schematic illustration of the electron yield as a function of the emitted electron energy

Backscattered electrons

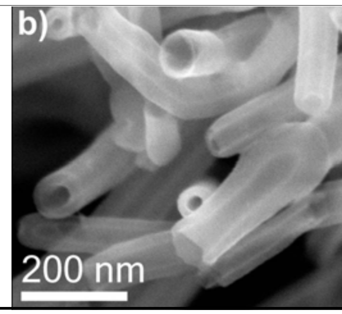
As we already know a fraction of the incident high energy electrons will be scattered at angles greater than π and these electrons have a finite probability of escape from the surface

The fraction of the incident beam backscattered depends rather sensitively on the atomic number of the specimen (cf. Rutherford cross section)

Backscattered electrons can be use for imaging with the ability to resolve local variations in mass density and result in atomic number contrast!

The resolution achievable in backscattering mode is slightly lower than what is achievable using secondary electrons (few nm in a modern FE-SEM)

Ex: Carbon nanotubes covered by 4-5 nm of vanadium oxide
The bright contrast on the inner and outer surface of the tubes is due to the vanadium oxide coating



Secondary electrons

We already know that secondary electrons are generated by ionization events caused by the incident radiation.

The secondary electrons can be conveniently “divided” in 3 distinct groups (cf. interaction radiation matter).
In SEM we are mostly interested in the slow secondary electrons

i.e. Electrons from the conduction or valence band.
You remember that not much energy is needed to eject them. Their kinetic energy is typically below about 50 eV

Only the electrons from the surface of the sample and having low kinetic energy can escape from the sample (i.e. mean free path of the electrons at these energy is very small)

Moreover, these low energy secondary electrons are efficiently collected (close to 100%) by applying a low bias voltage

For these reasons the secondary electron imaging mode in SEM is the one that permits to achieve the best resolution and is the most commonly used

Factors affecting secondary electrons emission

1. The work function, that is the energy barrier that has to be overcome by an electron at the Fermi level in the solid to permit it to escape from the sample into the vacuum

2. The incident electron beam energy and beam current.
As the beam energy increases more electrons are expected to be created in the specimen. However, at higher beam energy the depth at which they are created increases resulting in a lower portion of the secondary electrons that can escape from the surface.
On the other hand, the secondary electron generation is directly proportional to the current in the incident beam

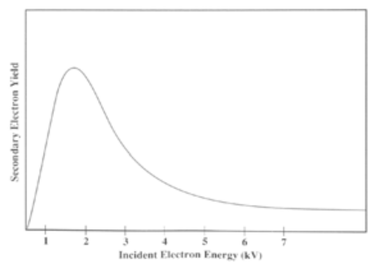


Figure 5.15 Schematic illustration of the expected secondary electron yield as a function of the incident electron beam energy.

3. There is also a dependence on Z even though less pronounced than in the case of backscattering mode. When Z increases the secondary electron emission yield increases.
   - Increase of the secondary electron generation and the fact that they are generated at lower depths (tend to increase the yield)
   - Decrease of the mean free path of the electrons (tend to decrease the yield)

4. Surface topography.
In general, changes in local curvature change the probability of secondary electrons generated near the surface that can escape.
A region protruding form the surface (a region having a positive radius of curvature) increases the chances of secondary electrons escaping
While any recessed region, having a negative radius of curvature, will reduce the secondary electron current by local trapping of the secondary electrons

The secondary electrons are commonly collected using a bias voltage applied to the collector, so even though some region of the samples are out of line of the sight.

From this we may conclude that the secondary electron image should provide topographic images of rough surfaces having both high resolution and high contrast.

The secondary electrons are commonly collected using a bias voltage applied to the collector, so even though some region of the samples are out of line of the sight

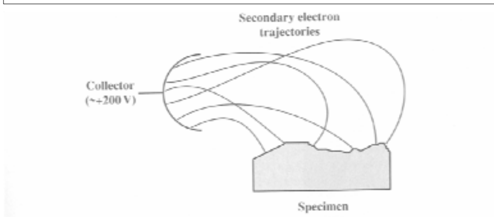


Figure 5.17 By applying a small bias voltage, the secondary electrons can be collected with high efficiency from regions of the surface that are not in the direct line of sight of the collector.

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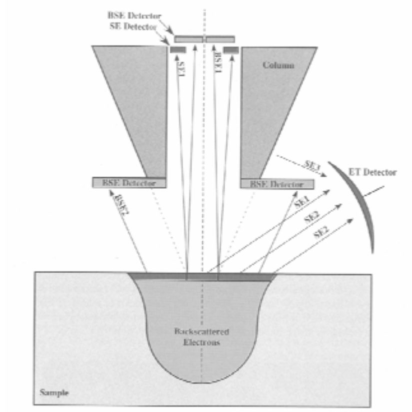

Figure 5.19 The location of secondary electron (SE) and backscattered electron (BSF) detectors. Secondary electrons of the first type (SE1) generated by the primary incident beam, are preferentially acquired by a detector placed within the electro-optical column to form a very high resolution image when compared with secondary electrons of the second type (SE2) that are acquired by an Everhart–Thornley (ET) detector. Similarly, BSE1 electrons, defined as primary beam electrons that have undergone very few inelastic scattering events, can be acquired by a BSE detector placed within the column, to improve the resolution for BSE micrographs when compared with those acquired with a conventional annular BSE detector that collects all the multiply scattered backscattered electrons.

The resolution was "recently" improved with the introduction of field emission guns, the reduction of the working distance of the probe lens to just a few millimeters and by placing a secondary electron detector inside the magnetic field of the pole pieces.

In this last geometry the emitted electrons in the forward directions are trapped by the magnetic field of the pole pieces and collected, while those generated by backscattered electrons at wider angles cannot reach the in-lens detector placed within the electro-optical column

The secondary electron signal detected in this way is much weaker but it improves the secondary electron image resolution from 20-50 nm to only 1-2 nm!

An additional advantage of the instruments equipped with a filed emission gun is that low energy incident electrons can be used without loosing resolution. At these low beam energies there is generally no problem of surface conductivity and no need to coat a nonconductiong sample. The beam penetration into the sample is limited and any electrostatic charging is compensated by a large yield of secondary electrons.

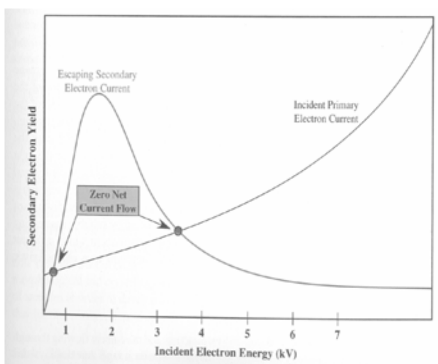

Figure 5.20 Schematic illustration demonstrating charge compensation by selecting an optimal accelerating voltage. The total incident current decreases as the accelerating voltage is decreased (black curve). This curve intersects the characteristic secondary electron yield at two points, where the total net current of electrons to the sample is zero and electrostatic charging of the surface is prevented.

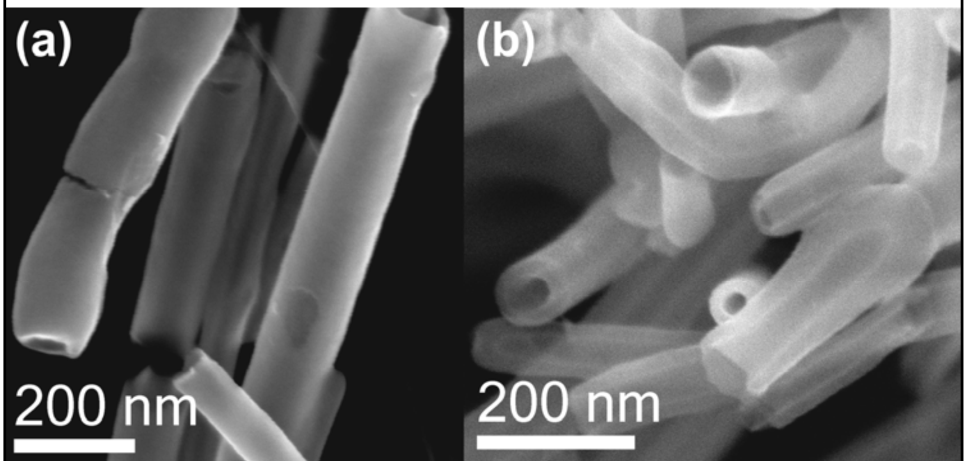

Fig. 2 SEM image recorded from V_2O_4-CNT (V_2O_4 film thickness: 4.5 nm) using secondary electrons (a) and backscattered electrons (b),

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Characteristic X-Ray images

The emission of X-ray is a competitive relaxation phenomena to the Auger effect.

Upon ionization of an atom the relaxation process can produce an Auger electron or X-ray emission. The two relaxation processes are in competition

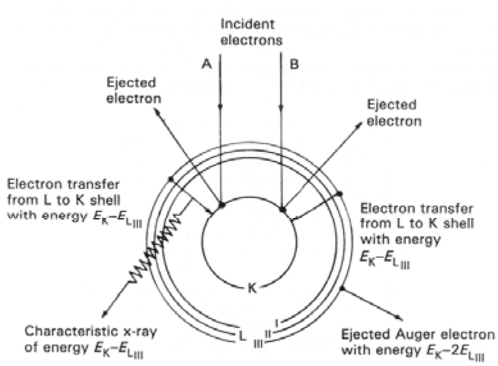

Fig. 2.3. Schematic diagram showing emission of characteristic x-ray by electron A and emission of Auger electron by electron B.

The characteristic X-rays are used in energy dispersive spectroscopy

Emission of X-ray: Transition notations

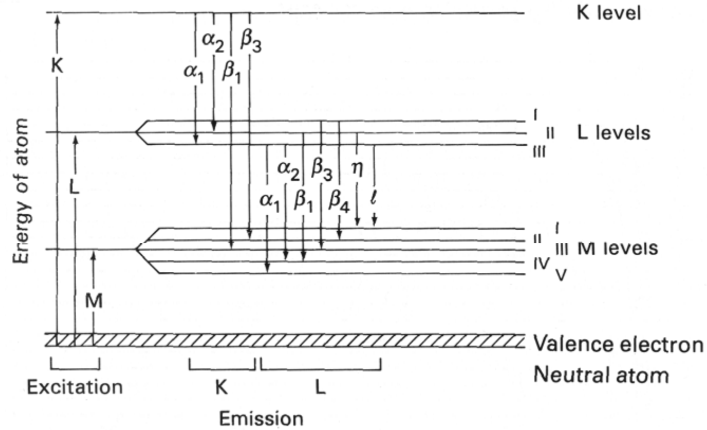

Fig. 2.2. Schematic diagram showing common x-ray emission lines with their designation for an element with atomic number (Z), where $29 < Z < 37$.

These transitions obey to the electric dipolar selection rules ($\Delta l \neq 0$, $\Delta j = 0, \pm 1$)

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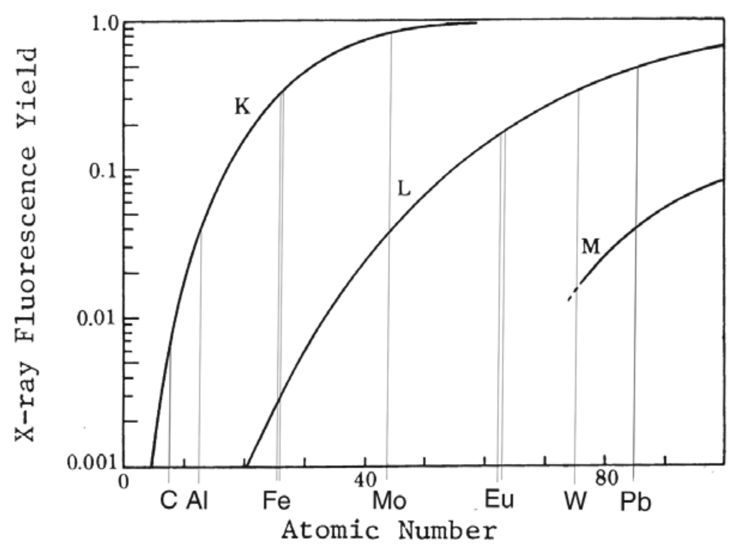
X-ray fluorescence yield for K, L and M shells



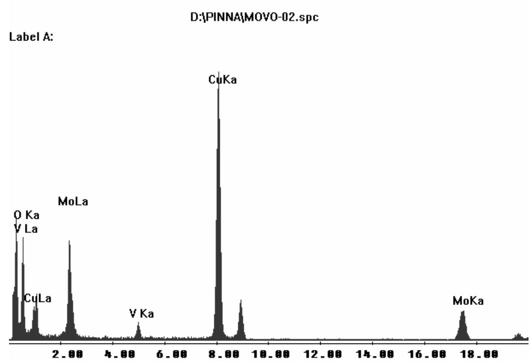
X-ray fluorescence yield for *K*-, *L*-, and *M*-shells, as a function of atomic number.

The efficiency of the X-ray generation is low and the X-rays are emitted in all the directions

A high proportion of the X-ray signal is either absorbed by the specimen or fails to reach the detection system

The X-ray signal can be displayed in three distinct formats:

1. An X-ray spectrum. This is primarily used to identify the chemical elements present form their characteristic X-Ray "fingerprints"



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2. An X-ray line-scan. In this mode the beam is scanned across a selected region of the sample in discrete steps and the signal prerecorded at each step

The best achievable spatial resolution is limited by the volume element of the sample from which X-Rays are generated (~ μm)

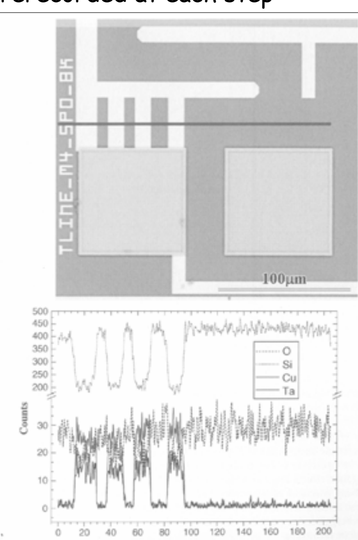
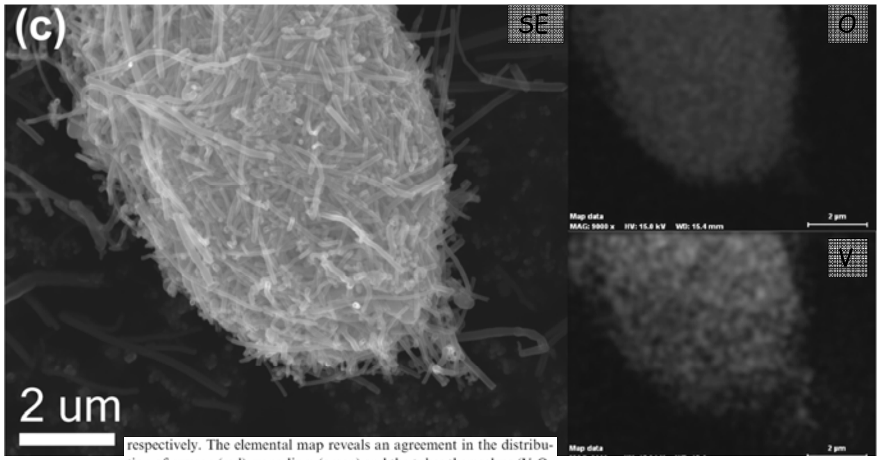


Figure 5.8 EDS line-scan across a printed circuit board. The red line in the secondary electron SEM micrograph indicates where the line-scan was acquired.

3. An X-ray concentration map. In this case, the incident electron beam is rastered across a selected area of the sample, and photon counts are collected for one or more energy windows that are characteristic of the chemical components of interest (spatial resolution ~ 1 μm – Compare to SE image!)



respectively. The elemental map reveals an agreement in the distribution of oxygen (red), vanadium (green) and the tubes themselves (V_2O_4 thickness 5 nm) (c). (d and e) show regions with defective coating for

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