LAB TECHNIQUES

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Contents

Mineral Analysis

Scanning electron microscopy (SEM).

- How does SEM work?
- Electron sample interaction.
- SE and BSE images.
- Chemical mapping.

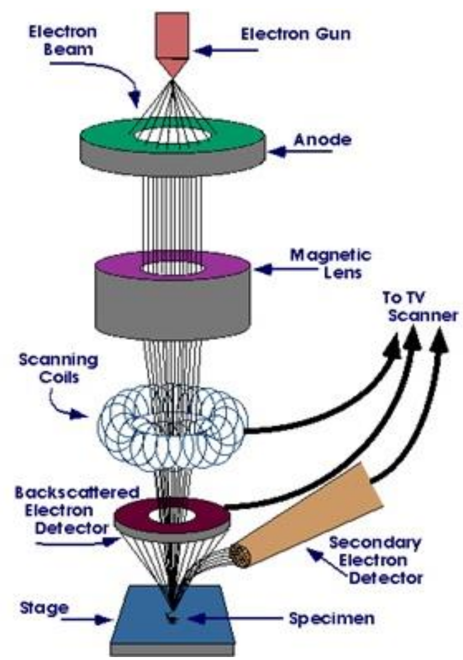
SEM

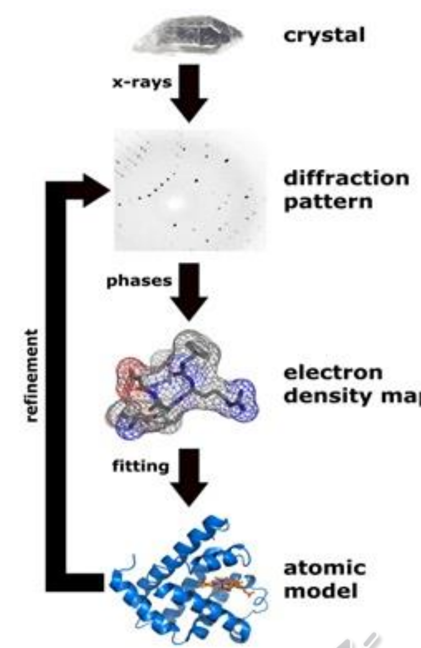
6. Scanning Electron Microscopy (SEM)

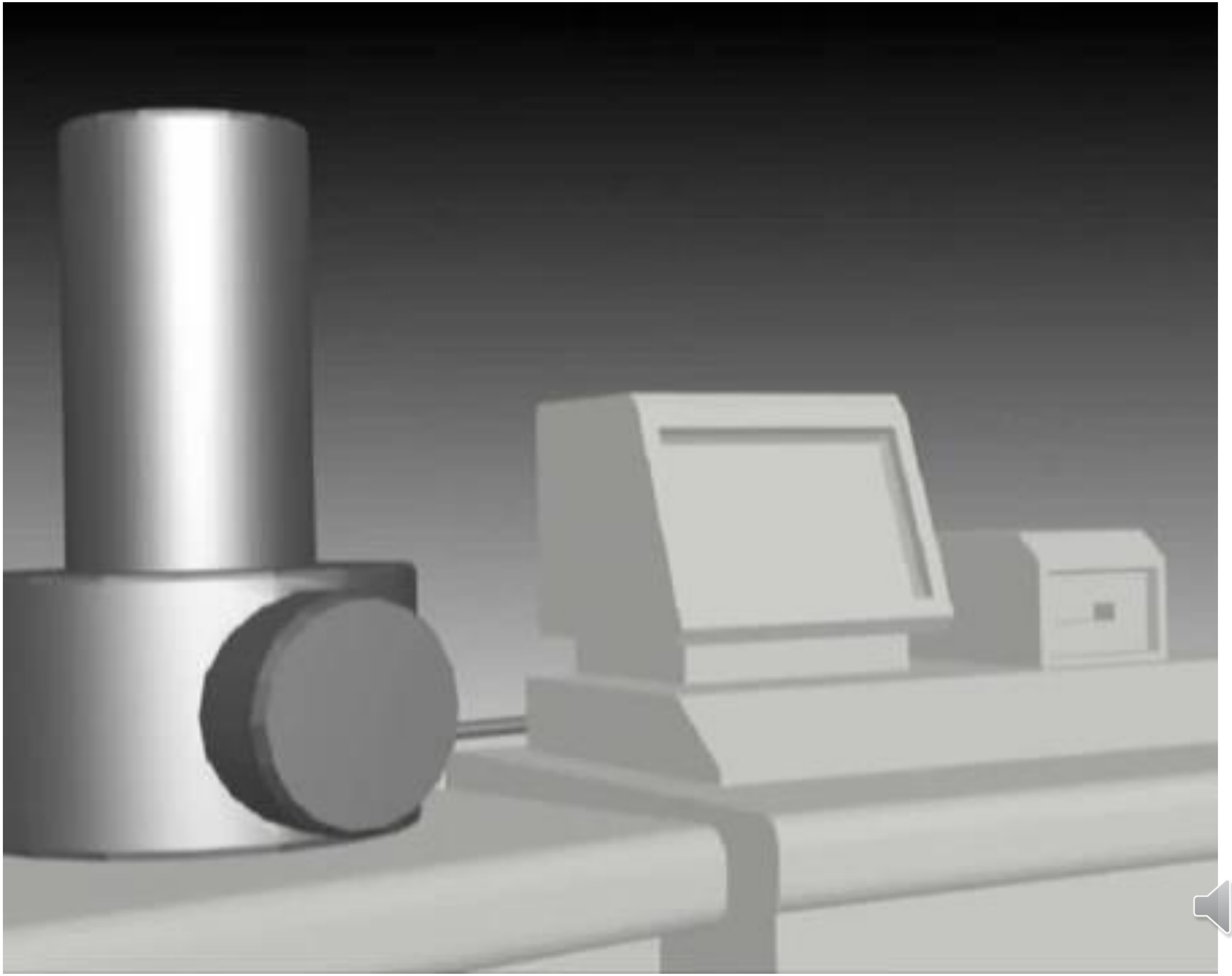
There are different types of Electron Microscopy. These can be split into two main categories, **transmission electron microscopes (TEM)** and **scanning electron microscopes (SEM).** The main differences between these are in the microscope **optics, the signal detection and the obtained information.**

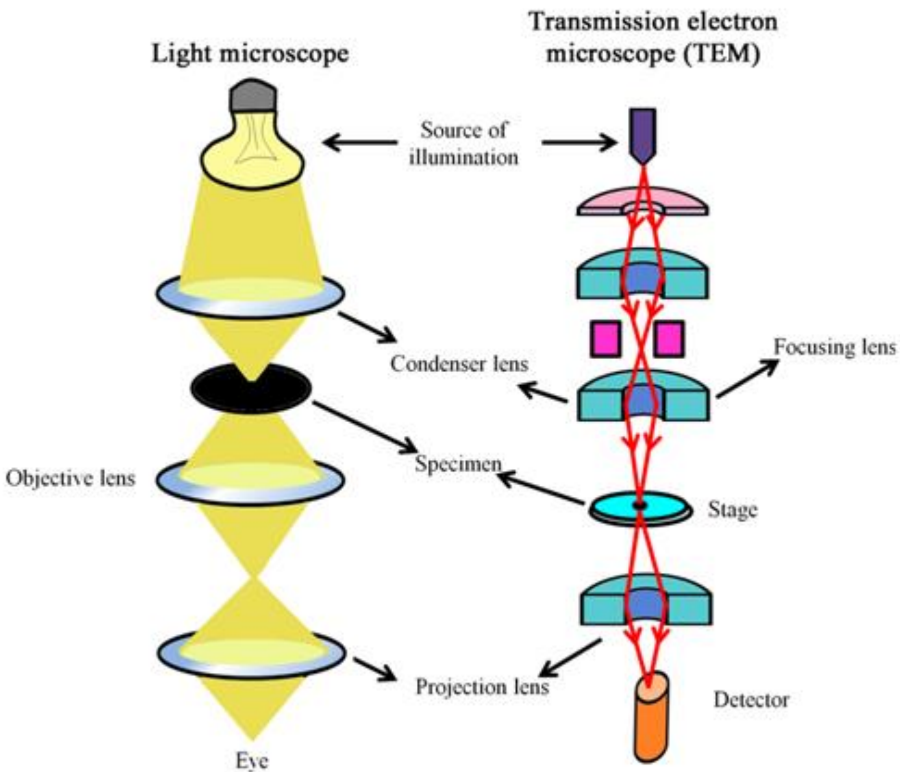
Both types of **EM** have an **electron gun/source**, which is **a filament that produces a cloud of electrons**, a Wehnelt cylinder (to form the beam) and an anode (to accelerate the beam).

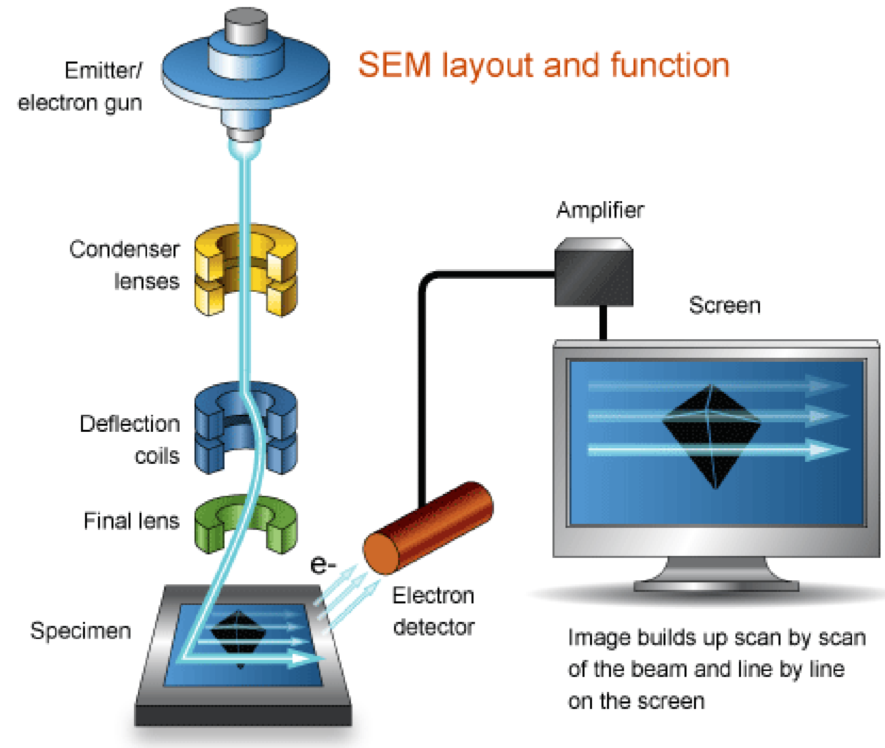
There are three main types of electron source: a tungsten filament, a lanthanum hexaboride (LaB_6) crystal and a field emission filament. The main signals that are relevant for the TEM are the transmitted and scattered electrons. For the SEM, the main signals are the secondary and backscattered electrons.







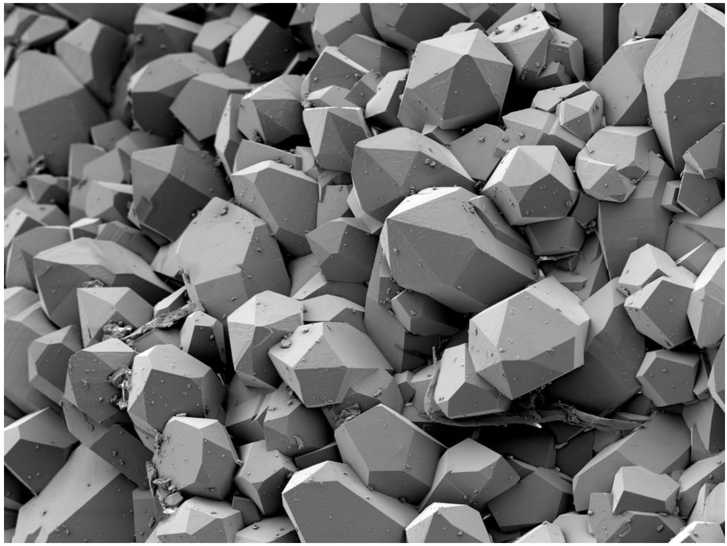


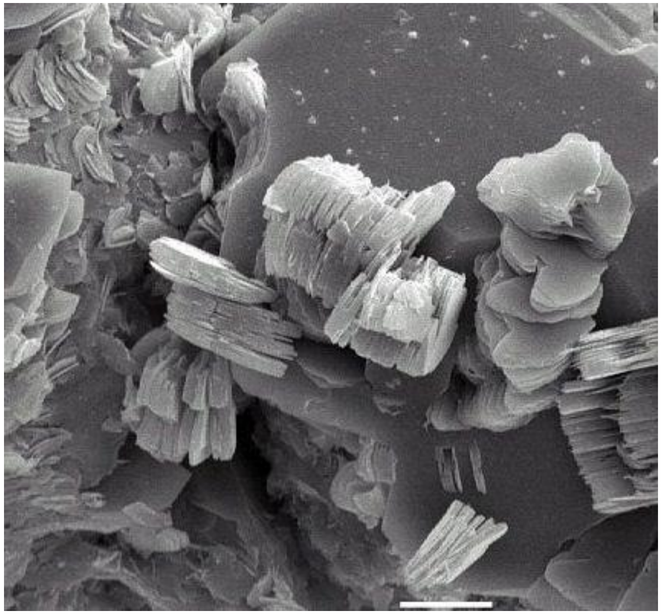


An **SEM** image is formed from signals that are emitted from the sample **as a result of the specimen-beam interaction.** SEM generate images using two types of electrons, **these are: secondary electrons (SE) and backscattered electrons (BSE).**

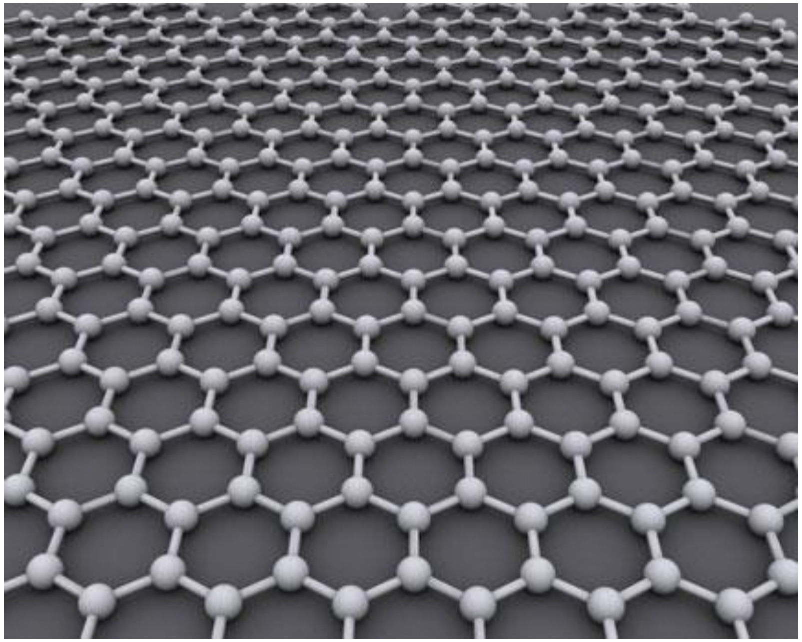
In addition, there are few applications that require **the detection of characteristic X-rays (energy dispersive X-ray spectroscopy EDX)** or **photons (cathodoluminescence). There are different types of detectors to collect these signals.**

SEM is used **to image the surface of bulk samples. Atomic contrast can be detected using backscattered electrons (BSE) as heavier atoms produce a brighter signal. SEM micrographs have an optical illusion that creates the impression of a 3D image when sample is tilted, however, no "z" dimension data** is available thus making the images 2D only when sample is not tilted plain.





TEM is appropriate for imaging **very thin samples** by **detecting the transmitted electron beam**. The image produced is analogous to an x-ray.



SEM V/S TEM

SEM	TEM
in SEM is based on scattered electrons	TEM is based on transmitted electrons
The scattered electrons in SEM produced the image of the sample after the microscope collects and counts the scattered electrons.	In TEM, electrons are directly pointed toward the sample.
SEM focuses on the sample's surface and its composition.	TEM seeks to see what is inside or beyond the surface.
SEM shows the sample bit by bit	TEM shows the sample as a whole.
SEM provides a three-dimensional image	TEM delivers a two-dimensional picture.
SEM only offers 2 million as a maximum level of magnification.	TEM has up to a 50 million magnification
SEM has 0.4 nanometers.	The resolution of TEM is 0.5 angstroms

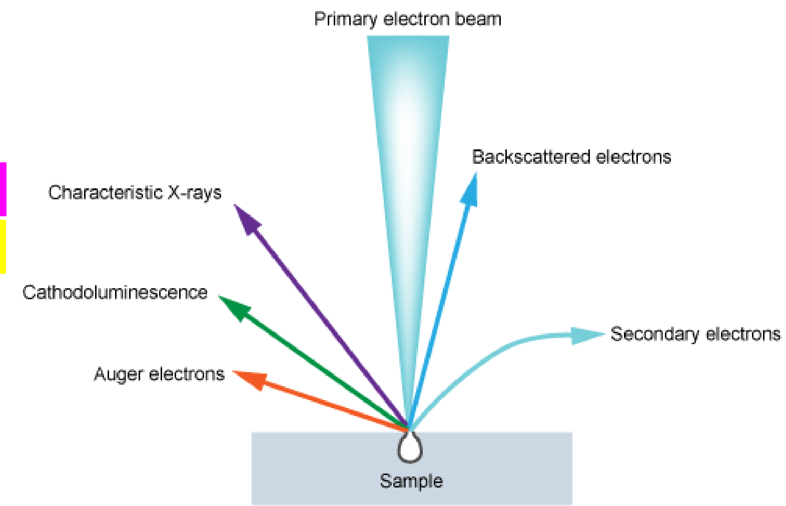
SEM vs. TEM

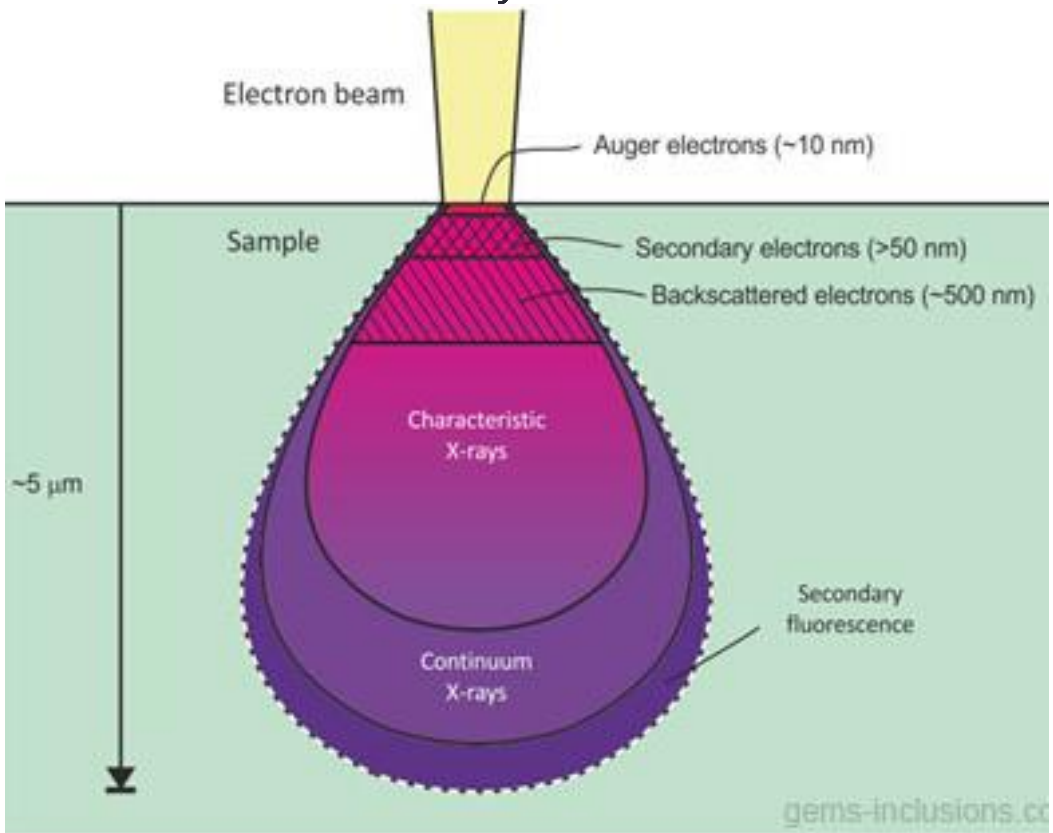
- SEM is based on scattered electrons while TEM is based on transmitted electrons.
- SEM focuses on the sample's surface and its composition whereas TEM provides the details about internal composition. Therefore TEM can show many characteristics of the sample, such as morphology, crystallization, stress or even magnetic domains. On the other hand, SEM shows only the morphology of samples.
- The sample in TEM has to be cut thinner whereas there is no such need with SEM sample.
- TEM has much higher resolution than SEM.
- SEM allows for large amount of sample to be analyzed at a time whereas with TEM only small amount of sample can be analyzed at a time.

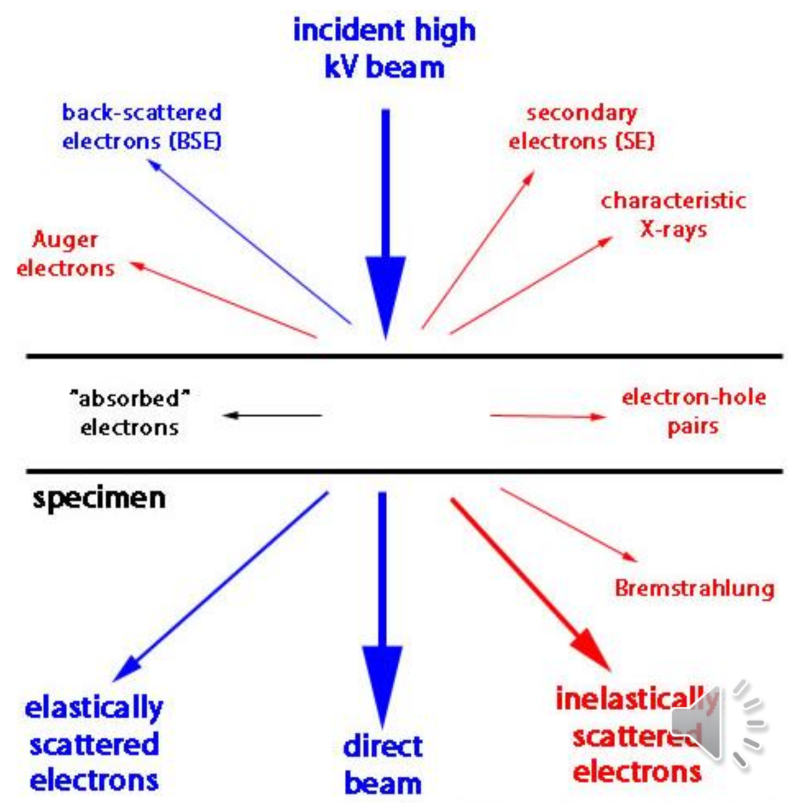
Properties	SEM	TEM
Principle	Based on scattered electrons.	Based on transmitted electrons.
Resolution	Up to 0.4 nm.	Up to 0.05 nm.
Maximum magnification	Up to a 2 million.	Up to a 50 million.
Objective	Focuses on the specimen's surface and its composition.	Focuses on the detail about internal composition.
Analytical Ability	Shows only the surface morphology of samples.	Show many characteristics of the sample, such as morphology, crystallization, tiny precipitates, stress or even magnetic domains.
Specimen section specification	Section cutting is not needed (no any constraint on specimen dimension).	The sample has to be cut into thinner sections (less than 100nm range).
Analysis time	Allows for large amount of sample to be analyzed at a time.	Only small amount of sample can be analyzed at a time.
2D/3D imaging	Provides a 2D/3D image.	Provides a 2D image.
Cost	The cost is relatively low.	The cost is much higher (two to three times then SEM).

How does SEM work?

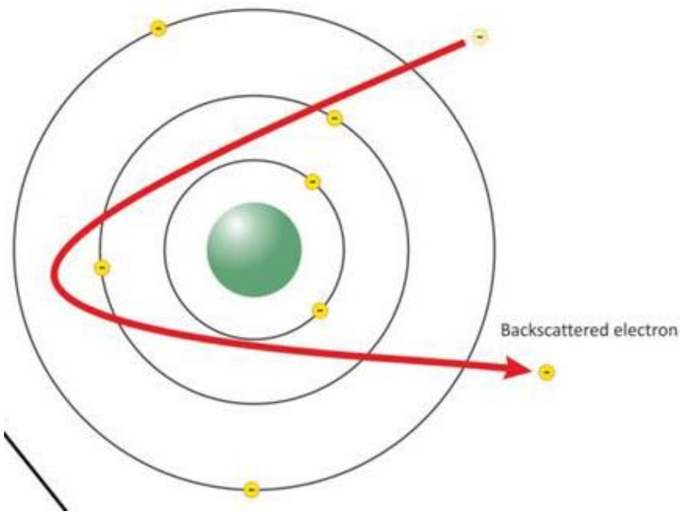
When the **electron beam** hits a sample it interacts with the atoms in that sample. **The interaction volume** is **a pear-shaped region on and below the surface of the sample** in which interactions take place. Depending on the interaction mechanism, electrons or photons (X-rays) may be emitted and collected by definite detectors.







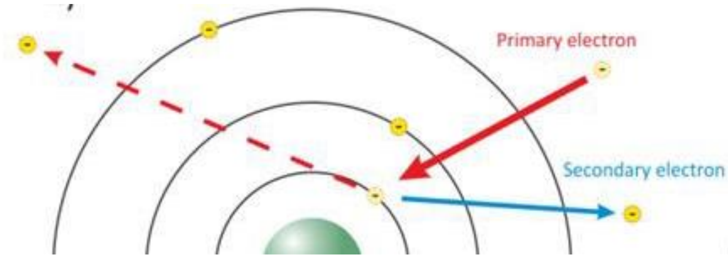
The electron-sample interactions are:

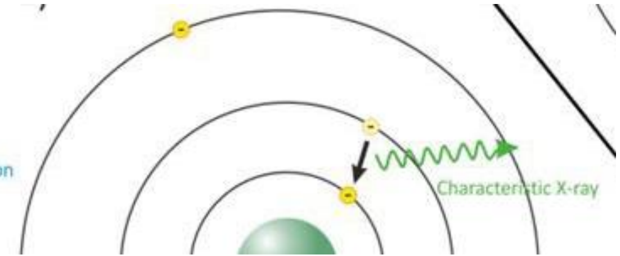


1. Some primary electrons are **bounced back** out of the sample, these are the **backscattered electrons (BSE).** The **BSEs** are **high-energy electrons reflected by elastic scattering (without energy loss)** further away from the surface, **conserving their energy but changing their direction. The detection of such electrons produce BSE images.**

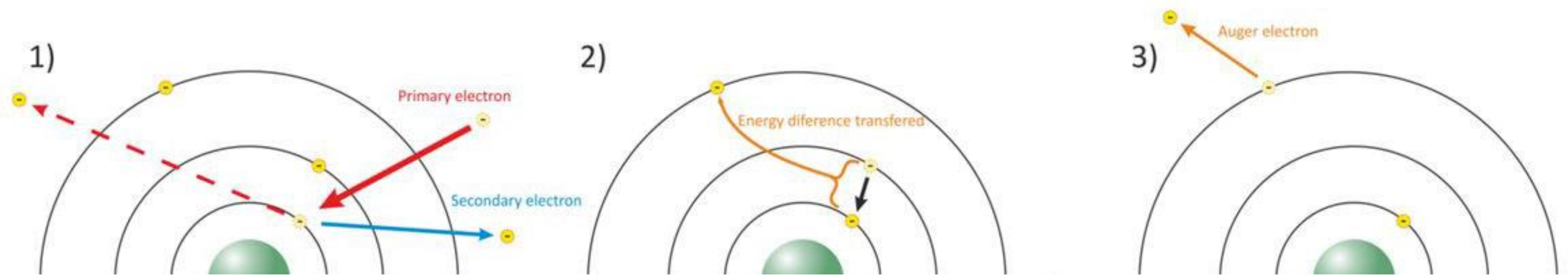
2. Some primary electrons **knock into atoms and scattered inelastically** displacing the **atoms electrons** that, in turn, come out of the sample, **these are secondary electrons SEs which are low-energy electrons**. **The detection of such electrons produce SE images.**

3. When a **SE** is ejected, a gap is born. This gap is filled by another electron from a higher energy level emitting a **characteristic X-ray** corresponds to the energy difference between the two levels. This **characteristic X-rays** are the basis of **EDX.**

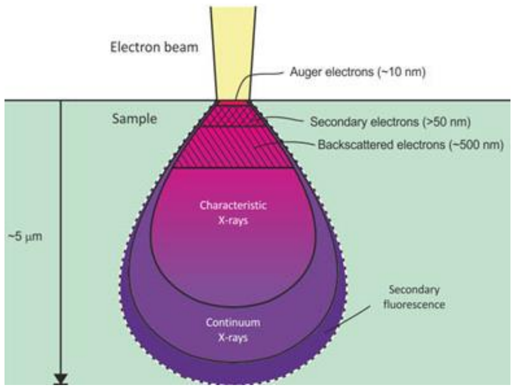


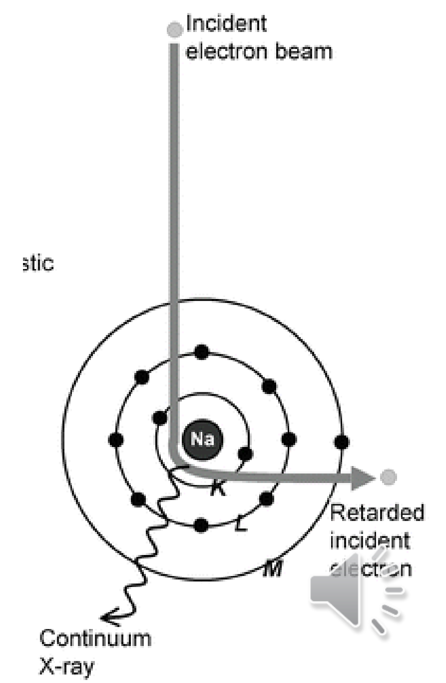


4. **Alternatively, the characteristic X-ray could be absorbed by a third electron from a further outer shell, prompting its ejection**. This ejected electron is called an **Auger electron**, and the method for its analysis is known as **Auger electron spectroscopy (AES).**



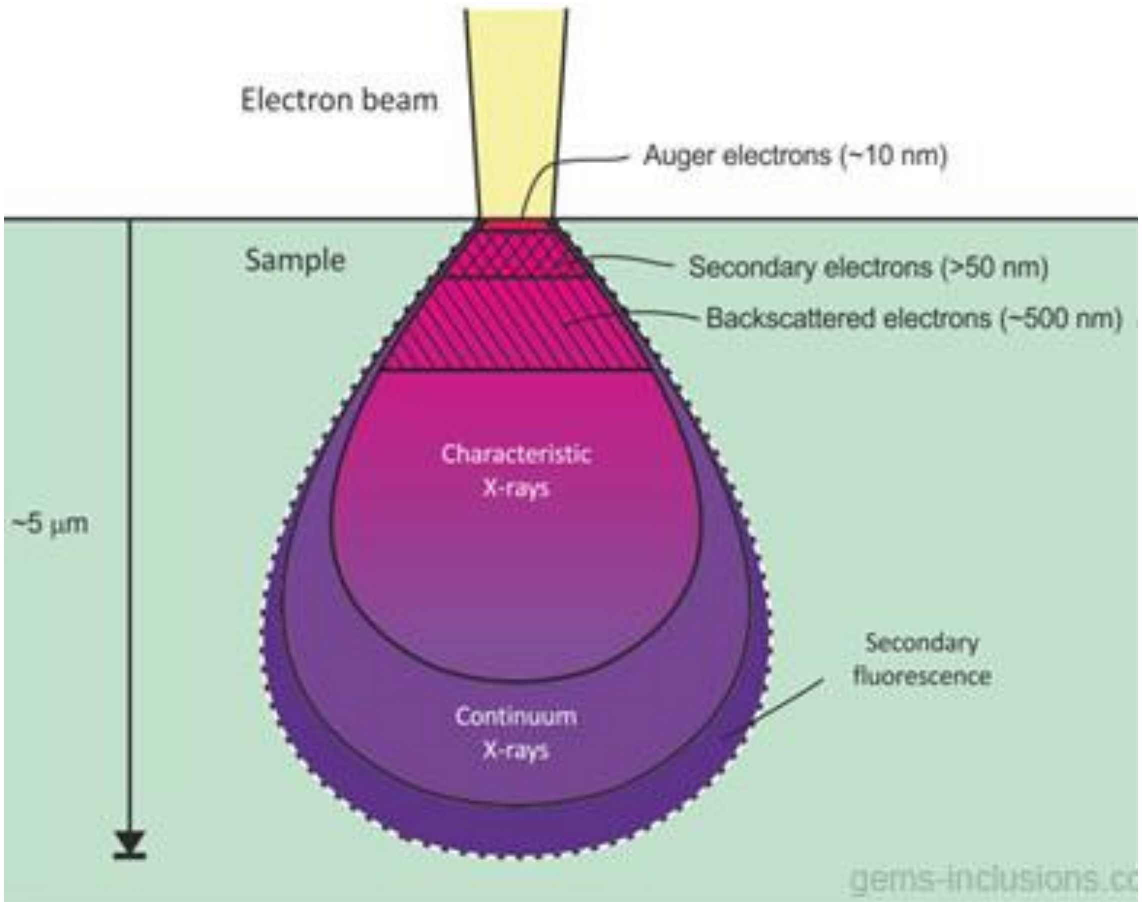
5. **A continuum X-rays** are produced when incident beam electrons are slowed down to varying degrees by the strong electromagnetic field of atomic nuclei in the sample. All degrees of **electron braking** are possible and, thus, **the resulting X-rays have a continuous range, i.e., not-characteristics**, of all energies and are not useful for the sample characterization.

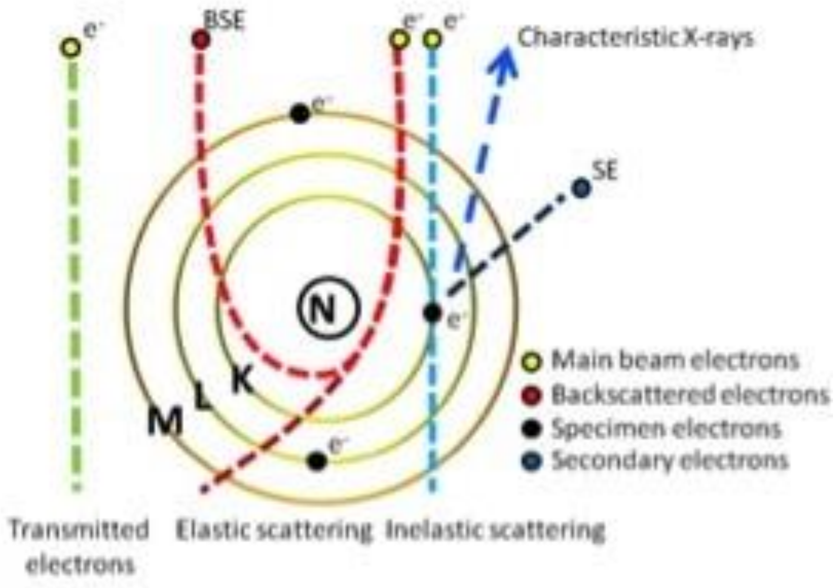


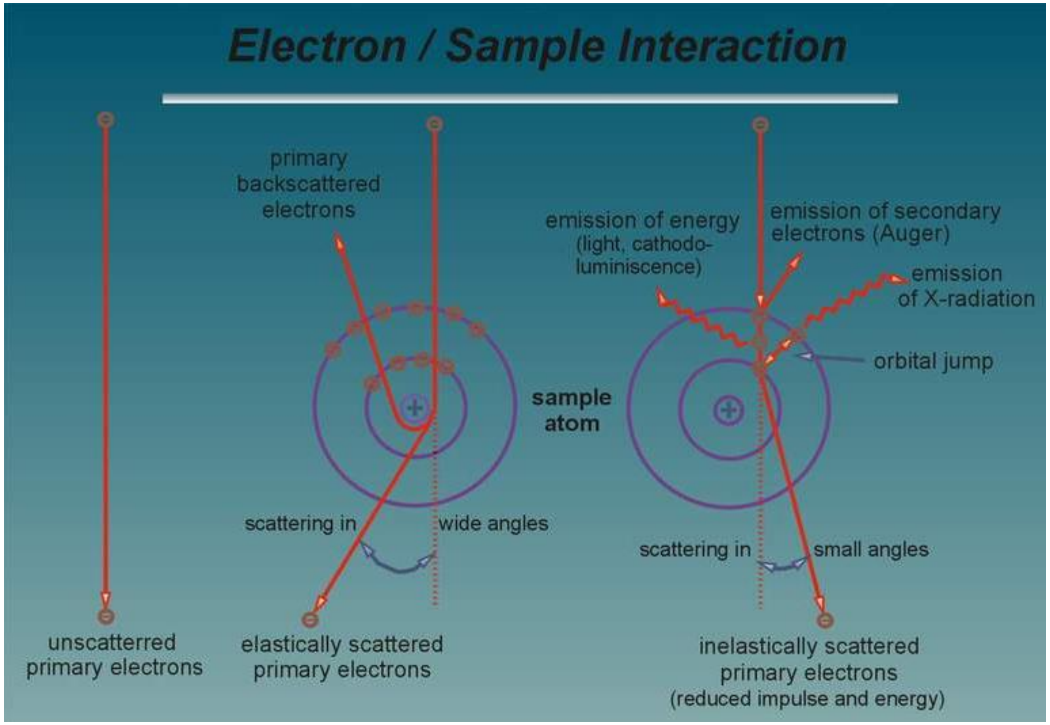


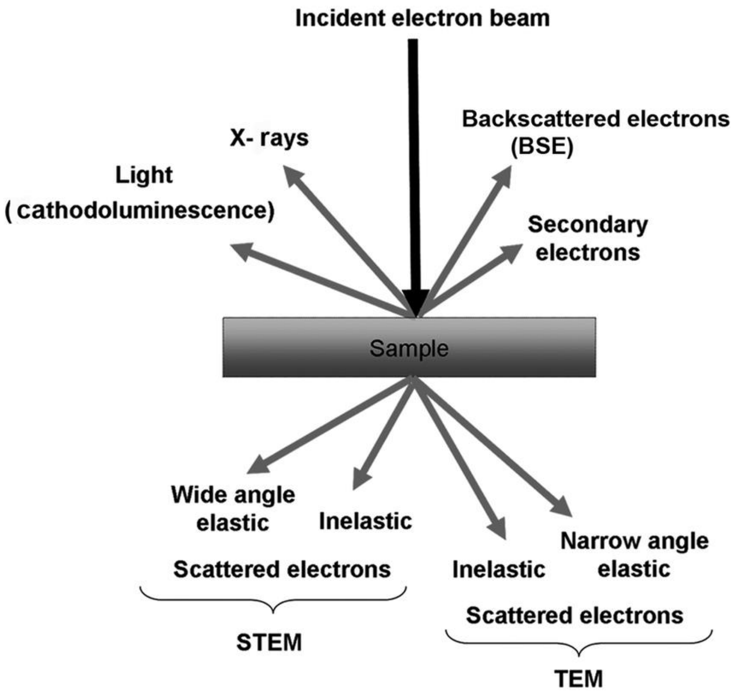
6. Some of the **characteristic X-rays** and the **continuum X-rays** are strong enough to act as **an excitation source**, **ejecting electrons from other atoms (usually of a different element),** **and therefore producing the corresponding radiation.** This phenomenon is known as **secondary fluorescence.**

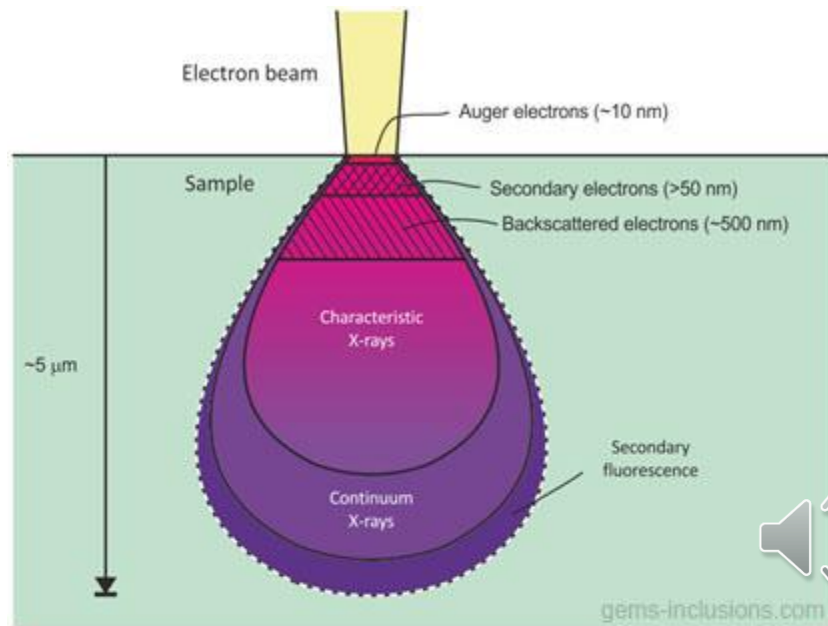
SEs and BSEs coming out of the samples are collected in order to produce the traditional SEM images (micrographs).



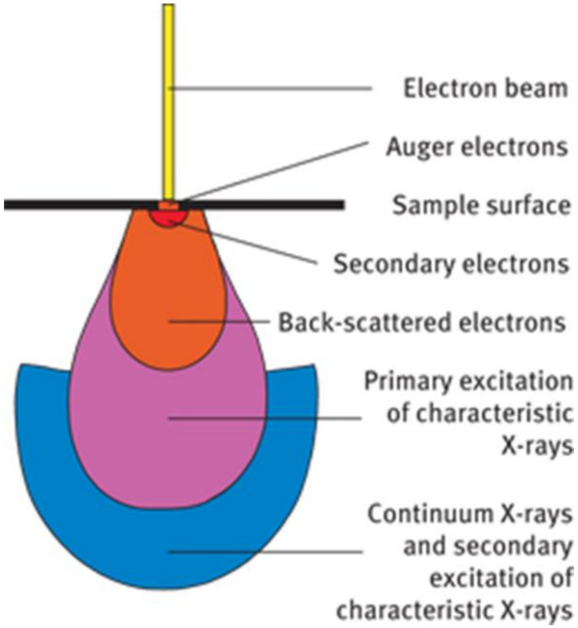

e-
BSE
e-
e-
Characteristic X-rays
e-
SE
N
e-
e-
K
L
M
e-
Main beam electrons
Backscattered electrons
Specimen electrons
Secondary electrons
Transmitted electrons
Elastic scattering
Inelastic scattering

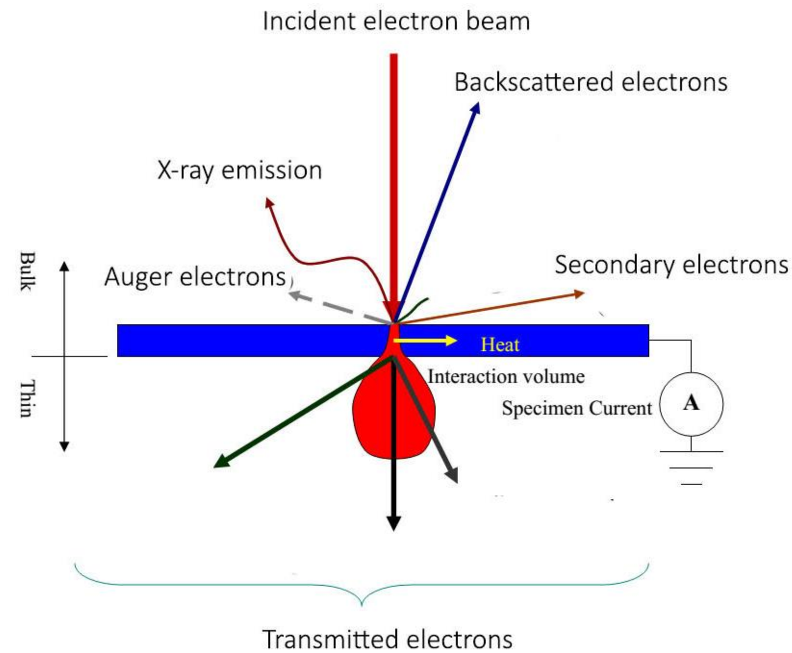

Electron / Sample Interaction
primary backscattered electrons
emission of energy (light, cathodo-luminiscence)
emission of secondary electrons (Auger)
emission of X-radiation
orbital jump
sample atom
scattering in
wide angles
scattering in
small angles
unscatterred primary electrons
elastically scattered primary electrons
inelastically scattered primary electrons (reduced impulse and energy)


Incident electron beam
X- rays
Backscattered electrons (BSE)
Light (cathodoluminescence)
Secondary electrons
Sample
Wide angle elastic
Inelastic
Scattered electrons
STEM
Inelastic
Narrow angle elastic
Scattered electrons
TEM


Electron beam
Auger electrons (~10 nm)
Sample
Secondary electrons (>50 nm)
Backscattered electrons (~500 nm)
~5 µm
Characteristic X-rays
Continuum X-rays
Secondary fluorescence
gems-inclusions.com

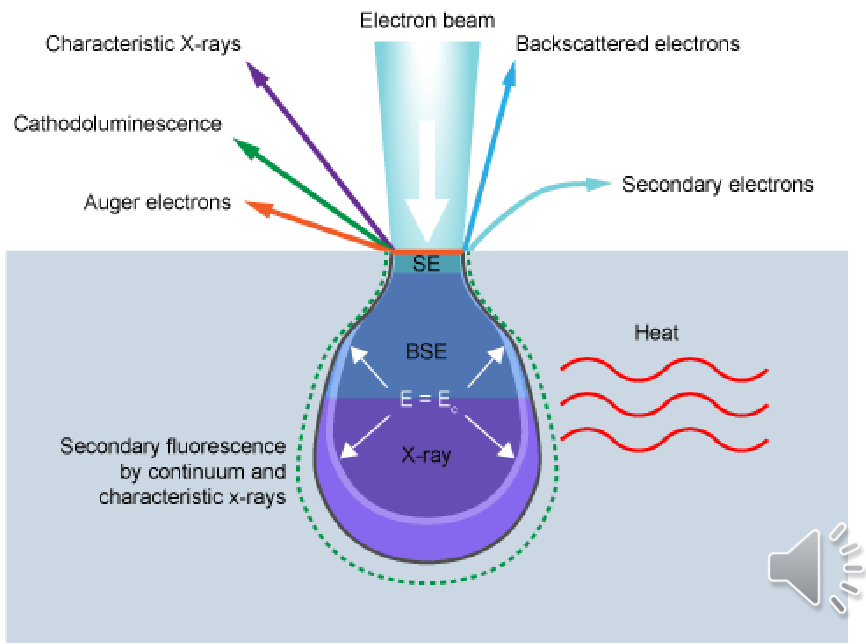
The volumes involved in the production of **secondary electron (SE), backscattered electron (BSE) and X-rays,** form into a shape that ranges from **a tear-drop to a semi circle within the specimen.**

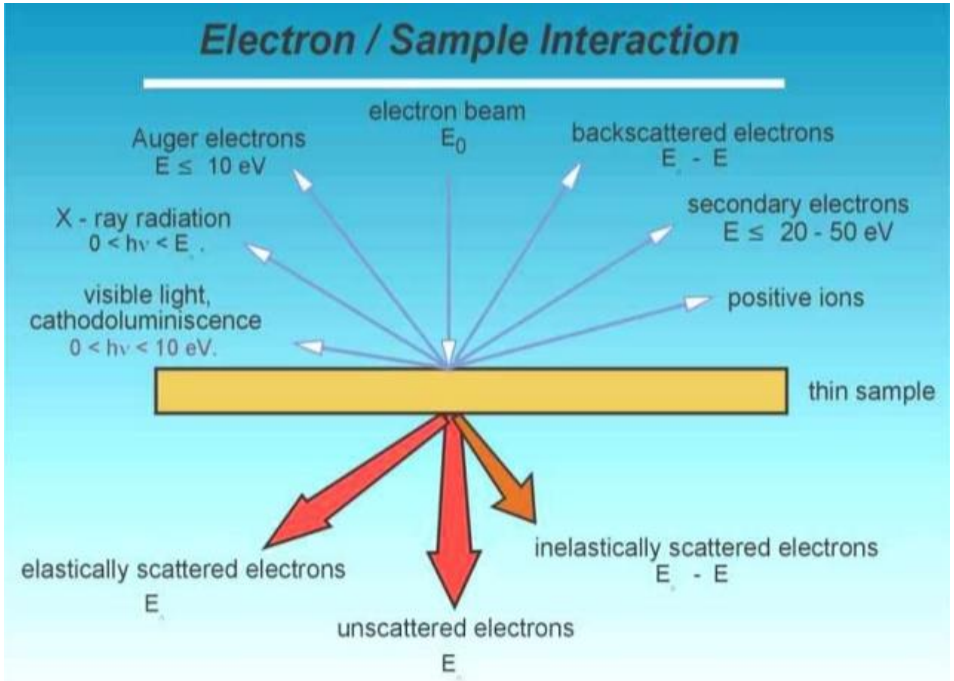


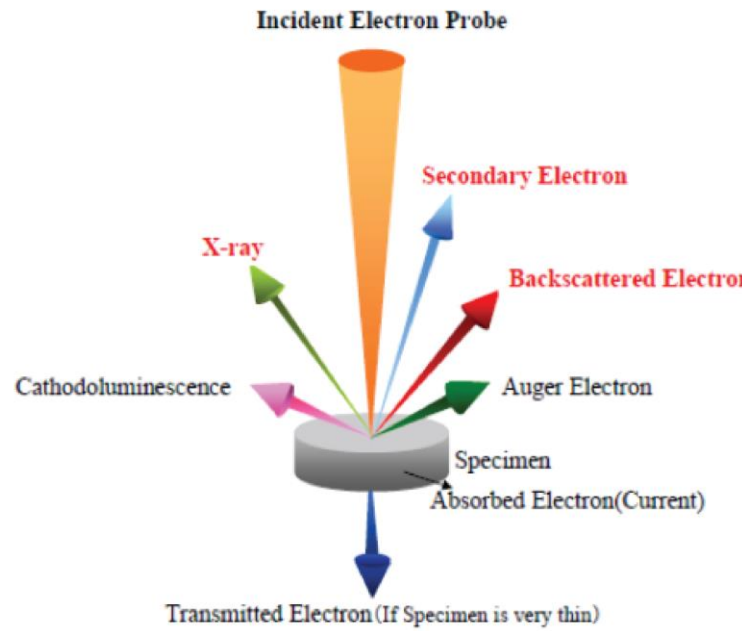


This shape is called an **interaction volume** and its depth and diameter depends on the **kV as well as the density of the specimen**.

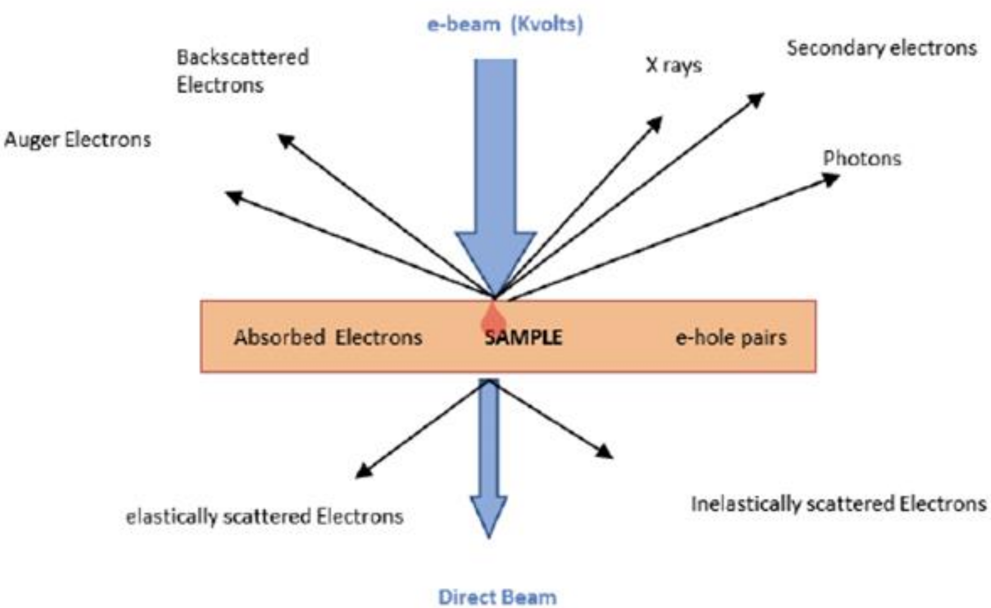
Approximately the top **15nm** of the volume comprises the zone from which **SE** can be collected, the top **40%** is the region from which **BSE** can be collected, however **X rays** can be collected from the entire region.

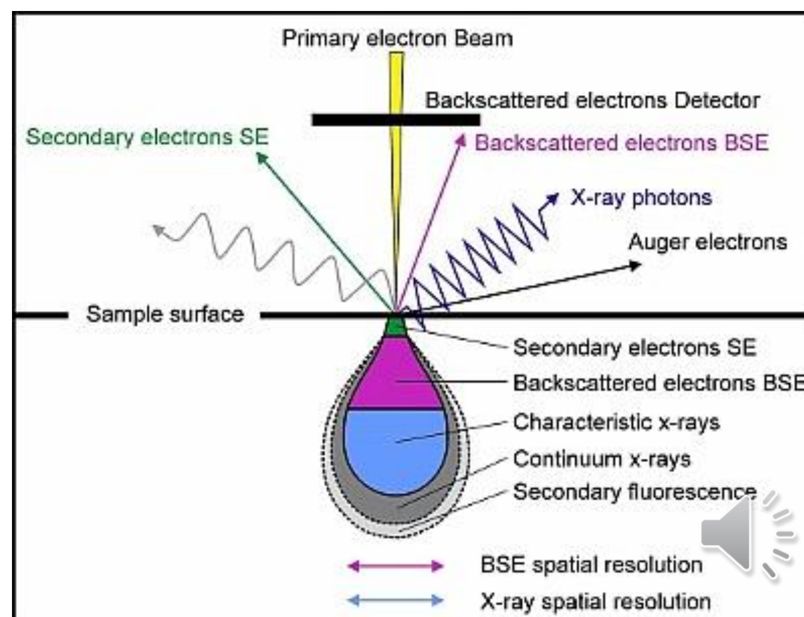


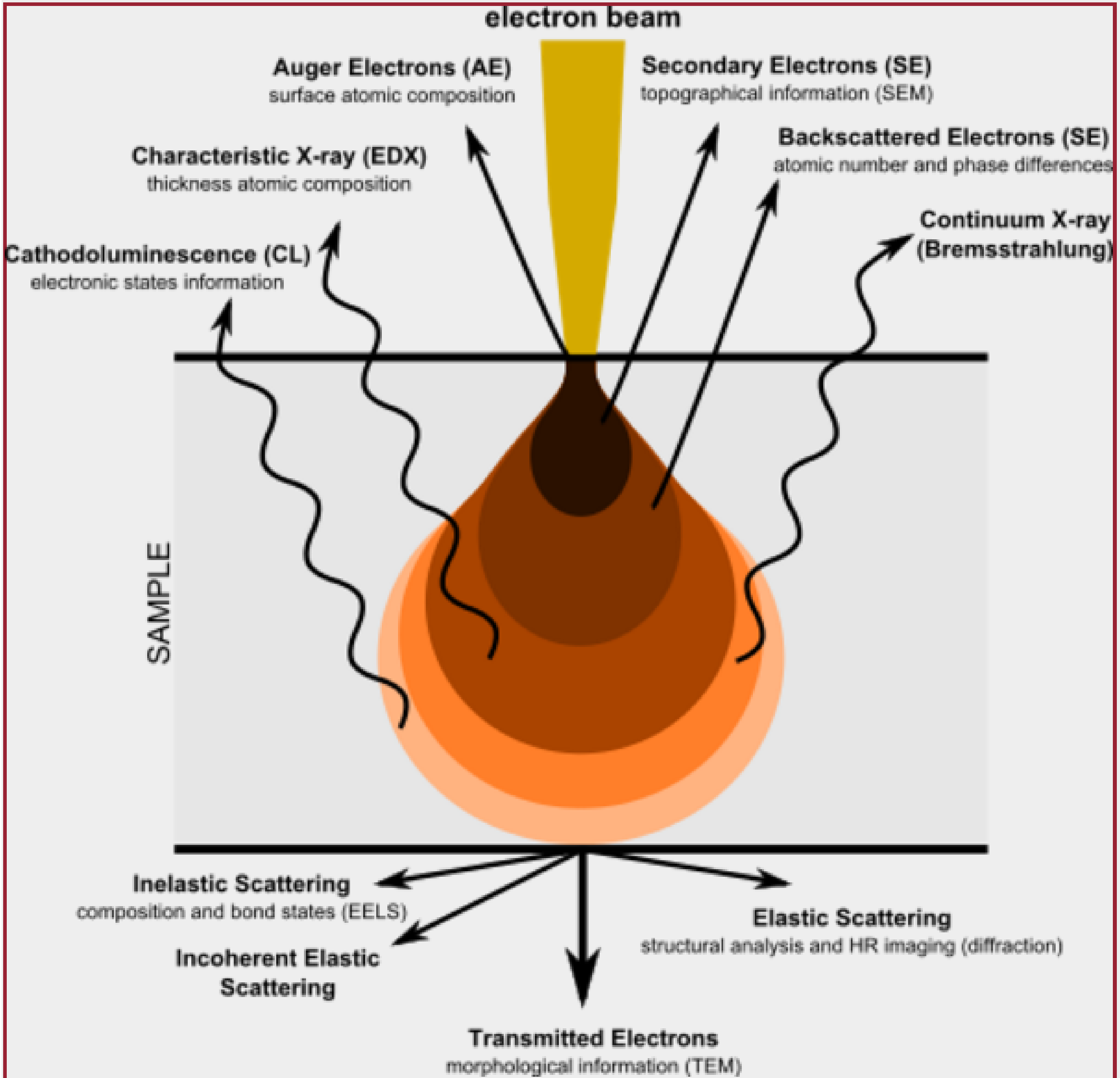




Signals generated by electron irradiation



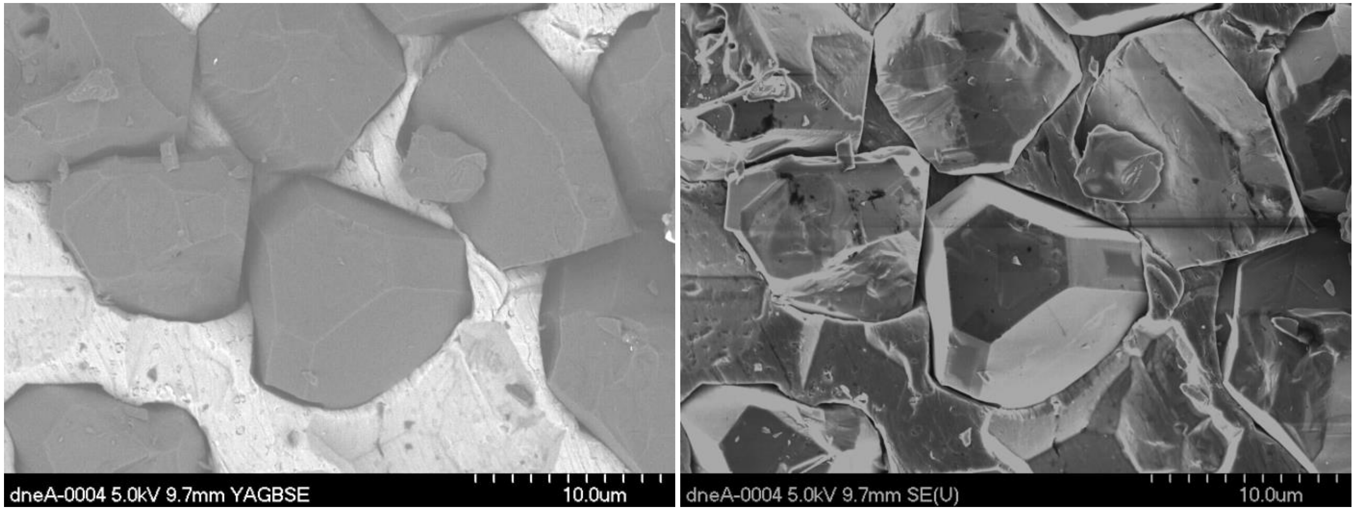


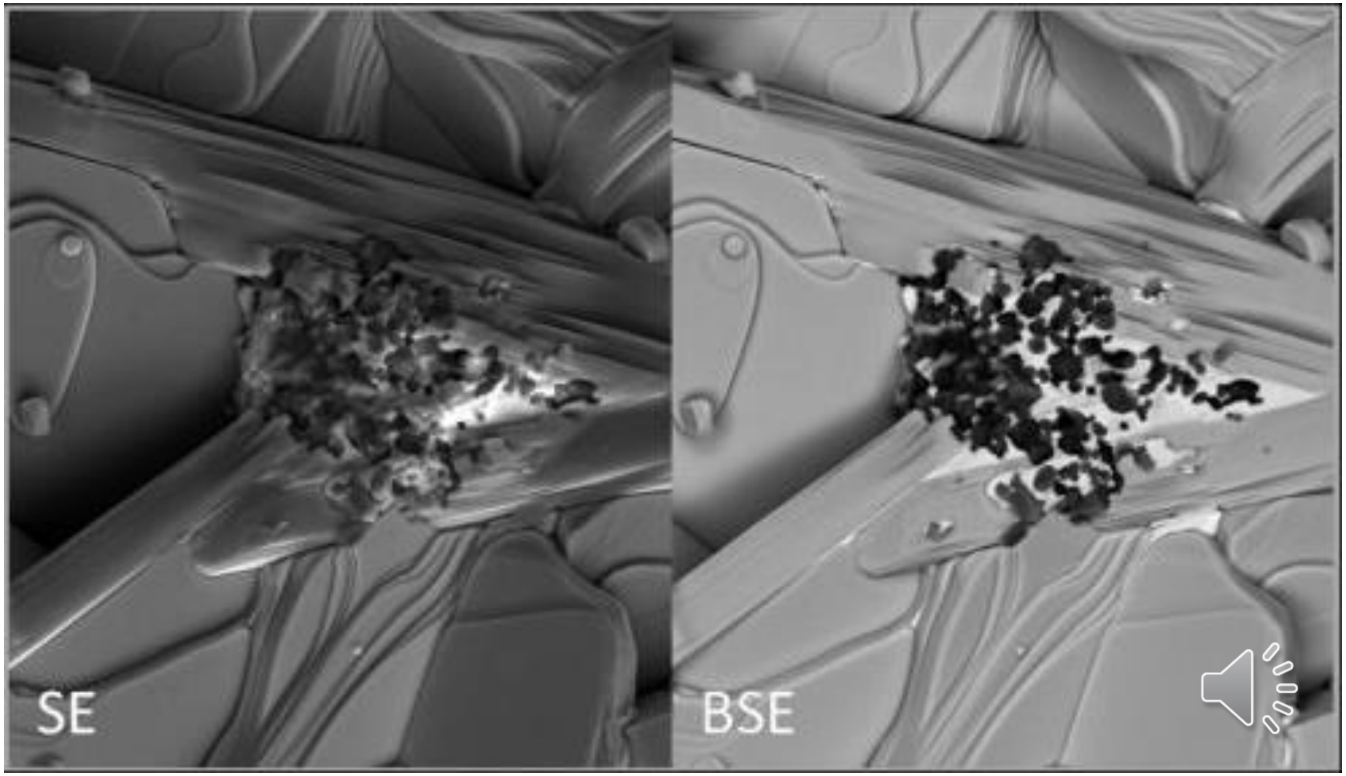

electron beam
Auger Electrons (AE)
surface atomic composition
Secondary Electrons (SE)
topographical information (SEM)
Characteristic X-ray (EDX)
thickness atomic composition
Backscattered Electrons (SE)
atomic number and phase differences
Cathodoluminescence (CL)
electronic states information
Continuum X-ray
(Bremsstrahlung)
SAMPLE
Inelastic Scattering
composition and bond states (EELS)
Incoherent Elastic
Scattering
Elastic Scattering
structural analysis and HR imaging (diffraction)
Transmitted Electrons
morphological information (TEM)

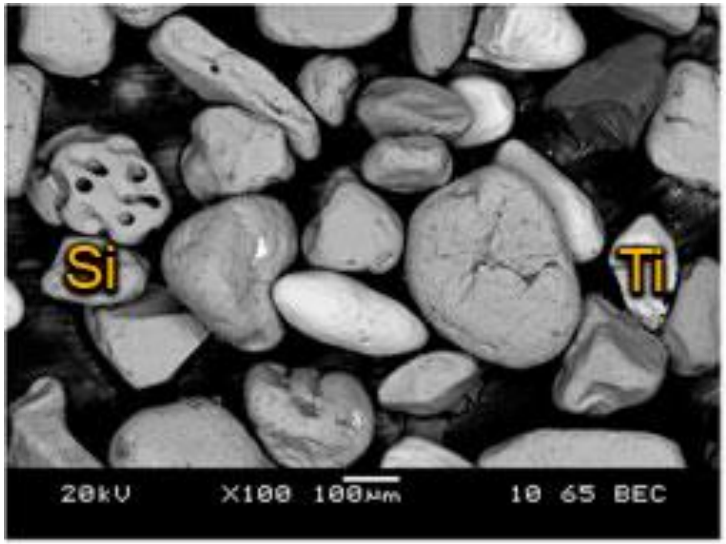
SE and BSE images

The **SE image** (right) shows clearly **the detail of the surface topography including the faceting نحت / نتوء of the crystals**.

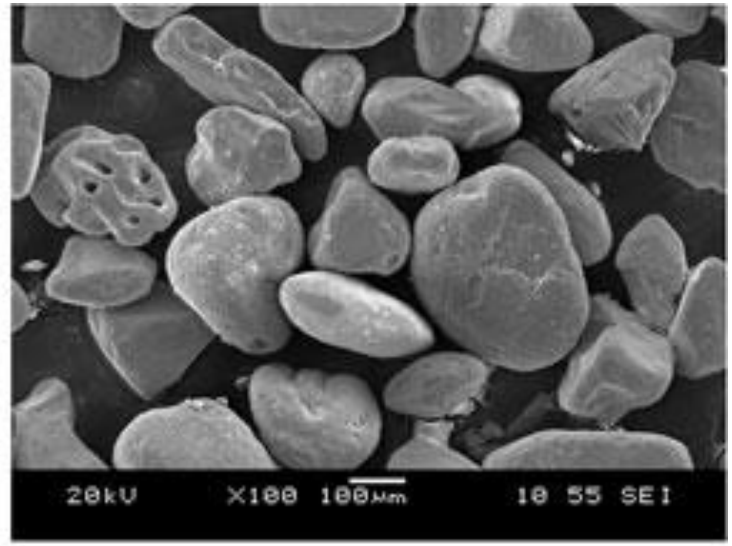
The **BSE image** (left) **shows much contrast between the crystals and the less ordered matrix they're growing on**. As the intensity of **backscattering is proportional to the mean atomic number of the atoms,** images developed from them provide **information on variations in sample composition.**



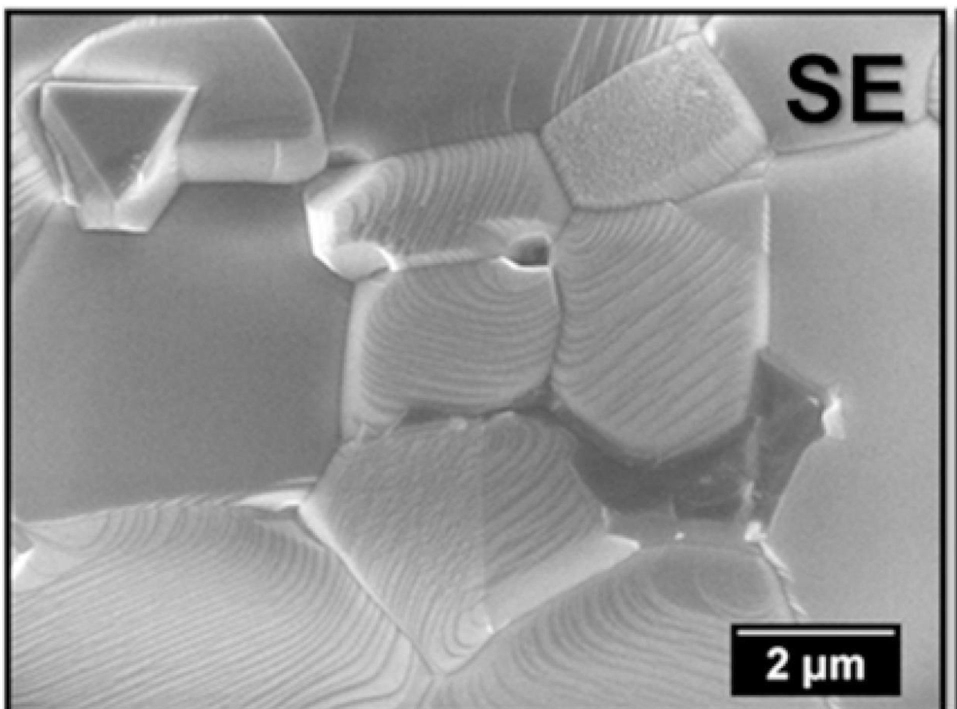


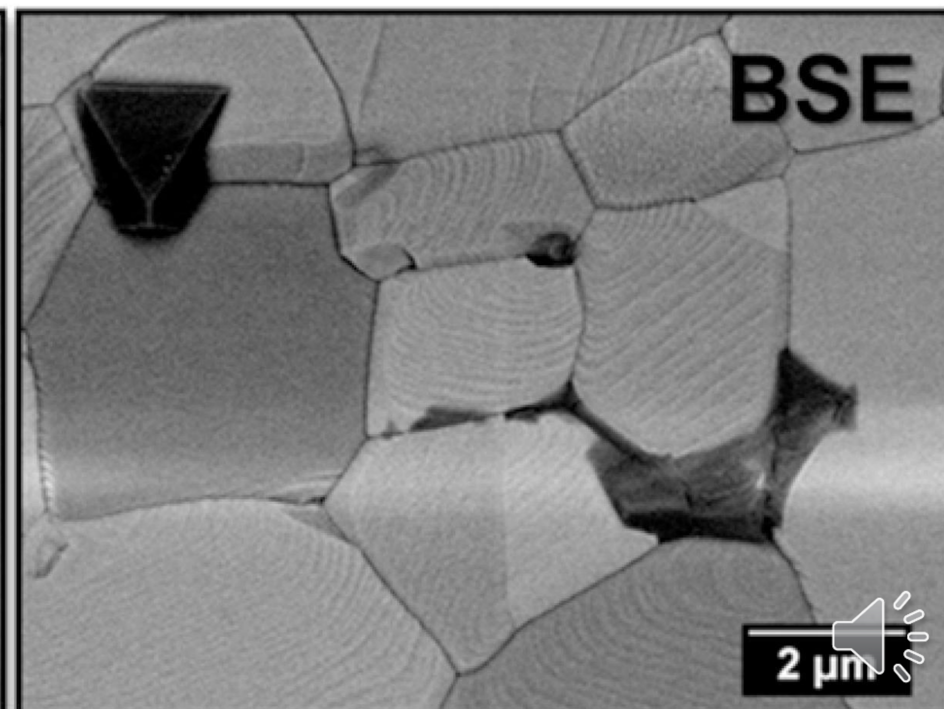


Backscattered electon image (BSE)

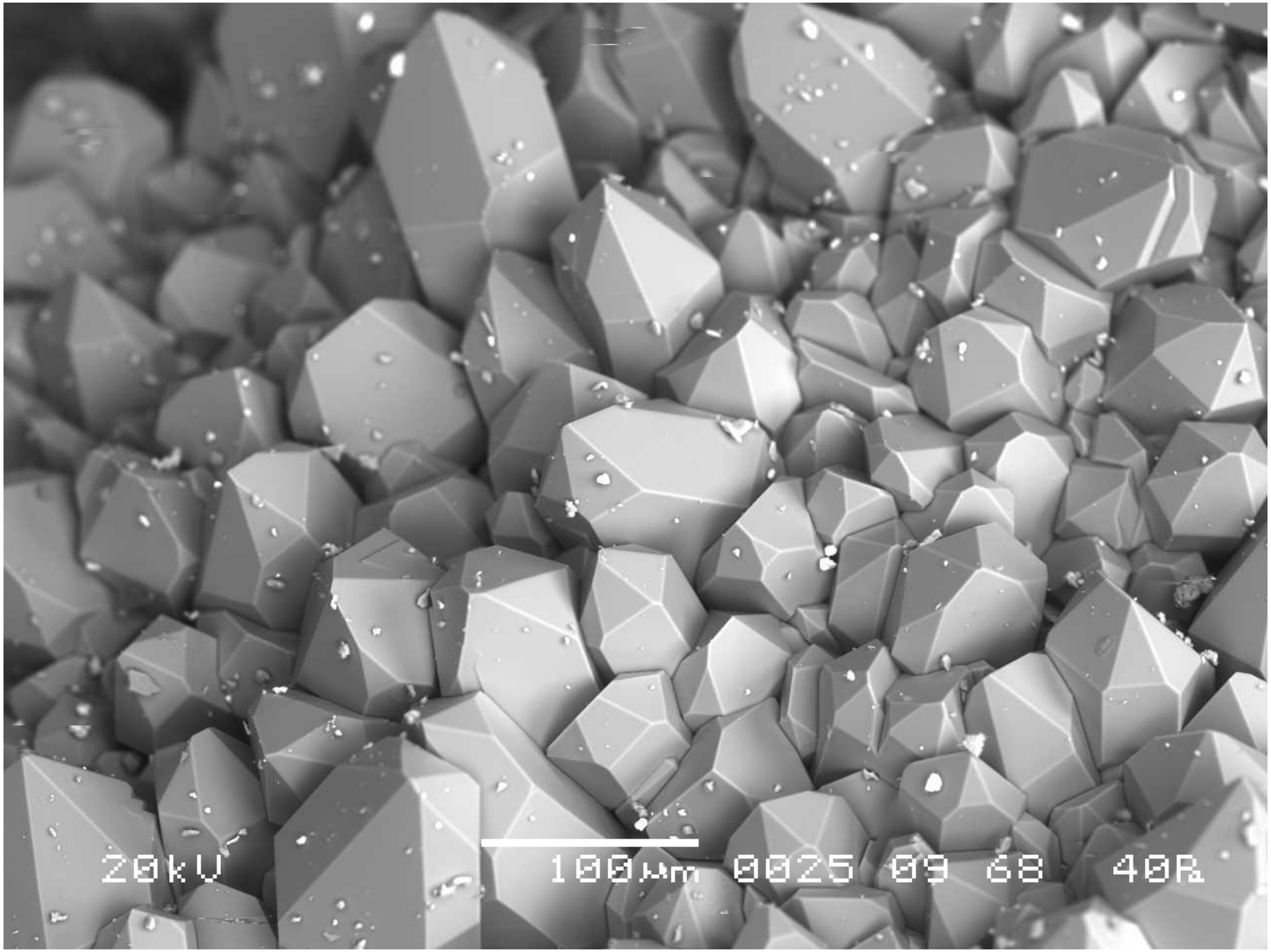


Secondary electron image (SE)

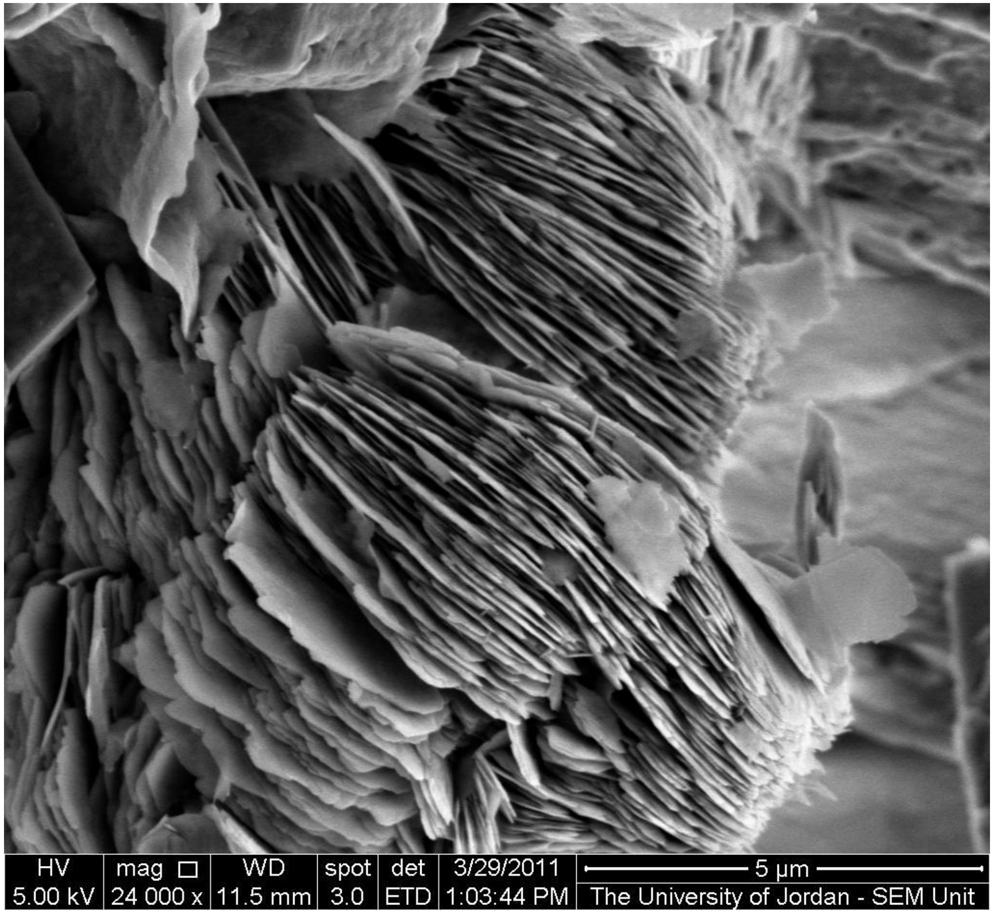




Examples of the SEM images of some minerals and rocks



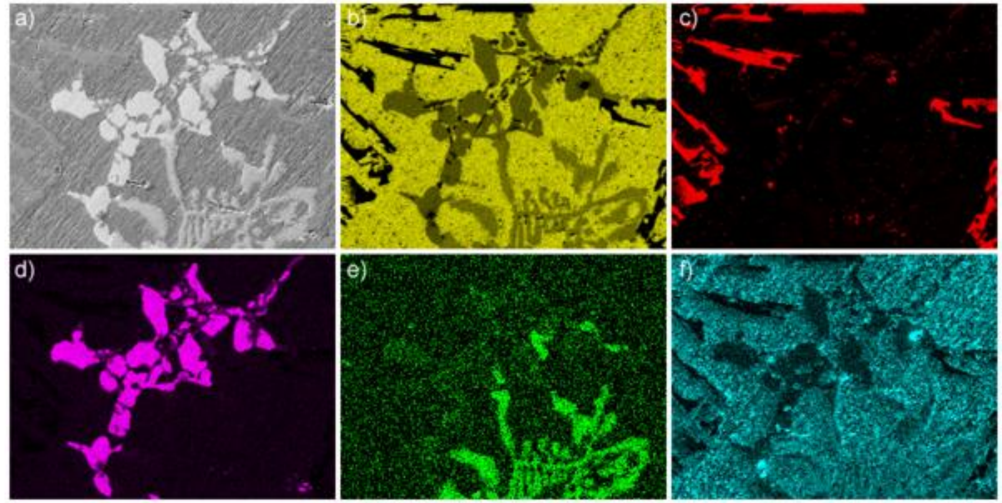
SEM of Quartz



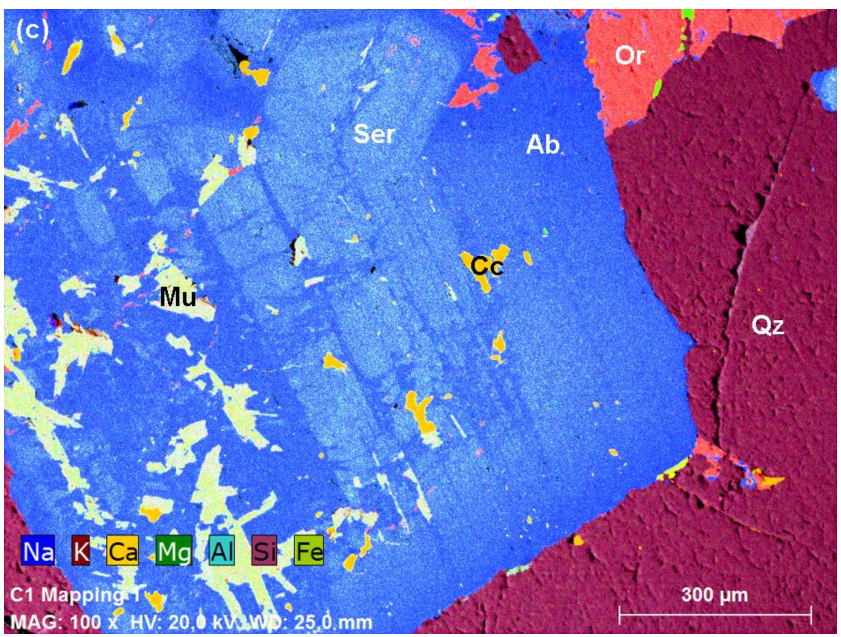
SEM of Kaolinite

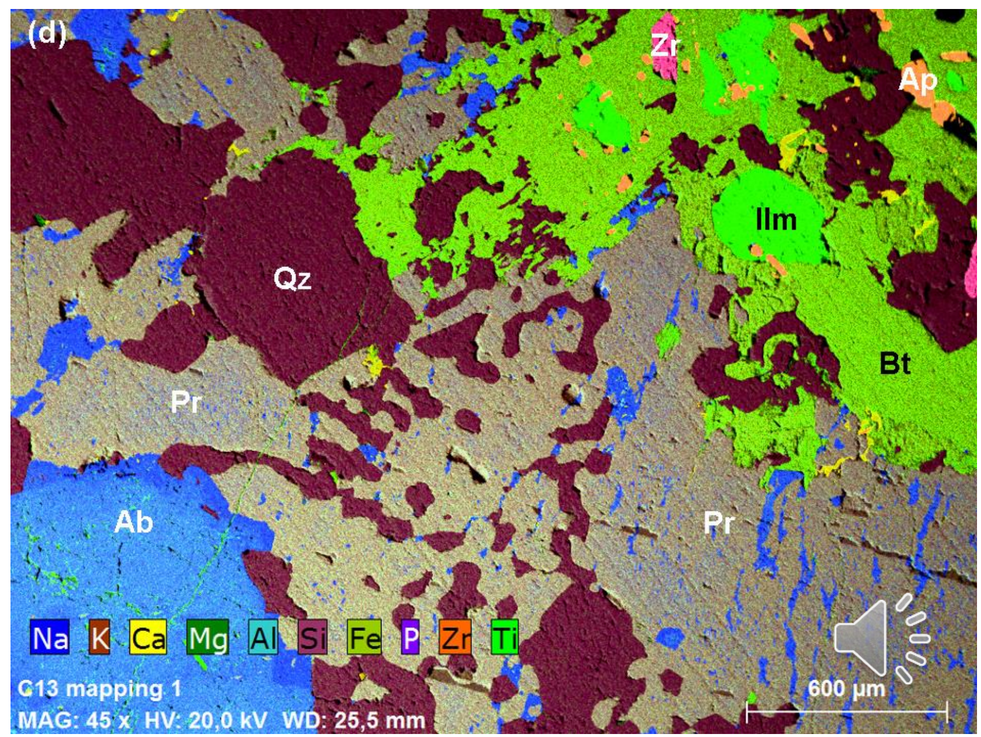
Chemical mapping

If a **semi-quantitative analysis** of each mineral grain is required, **chemical contrast from EDX is needed**.

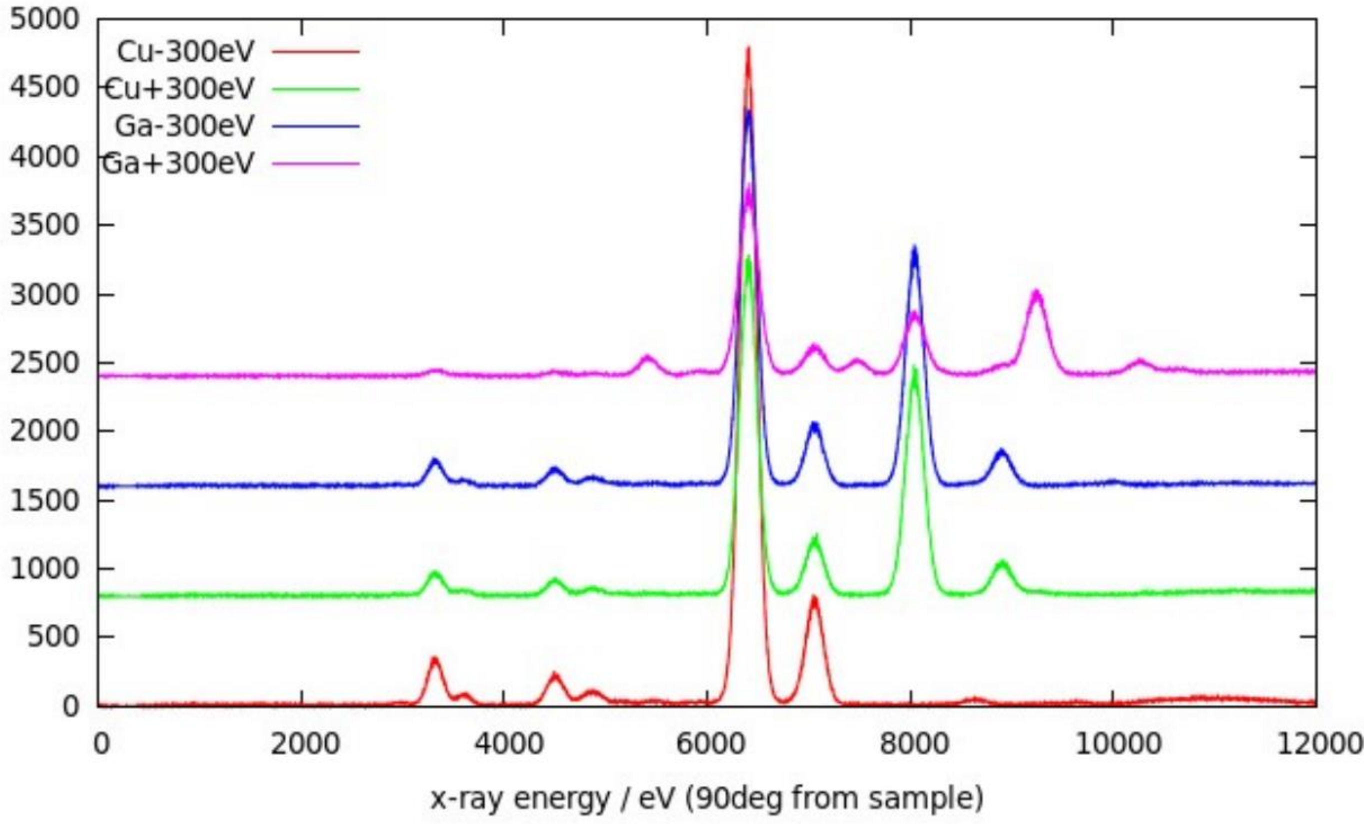


The figure shows the distribution of Al, Si, Cu, Fe, and O across the secondary-electron image top left. It becomes clear that Si (red) and Cu (purple) are totally segregated in this alloy, while Al (yellow) and iron (green) are present in all grains, although with variable concentrations.

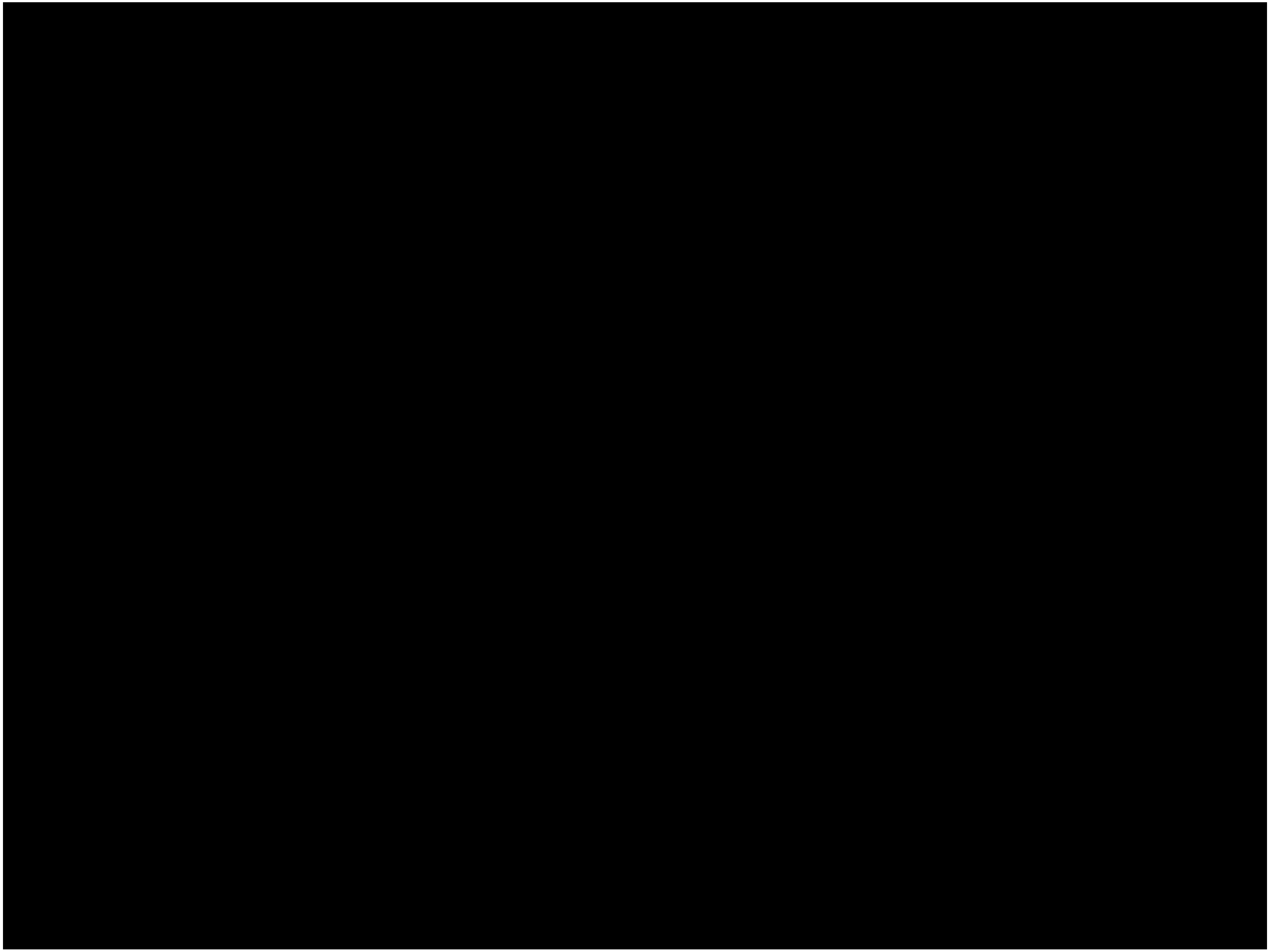




The **composition map** is only part of the information available with **SEM-EDX**. For each region, a full **characteristic** x-ray emission spectrum can be determined, showing the characteristic emission lines of each element present in the region with its relative strength. The figure shows the Kα and Kβ emission lines of potassium, iron, copper and gallium in a sample containing these elements (although this particular spectrum was recorded using incident x-rays rather than electrons to excite the emission). This demonstrates that an emission can only be excited if the source of the excitation is at least as energetic as the emission line itself.




Scanning Electron Microscope



https://www.youtube.com/watch?v=Mr9-1Sz_CK0