

## SEM: Points for discussion

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### 1. Explain the basic principle of SEM

Scanning electron microscopy was invented by Manfred Von Ardenne. It is surface microscopy, where the electrons are reflected from the surface and uses the behaviour of electrons to create a 3D image. Works on the principle of scattering of electrons on the surface of the sample

- a. Which are the typical acceleration voltages of the electron beam?  
between 0.1 kV and 50 kV

i. *How does the acceleration voltage affect the acquired SEM image?*

The focal length depends on the electron energy, which can be controlled by the applied acceleration voltage.

Lower acceleration voltage results in lower resolution of some features, with an image showing only the actual surface.

Higher acceleration voltage results in electron penetration within the sample. Features or components below the surface may show up in the image (higher resolution of multiple features), but the surface features may be reduced.

- b. Why is it called “scanning” microscopy?

Because in the characterization process, detection coils move the electron beam over the sample.

- c. What are secondary electrons, and how are they generated?

They are generated from inelastic electron collisions with a sample, and are used to get topographic information from the sample.

- d. What is the difference between secondary electrons and back scattered electrons?

Secondary Electrons (SE)	Backscattered Electrons (BSE)
Most used to form SEM Images	By convention, the limit between SE and BSE energy is defined as 50 eV
2-5 eV energies	Inelastic scattering with multiple collisions, with dispersion at large angles and with multiple energy losses
Generated by inelastic collisions	Backscattering coefficient is a function of atomic number, Z
Easily collected by a positively charged grid	BSE allows differentiating phases with different composition
SE exit the surface from a thin layer of a few nanometers	

- e. Explain how SEM images are produced?

In short, a detector collects secondary electrons to form an image. The electron collisions with the sample generate secondary electrons with different intensities. Emission intensity depends mostly on sample topography.

## 2. What is the resolution of SEM?

Is the ability to resolve and identify two points separated by a distance, which is limited by the electron beam diameter.

[1] Y. I. Vega, Introduction to Electron Microscopy: Scanning Electron Microscopy and EDS: Energy Dispersive X-ray Spectroscopy, in: M5052 Charact. Mater. Nanomater. - Grad. Progr. Nanotechnol., N.L. Monterrey, 2015.

## 3. Explain the main differences between SEM and TEM

SEM	TEM
Scanning Electron Microscopy generate images with electrons that are reflected from the surface.	Transmission Electron Microscopy creates images with electrons that go through a thin sample.
Image contrast depends on the intensity of the emitted secondary electrons, which is dictated by the sample topography.	Image contrast depends on the intensity of electrons that go through the sample in function of atomic number, density, atomic order (e.g. crystallinity), and sample thickness.
Typical maximum resolution is about 1.5–3 Nm. Resolution < 1nm is possible.	Typical maximum resolution is about 0.1 nm. 0.05 nm resolution is possible in special cases
Relatively simple sample preparation by surface observation. Sample almost always must be conducting	Sample must be thin enough (<100 nm). Complex sample preparation.
Large field of view at low magnification	Very small field of view
Operates in high vacuum. Low pressure operation options exist	High vacuum operation

[1] Y. I. Vega, Introduction to Electron Microscopy: Scanning Electron Microscopy and EDS: Energy Dispersive X-ray Spectroscopy, in: M5052 Charact. Mater. Nanomater. - Grad. Progr. Nanotechnol., N.L. Monterrey, 2015.

## 4. What are the main components of a scanning electron microscope?

- High power sources
- Vacuum environment for the electron beam
- Electron source/gun
- Lenses
- Scanning coils
- Final aperture
- Stigmator

[1] Y. I. Vega, Introduction to Electron Microscopy: Scanning Electron Microscopy and EDS: Energy Dispersive X-ray Spectroscopy, in: M5052 Charact. Mater. Nanomater. - Grad. Progr. Nanotechnol., N.L. Monterrey, 2015.

## 5. Why is it necessary to adjust the stigmator before acquiring SEM images?

Astigmatism is an asymmetric focus due to small mechanical imperfections within a lens, from where electrons are focused at different distances from the lens, causing lens aberrations. The stigmator compensates the defects on the beam focusing making sharper images. The stigmator is a lens rotated 90 degrees with respect to the distortion that induces antigmatism.

[1] Y. I. Vega, Introduction to Electron Microscopy: Scanning Electron Microscopy and EDS: Energy Dispersive X-ray Spectroscopy, in: M5052 Charact. Mater. Nanomater. - Grad. Progr. Nanotechnol., N.L. Monterrey, 2015.

## 6. What is the difference between magnification and resolution?

Magnification is the ratio between the area in which the image is projected and the observed area. This refers to the image size

Resolution is the ability to differentiate two points separated by a distance. This refers to resolving image detail.

[1] Y. I. Vega, Introduction to Electron Microscopy: Scanning Electron Microscopy and EDS: Energy Dispersive X-ray Spectroscopy, in: M5052 Charact. Mater. Nanomater. - Grad. Progr. Nanotechnol., N.L. Monterrey, 2015.

## 7. Why the sample has to be conducting?

To prevent charging during observation. The sample shall be electrically grounded to prevent static charging and allow the formation of secondary electrons from the sample (and prevent the formation secondary electrons from the SEM chamber walls).

[1] Y. I. Vega, Introduction to Electron Microscopy: Scanning Electron Microscopy and EDS: Energy Dispersive X-ray Spectroscopy, in: M5052 Charact. Mater. Nanomater. - Grad. Progr. Nanotechnol., N.L. Monterrey, 2015.

## 8. Describe sample preparation for SEM?

1. Conductive samples can be observed directly
2. Non-conductive samples require a thin conductive coating
3. Sample shall be dry and not contain volatile components
4. Sample shall be firmly mounted into the sample holder (no loose powders)

[1] Y. I. Vega, Introduction to Electron Microscopy: Scanning Electron Microscopy and EDS: Energy Dispersive X-ray Spectroscopy, in: M5052 Charact. Mater. Nanomater. - Grad. Progr. Nanotechnol., N.L. Monterrey, 2015.

## 9. How can you obtain images of non-conductive samples?

By the application of a thin conductive coating over the sample prior characterization.

[1] Y. I. Vega, Introduction to Electron Microscopy: Scanning Electron Microscopy and EDS: Energy Dispersive X-ray Spectroscopy, in: M5052 Charact. Mater. Nanomater. - Grad. Progr. Nanotechnol., N.L. Monterrey, 2015.

## 10. Describe advantages of SEM

- Relatively easy to use
- High resolution (about  $\leq 1$  nm)
- Large interval of magnification (From tens to hundreds of thousands)
- Large Depth of Field (300 times better than in optical microscopy and 3D appearance)
- Wide field of view
- Less sample preparation than TEM
- Direct Imaging of the Surface
- Can be combined with compositional analyses (e.g. EDS, BSE, TEM)

[1] Y. I. Vega, Introduction to Electron Microscopy: Scanning Electron Microscopy and EDS: Energy Dispersive X-ray Spectroscopy, in: M5052 Charact. Mater. Nanomater. - Grad. Progr. Nanotechnol., N.L. Monterrey, 2015.

## 11. Describe disadvantages and limitations of SEM

- High vacuum operations ( $10^{-5}$  torr) limits use for wet or biological samples
- Sample preparation may alter the sample
- Lower resolution than TEM
- Only the surface is observed
- Observation of 2D images of 3D surfaces
- Charge accumulation in insulating samples
- Thermal decomposition can take place in organic, biological and polymeric samples
- Radiation induced decomposition of organic materials

[1] Y. I. Vega, Introduction to Electron Microscopy: Scanning Electron Microscopy and EDS: Energy Dispersive X-ray Spectroscopy, in: M5052 Charact. Mater. Nanomater. - Grad. Progr. Nanotechnol., N.L. Monterrey, 2015.

## 12. What is ESEM?

The term ESEM (or Environmental SEM) is used to denote that a specific environment (atmosphere) is used. Can be used to image non-conductive samples.

[1] Y. I. Vega, Introduction to Electron Microscopy: Scanning Electron Microscopy and EDS: Energy Dispersive X-ray Spectroscopy, in: M5052 Charact. Mater. Nanomater. - Grad. Progr. Nanotechnol., N.L. Monterrey, 2015.

## 13. What is the difference between image acquisition in ESEM and SEM?

ESEM can produce images of wet samples or contained in low vacuum or gas. This technique facilitates the characterization of biological samples that are unstable in the high vacuum of typical electron microscopes.

[2] What are the main differences between an SEM, an ESEM, an SEM-FIB and an (S)TEM?, (n.d.). [https://www.horiba.com/en\\_en/cathodoluminescence-spectroscopy-sem-esem-sem-fib-stem/](https://www.horiba.com/en_en/cathodoluminescence-spectroscopy-sem-esem-sem-fib-stem/) (accessed 7 May 2020).

## 14. Describe what is EDS

Energy Dispersive (X-ray) Spectroscopy or EDX is an accessory for SEM and TEM to measure X-rays. Due to quantization of energy levels, the X-ray photon energies are characteristic of the element and the energy levels involved. However, EDS does not provide chemical information. X-rays are generated when electrons lose energy by interacting with positive nucleus (through the emission of photons).

[1] Y. I. Vega, Introduction to Electron Microscopy: Scanning Electron Microscopy and EDS: Energy Dispersive X-ray Spectroscopy, in: M5052 Charact. Mater. Nanomater. - Grad. Progr. Nanotechnol., N.L. Monterrey, 2015.

ESEM is especially useful for non-metallic, uncoated, non-conductive and biological materials. The presence of gas, mainly Argon, around a sample permits to work with pressure greater than 500 Pa compared to conventional SEM requirements samples under vacuum about  $10^3$  to  $10^4$  Pa.

[2] What are the main differences between an SEM, an ESEM, an SEM-FIB and an (S)TEM?, (n.d.). [https://www.horiba.com/en\\_en/cathodoluminescence-spectroscopy-sem-esem-sem-fib-stem/](https://www.horiba.com/en_en/cathodoluminescence-spectroscopy-sem-esem-sem-fib-stem/) (accessed 7 May 2020).

## 15. How EDS is used in combination with electron microscopy?

EDS is implemented when there is no information about the expected composition of the sample, a spectrum can be acquired instead from the whole observed area under SEM. The spectrum will show characteristic X-rays of all elements under the studied area, which allows the identification of concentration gradients and different phases.

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## 16. What are the applications of EDS?

- Detection of chemical residues at the nanogram level
- Study of morphology of particles and elemental composition

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## 17. Explain the difference between EDS and XRD

Energy Dispersive X-Ray Spectroscopy (EDS) is used for chemical identification of elements and their concentration. X-Ray Powder Diffraction (XRD) is used to identify spatial arrangements of atoms in crystalline phases.

In EDS, electrons knock out electrons from atoms, producing X-rays of a characteristic wavelength. These X-Rays are then detected to identify the element from which they were produced. In XRD, X-

rays of known wavelength are used to probe the structure of the material, using the lattice as a diffraction grating.

To perform an EDS you need an electron source. For XRD you need an X-ray source. When looking at chemical composition, concentration gradients, or solute segregation, use EDS. When looking at phase distribution, lattice strain fields or stored defect content, use XRD.

[3] C.M. Zamudio-Ortega, R. Contreras-Bulnes, R.J. Scougall-Vilchis, R.A. Morales-Luckie, O.F. Olea-Mejía, L.E. Rodríguez-Vilchis, Morphological, chemical and structural characterisation of deciduous enamel: SEM, EDS, XRD, FTIR and XPS analysis, *Eur. J. Paediatr. Dent.* 15 (2014) 275–280.

[4] What is the difference between XRD and EDX techniques?, (n.d.).

[https://www.researchgate.net/post/What\\_is\\_the\\_difference\\_between\\_XRD\\_and\\_EDX\\_techniques](https://www.researchgate.net/post/What_is_the_difference_between_XRD_and_EDX_techniques) (accessed 14 May 2020).