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# Precision and Detail Ensured by Scanning Electron Microscopy

A Scanning Electron Microscope equipped with Energy-Dispersive X-ray Spectroscopy (SEM-EDS) represents a powerful tool and robust analytical technique that offers an extensive array of data regarding the microstructural and compositional attributes of a diverse spectrum of materials. ALS Laboratories are equipped with a modern scanning electron microscope Tescan VEGA 3 LMU with an energy-dispersive detector (EDS) Oxford X-Max 20. It is an ideal technique for surface inspection, identification of the elemental composition of unknown particles in a sample, or advanced determination of particle size or type distribution.


*Figure 1: Scanning Electron Microscop*

## The Principle and Applications of SEM-EDS

SEM-EDS is characterised by its versatility, rapid analytical capabilities, and non-destructive nature, making it an invaluable tool in various domains such as geology, petrology, metallurgy, electrical engineering, pharmaceuticals, and environmental sciences.

The capability to perform high-resolution imaging in conjunction with elemental analysis enables thorough investigations into material properties, allowing for detailed characterisation and fostering scientific discoveries.

Traditional optical microscopes use light beams for imaging, with the wavelength of light limiting the maximum magnification to 1500–2000 times. In contrast, an electron microscope employs focused electrons instead of light. These electrons have a much shorter wavelength, enabling magnifications of up to one million times under ideal conditions.

The principle of SEM is based on "bombarding" the surface of the sample with a focused beam of so-called primary electrons. At the point of impact on the sample's surface, a range of interactions occurs between the primary electrons and the electrons in the atoms of the sample, resulting in the emission of detectable signals. This point of impact is referred to as the excitation volume, and its size primarily depends on the energy of the primary electrons and the elemental composition of the sample. The electron beam typically does not penetrate deeper than 1-2 μm.

## The most significant signals:

- **Secondary electrons** are ejected from the surface of the sample and have low energy values. They carry information about the surface topography of the sample and are used to create highly sharp images that can appear almost three-dimensional.
- **Backscattered electrons** are reflected from the sample surface and have high energy values. They are sensitive to differences in atomic number, thereby providing information about the elemental composition of the sample.
- **X-rays** are emitted when electrons transition between atomic energy levels and are detected using an Energy-Dispersive Spectroscopy (EDS) detector. The emission of these X-rays is unique to each element, allowing for the detection of individual elements within the sample.

### Requirements for analysed samples

The sample for SEM analysis must be dry, stable in a vacuum, conductive, and have to fit within the microscope chamber.

## Detailed Images

The SEM was primarily designed for surface observation since can provide detailed images of material surfaces and particles ranging from a few micrometers to several centimeters in size. It excels in the **identification of defects** such as cracks, wear, and corrosion, as well as in **surface topography**, including homogeneity, deposits, roughness, and shape, and the **size and shape of particles** measurements.

For example, SEM can produce images of metal parts to assess the homogeneity of an anti-corrosive phosphate layer on their surface (see Figure 2A), or to reveal early-stage corrosion on a cylinder (see Figure 2B, arrows indicate corrosion).


*Figure 2: (A) anti-corrosive layer; (B) corrosion traces*

## Elemental Analysis

An effective complement to the microscope itself is the aforementioned EDS detector, which can detect characteristic X-rays and assign them to specific elements. The latest systems are capable of detecting elements heavier than boron, i.e., elements with an atomic number >5. The Oxford AZtec X-Max 20 EDS detector in our laboratories can very quickly confirm or exclude the presence of elements heavier than beryllium (excluding hydrogen, helium, and lithium). The result is a spectrum from which the composition of the examined particle can be determined. This capability can be utilised, for example, to compare the sample material with a supplied standard or to observe changes occurring in the sample when exposed to various processes and conditions.

**Figures 3 (A/B)** show a detailed image and elemental spectrum of an unknown particle in a solution, which were used to identify its origin. Based on the sharp edges and elemental spectrum, the particle was identified as glass from the original ampoule containing the solution.

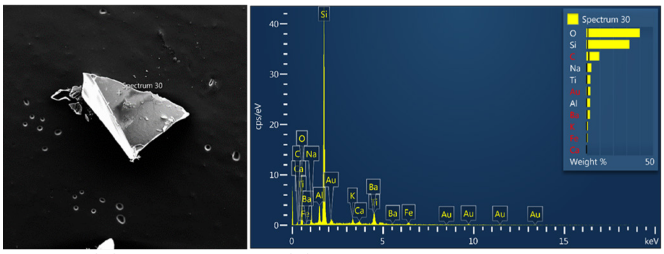


*Figure 3: (A) a piece of glass; (B) spectrum of glass*

## Feature Analysis

Using the software module "Feature Analysis," it is possible to automatically analyse a large number of particles in terms of morphology, chemical composition, or a combination of both. A significant advantage over common techniques such as laser diffraction is the ability to directly image the analysed particles. The particles can be sorted entirely automatically, either by morphology, by defining parameters such as length, area, shape, etc., or by chemical composition, for example, whether they contain a specific element or not. The outputs can include various graphs and tables (see **Figures 4 (A/B)**.

Using Feature Analysis module, for example, the efficiency of the filtration system, the homogeneity of the material, or only selected particles (e.g. respirable fibres according to WHO, length >5 um, width <3 um, ratio >3:1) can be analysed from the sample.

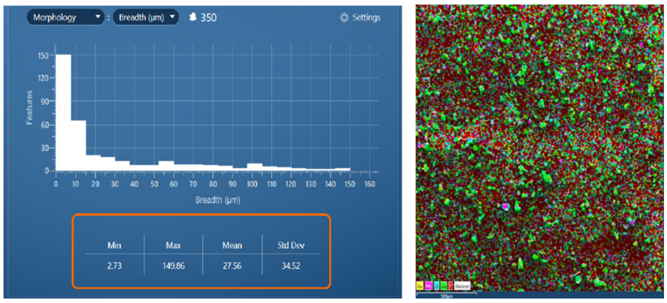


*Figure 4: (A) a distribution diagram of particles; (B) highlighted particles of material*

The most common analyses in ALS Laboratories include: the anti-corrosion coating of car parts, the specification of foreign particles, a very frequent analysis is an identification of deposits on filters, identification of sediments, or stains, and defects on the surface of various materials and products. SEM-EDS analysis is also often an integral part of production processes, where quality control of intermediate and final products is crucial.

**There are many possible applications of the technique.**

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## References

LINK https://myscope.training/SEM_SEM_Basics

LINK https://nano.vscht.cz/wp-content/uploads/navody/charakterizace/EM.pdf