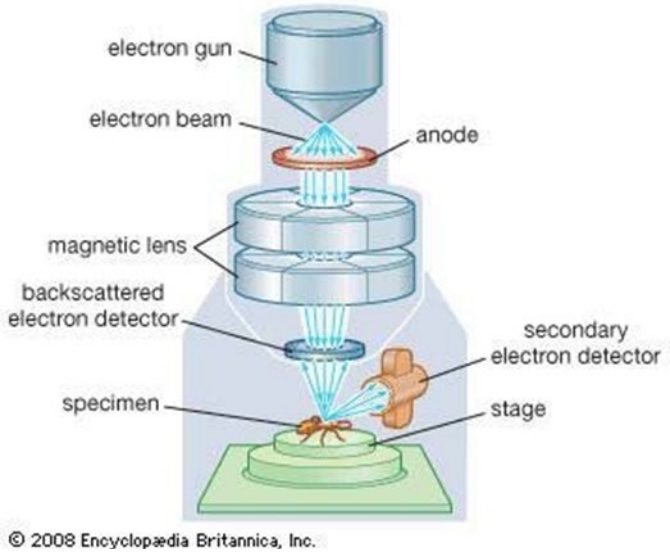
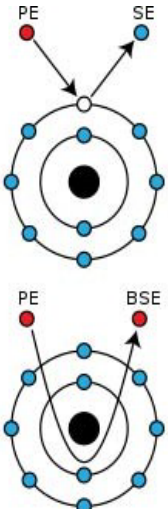
Scanning Electron Microsope

A **scanning electron microscope** (**SEM**) is a microscope that produces images of a sample by scanning it with a focused beam of electrons produced by an electron gun. The electrons interact with atoms in the sample, producing various signals (scattered electrons, X-rays, etc.) that contain information about the sample's surface topography and composition. The electron beam is generally scanned across the surface, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer (nm). Specimens can be observed in high vacuum, in low vacuum, in wet conditions (i.e. environmental SEM), and at a wide range of cryogenic or elevated temperatures.

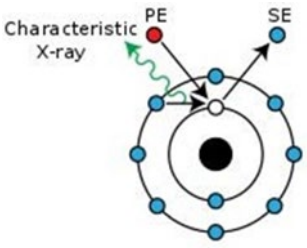


Scanning Electron Microscope Diagram
(https://www.britannica.com/technology/scanning-electron- microscope)



Mechanisms of emission of secondary electrons, backscattered electrons, and characteristic X-rays from atoms of the sample By Rob Hurt - Own work, CC BY-SA 4.0, https://commons.wikimedia.org/w/index.php?curid=50931451

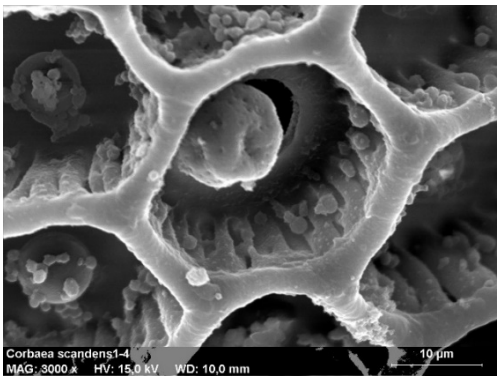
The signals result from interactions of the electron beam with atoms at various depths within the sample. In the most common or standard detection mode, secondary electron imaging or SEI, the secondary electrons are emitted from very close to the specimen surface. Consequently, SEM can produce very high-resolution images of a sample surface, revealing details less than 1 nm in size. Back-scattered electrons (BSE) are beam electrons that are reflected from the sample by elastic scattering.



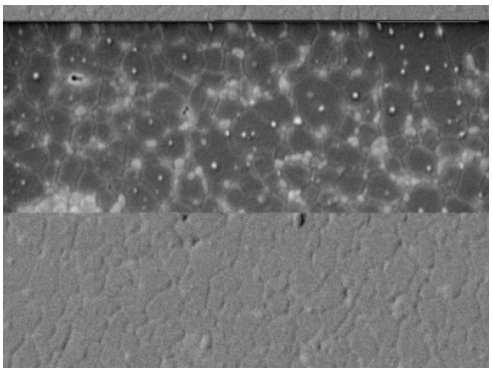
They emerge from deeper locations within the specimen and consequently the resolution of BSE images is generally poorer than SE images. However, BSE are often used in analytical SEM along with the spectra made from the characteristic X- rays, because the intensity of the BSE signal is strongly related to the atomic number of the specimen. BSE images can provide information about the distribution of different elements in the sample. For the same reason, BSE imaging can image colloidal gold immuno-labels of 5 or 10 nm diameter, which would otherwise be difficult or impossible to detect in secondary electron images in biological specimens. Characteristic X-rays are emitted when the electron beam removes an inner shell electron from the sample, causing a higher-energy electron to fill the shell and release energy. These characteristic X-rays are used to identify the composition and measure the abundance of elements in the sample.

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. A wide range of magnifications is possible, from about 10 times (about equivalent to that of a powerful hand-lens) to more than 500,000 times, about 250 times the magnification limit of the best light microscopes.

Example Data:



SEM image of Cobaea scandens pollen. Note the scale bar. By Marie Majaura - Own work, CC BY-SA 3.0, https://commons.wikimedia.org/w/index.php?curid=3105565



Comparison of SEM techniques: Top: backscattered electron analysis – composition Bottom: secondary electron analysis – topography Public Domain, https://en.wikipedia.org/w/index.php?curid=14672854

References:

Much of the above text was modified from https://en.wikipedia.org/wiki/Scanning_electron_microscope

- http://serc.carleton.edu/research_education/geochemsheets/techniques/SEM.html
- https://cmrf.research.uiowa.edu/scanning-electron-microscopy
- http://science.howstuffworks.com/scanning-electron-microscope.htm

Video Links:

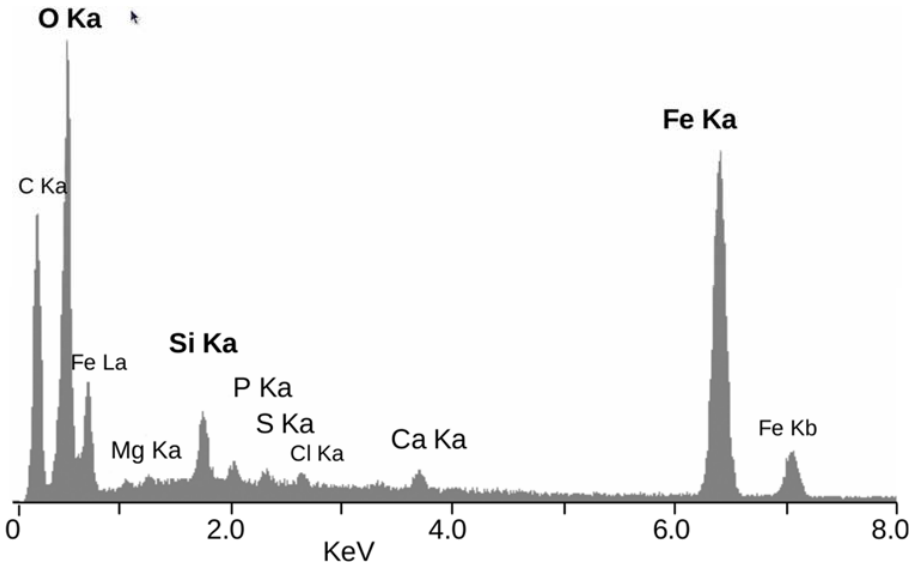
- https://www.**youtube**.com/watch?v=sfogToduqGQ
- ttps://www.**youtube**.com/watch?v=bfSp8r-YRw0
- https://www.**youtube**.com/watch?v=GY9lfO-tVfE

Energy-dispersive X-ray spectroscopy

Energy-dispersive X-ray spectroscopy (**EDS**, **EDX**, or **XEDS**), sometimes called **energy dispersive X-ray analysis** (**EDXA**) or **energy dispersive X-ray microanalysis** (**EDXMA**), is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing a unique set of peaks on its electromagnetic emission spectrum (which is the main principle of spectroscopy).

To stimulate the emission of characteristic X-rays from a specimen, a high-energy beam of charged particles such as electrons (like the beam used in scanning electron microscopy) or protons, or a beam of X-rays, is focused into the sample being studied. At rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells bound to the nucleus. The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron was. An electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer. As the energies of the X-rays are characteristic of the difference in energy between the two shells and of the atomic structure of the emitting element, EDS allows the elemental composition of the specimen to be measured.

Example Data:



EDS spectrum of the mineral crust of the vent shrimp Rimicaris exoculata *Most of these peaks are X-rays given off as electrons return to the K electron shell.(K-alpha and K-beta lines) One peak is from the L shell of iron.CC BY 3.0, https://commons.wikimedia.org/w/index.php?curid=7676417*

Video Links:

https://www.**youtube**.com/watch?v=TLeKyoGHiMA

https://www.**youtube**.com/watch?v=Zmvm-eX4H18

ttps://www.**youtube**.com/watch?v=Kl_K4N3EHbA

References:

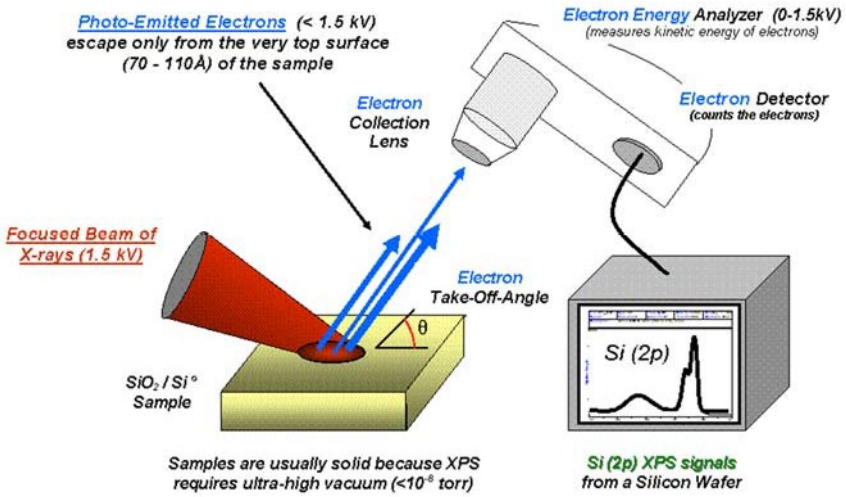
Much of the above text was modified from https://en.wikipedia.org/wiki/Energy-dispersive_X-ray_spectroscopy

http://www.charfac.umn.edu/instruments/eds_on_sem_primer.pdf

X-ray Photoelectron Spectroscopy (XPS)

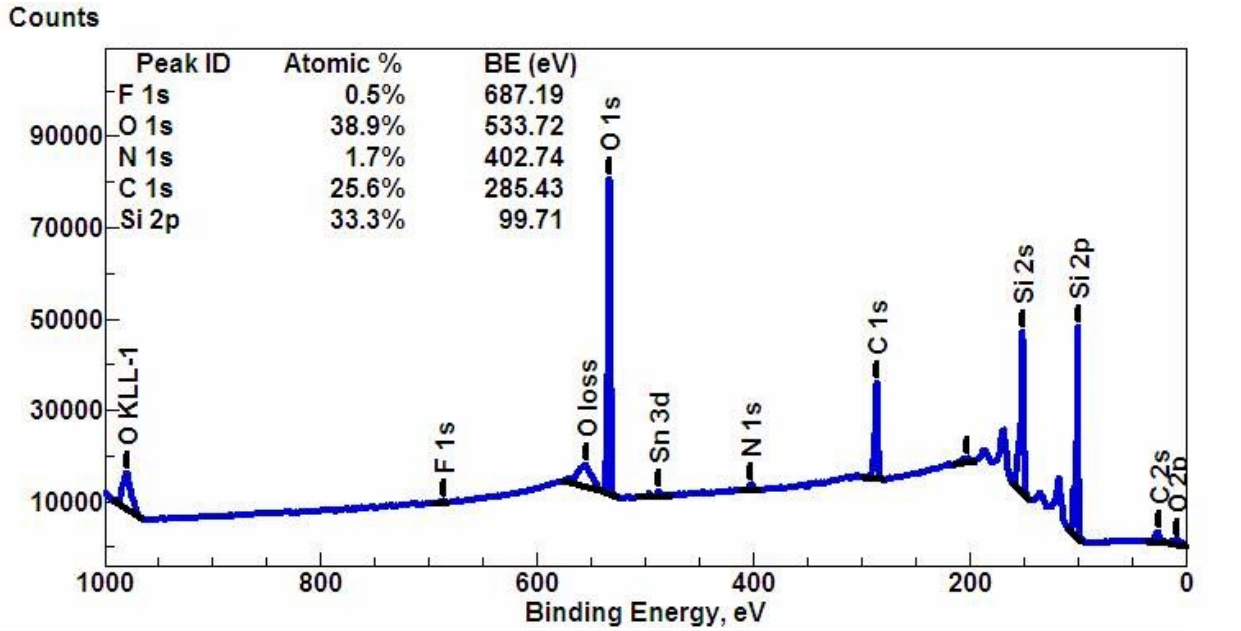
(Other name: Electron Spectroscopy for Chemical Analysis, ESCA)

X-ray photoelectron spectroscopy (XPS) is a surface-sensitive quantitative spectroscopic technique that measures the elemental composition at the parts per thousand range, empirical formula, chemical state and electronic state of the elements that exist within a material. XPS spectra are obtained by irradiating a material with a beam of X-rays while simultaneously measuring the kinetic energy and number of electrons that escape from the top 0 to 10 nm of the material being analyzed. XPS requires high vacuum (P ~ 10^{-8} millibar) or ultra-high vacuum (UHV; P < 10^{-9} millibar) conditions. A typical XPS spectrum is a plot of the number of electrons detected (sometimes per unit time) (y-axis) versus the binding energy of the electrons detected (x-axis). Each element produces a characteristic set of XPS peaks at characteristic binding energy values that directly identify each element that exists in or on the surface of the material being analyzed. (Electron binding energy is a measure of the energy required to free electrons from their atomic orbits. For each electron in a molecule there will be a specific binding energy required to remove it.) These characteristic spectral peaks correspond to the electron configuration of the electrons within the atoms, e.g., 1*s*, 2*s*, 2*p*, 3*s*, etc. The number of detected electrons in each of the characteristic peaks is directly related to the amount of element within the XPS sampling volume.



To count the number of electrons during the acquisition of a spectrum with a minimum of error, XPS detectors must be operated under ultra-high vacuum (UHV) conditions because electron counting detectors in XPS instruments are typically one meter away from the material irradiated with X-rays. This long path length for detection requires such low pressures.

Example Data:



Wide-scan or survey spectrum of a silicon wafer, showing all elements present. A survey spectrum is usually the starting point of most XPS analyses because it shows all elements present on the sample surface and allows one to set up subsequent high-resolution XPS spectra acquisition. The inset shows a quantification table indicating all elements observed, their binding energies, and their atomic percentages. By Bvcrist - Own work (enwiki), CC BY-SA 3.0, https://commons.wikimedia.org/w/index.php?curid=17267639

References:

Much of the above text was modified from https://en.wikipedia.org/wiki/X-ray_photoelectron_spectroscopy

https://en.wikipedia.org/wiki/Ionization_energy

http://xpssimplified.com/whatisxps.php

http://xpssimplified.com/knowledgebase.php

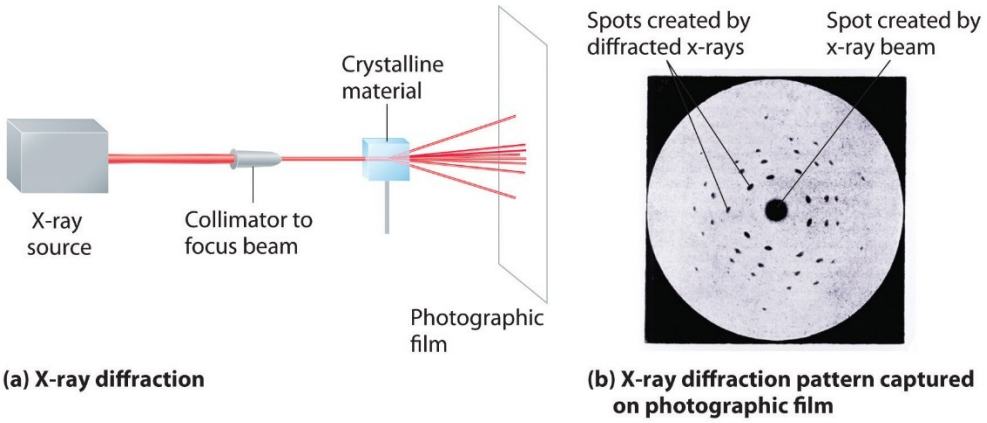
Video Links:

https://www.**youtube**.com/watch?v=8njmZdnvjZs

https://www.**youtube**.com/watch?v=rR7GwTqxFOE

X-ray Diffraction

X-ray diffraction (XRD) is a tool used for identifying the atomic and molecular structure of a crystal, in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific directions. Many materials can form crystals—such as salts, metals, minerals, semiconductors, as well as various inorganic, organic and biological molecules. In a single-crystal X-ray diffraction measurement, a crystal is mounted on a goniometer. The goniometer is used to position the crystal at selected orientations. The crystal is illuminated with a finely focused monochromatic beam of X-rays, producing a diffraction pattern of regularly spaced spots known as *reflections*.



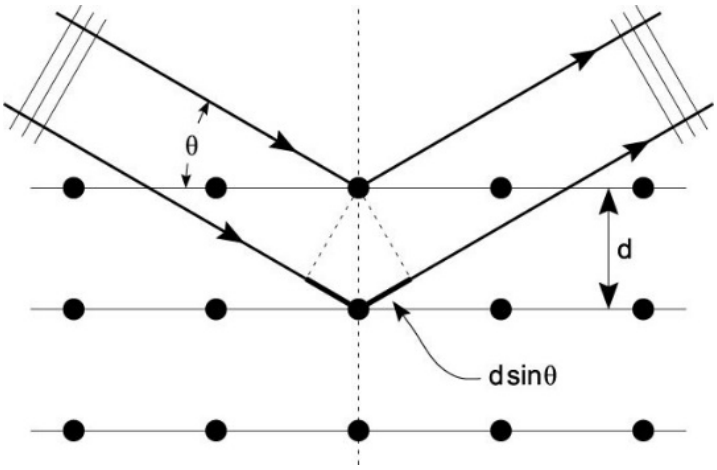
(a) X-ray diffraction

(b) X-ray diffraction pattern captured on photographic film

These illustrations show (a) a schematic drawing of x-ray diffraction and (b) the x-ray diffraction pattern of a zinc blende crystalline solid captured on photographic film.

https://saylordotorg.github.io/text_general-chemistry-principles-patterns-and-applications-v1.0/s16-03-structures-of- simple-binary-co.html

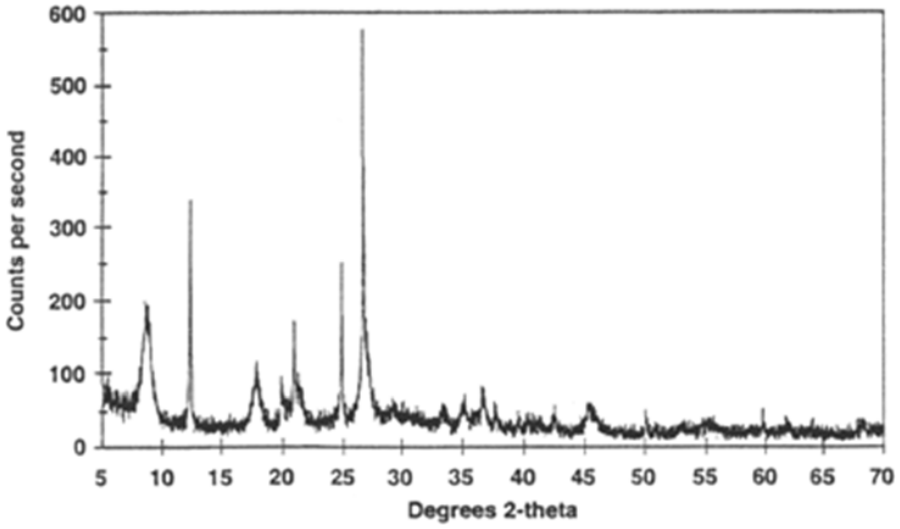
Crystals are regular arrays of atoms, and X-rays can be considered waves of electromagnetic radiation. Atoms scatter X-ray waves, primarily through the atoms' electrons. Just as an ocean wave striking a lighthouse produces secondary circular waves emanating from the lighthouse, so an X-ray striking an electron produces secondary spherical waves emanating from the electron. This phenomenon is known as elastic scattering, and the electron (or lighthouse) is known as the *scatterer*. A regular array of scatterers produces a regular array of spherical waves. Although these waves cancel one another out in most directions through destructive interference, they add constructively in a few specific directions: when conditions satisfy Bragg's Law: $2d \sin 0 = n$;i_



The incoming X-ray (coming from upper left) causes each atom (scatterer) to re-radiate a small portion of its intensity as a spherical wave. If atoms are arranged symmetrically with a separation d, these spherical waves will be in sync (add constructively) only in directions where their path-length difference 2d sin θ equals an integer multiple of the wavelength λ. In that case, part of the incoming beam is deflected by an angle 2θ, producing a reflection spot in the diffraction pattern. By Hydrargyrum - Own work, CC BY-SA 3.0, https://commons.wikimedia.org/w/index.php?curid=17543875

Here d is the spacing between diffracting planes, θ is the incident angle, n is any integer, and λ is the wavelength of the beam. This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample. These diffracted X-rays are then detected, processed and counted. By scanning the sample through a range of 2θangles, all possible diffraction directions of the lattice should be attained due to the random orientation of the material. Conversion of the diffraction peaks to d-spacings allows identification of the material because each material has a set of unique d-spacings. Typically, this is achieved by comparison of d-spacings with standard reference patterns.

Example Data:



X-ray powder diffractogram. Peaks occur where the X-ray beam has been diffracted by the crystal lattice. The unique set of d-spacings derived from this pattern can be used to 'fingerprint' the material.

http://serc.carleton.edu/details/images/8418.html

Video Links:

https://www.youtube.com/watch?v=UfDW0-kghmI

https://www.youtube.com/watch?v=A1mbgDXag7c **References:**

https://en.wikipedia.org/wiki/X-ray_crystallography

http://serc.carleton.edu/research_education/geochemsheets/techniques/XRD.html

http://ocw.mit.edu/courses/materials-science-and-engineering/3-091sc-introduction-to-solid-state- chemistry-fall-2010/crystalline-materials/18-x-ray-diffraction-techniques/

https://saylordotorg.github.io/text_general-chemistry-principles-patterns-and-applications-v1.0/s16-03- structures-of-simple-binary-co.html