TCR Advanced Engineering P. Ltd

Experts in Metallography, Failure Investigation and RLA

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Energy dispersive X-ray Spectroscopy (EDS):

Principle, Advantages and Applications.

What is EDS technique?

Energy Dispersive X-Ray Analysis (EDX), also known as EDS or EDAX, is an x-ray spectroscopy technique used for the elemental analysis or chemical characterization of a sample. This technique can be used for qualitative, semi-quantitative, quantitative analysis of solid samples. It can find the chemical composition of materials down to a spot size of a few microns, and can also provide spatial distribution of elements through elemental mapping. All elements from atomic number 4 (Be) to 92 (U) can be detected in principle by EDS, though not all instruments are equipped for 'light' elements (i.e. those having atomic number < 10).

EDS is a microanalysis technique used in conjunction with scanning electron microscopy (SEM) or Transmission Electron Microscopy (TEM). The data generated by EDS analysis consists of spectra showing peaks corresponding to the elements present in the sample being analysed.

EDS is a quick, versatile, relatively inexpensive and a widely available technique for elemental analysis. The detection limits are typically about 1000 ppm (by weight) but can be reduced by using longer counting times. For major elements, it is usually possible to obtain a statistical precision of ±1%.

What is the underlying principle?

EDS relies on the investigation of a sample through interactions between electromagnetic radiation such as electrons and the matter, analysing the emitted X-rays by the matter in response to being hit with electrons. It is based on the fact that each element has a unique atomic structure that produces a unique set of X-ray peaks on the electromagnetic spectrum to be identified uniquely from each other.

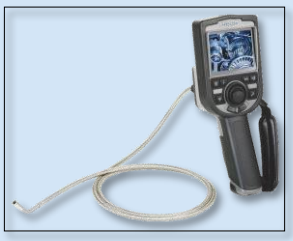
How does an EDS work?

A high energy beam of electrons in a SEM is bombarded onto the sample being studied. When at rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells

TCR News

- It is matter of pride for TCR for being associated in for providing material testing services, viz. Destructive and Non destructive testing for Statue Of Unity, the tallest statue of the world located at Kevadia, Gujarat.

- An additional videoscope machine is included in the existing fleet of the NDT equipment at TCR. This machine has 3 meter long flexible probe with 7 mm probe dia. and 360 degree articulation.



EVOLVE by TCR

- Launched new website www.evolvetcr.com This website features facilities at Evolve, highlights proposed courses, workshops and training programs along with the details of faculty.
- Conducted a "Train the Trainers" workshop at Evolve for improving communication and presentation skills of key persons.

(such as K, L, M, N and O shells) bound to the nucleus. The incident electron beam excites an electron in an inner shell (such as K or L shell), and ejects it from its normal location and thereby creating an electron hole. In turn, an electron from an outer, higher-energy shell (such as M or N shell) jumps and fills this hole leading to release of x-rays corresponding to the difference in energy between the higher-energy shell and the lower energy shell. The number and energy of the X-rays emitted from a specimen is measured by an energy dispersive spectrometer. As the energy of the X-rays are characteristic of the difference in energy between the two shells, and of the atomic structure of the element from which they were emitted, this allows the elemental composition of the specimen to be measured.

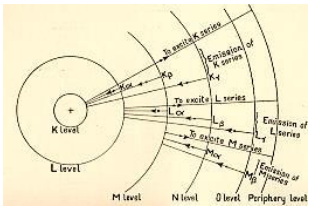


Fig 1.: Showing X_Ray emission energy level's nomenclature.

EDS detector:

The EDS system consists of a sensitive x-ray detector, a liquid nitrogen dewar for cooling, and software to collect and analyze energy spectra. The detector is mounted in the sample chamber of the SEM at the end of a long arm, which is itself cooled by liquid nitrogen.

Traditionally, the EDS detector is a lithium-drifted silicon, solid-state device made of Si(Li) crystals that operate at low voltages to improve sensitivity. Modern machines use "silicon drift detectors" that that operate at higher count rates without liquid nitrogen cooling.

When an incident X-ray strikes the detector, it creates a charge pulse that is proportional to the energy of the X-ray. The charge pulse is converted to a voltage pulse (which remains proportional to the X-ray energy) by a charge-sensitive pre-amplifer. The signal is then sent to a multichannel analyzer where the pulses are sorted by voltage. The energy, as determined from the voltage measurement, for each incident X-ray is sent to a computer for display and further data evaluation. The spectrum of X-ray energy versus counts is evaluated to determine the elemental composition of the sampled volume.

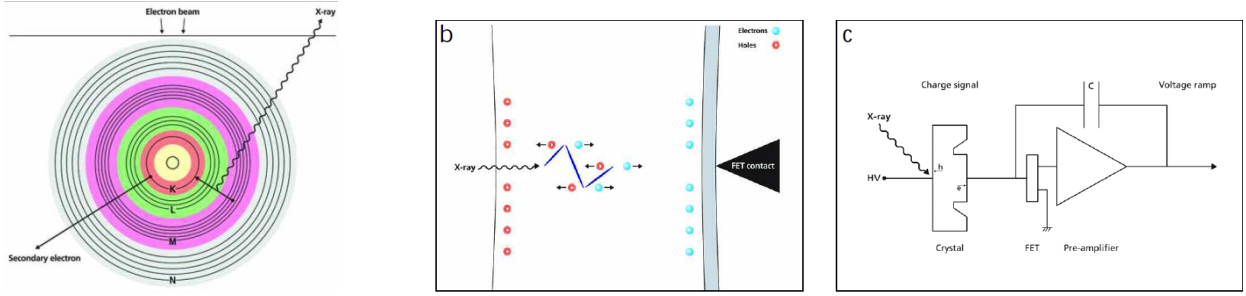
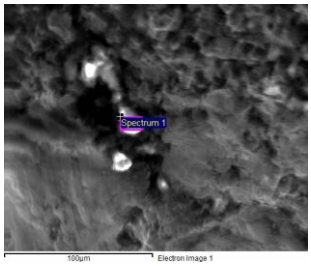
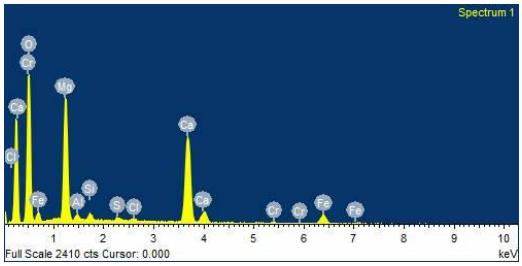


Fig 2.: Schematic showing basic principle of EDS

A typical ED spectrum consists of x-axis representing X-ray energy (usually in channels 10 or 20 eV wide) and the Y-axis representing the number of counts per channel. It is essentially a histogram of the number of X-rays measured at each energy.





Element	Weight%	Atomic%
O K	40.85	58.11
Mg K	25.72	24.08
Al K	1.17	0.99
Si K	1.48	1.20
S K	0.74	0.52
Cl K	0.58	0.37
Ca K	16.91	9.60
Cr K	0.60	0.26
Fe K	11.94	4.87

Fig. 3 : Typical EDX spectrum showing the number of counts on y-axis and the energy of the X-rays along the x-axis. The position of the peaks in the spectrum gives the identification of the elements and the peak height gives the concentration of each element in the sample.

Sample requirement:

The sample should preferably be well polished. In principle, sample having any size and shape can be analysed, provided it can be easily accommodated in the SEM sample chamber. Sample holders are commonly provided for 25mm (1") diameter round specimens and for rectangular glass slides. Samples must also be compatible with a moderate vacuum atmosphere (pressures of 2 Torr or less).

Qualitative analysis using EDS:

The object of qualitative analysis is to find which elements are present in an 'unknown' sample by identifying the lines in the X-ray spectrum using tables of energies or wavelengths. Qualitative analysis involves the identification of the lines in the spectrum and is fairly straightforward. This is ensured by comparing the X-ray energy values from the EDS spectrum of sample with known characteristic X-ray energy values.

Quantitative analysis using EDS:

Quantitative analysis (determination of the concentrations of the elements present) involves measuring the line (X-ray) intensities for each element in the sample and for the same elements in calibration Standards of known composition. X-ray intensities are measured by counting pulses generated in the detector by X-ray photons, which are emitted by the sample.

Quantitative results can be obtained from the relative x-ray counts at the characteristic energy levels for the sample constituents. Semi-quantitative results are readily available without standards by using mathematical corrections based on the analysis parameters and the sample composition.

Elemental Mapping:

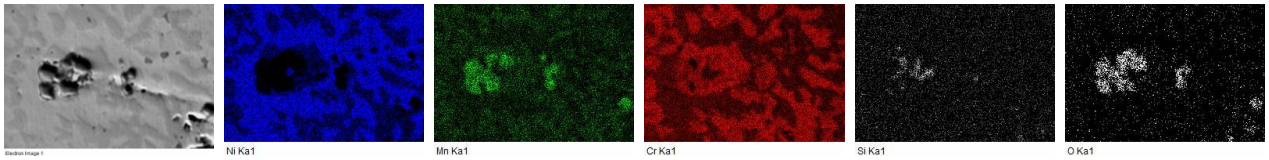


Fig. 4: SEM image and elemental maps (Nickel,Manganese, Chromium and Silicon and Oxygen) of micro constituents in metal

Here the characteristic X-ray intensity is measured relative to lateral position on the sample. Variations in X-ray intensity at any characteristic energy value indicate the relative concentration for the applicable element across the surface. One or more maps can be recorded simultaneously using image brightness intensity as a function of the local relative concentration of the element(s) present.

Line Profile Analysis:

The SEM electron beam is scanned along a preselected line across the sample while X-rays are detected for discrete positions along the line. Analysis of the X-ray energy spectrum at each position provides plots of the relative elemental concentration for each element versus position along the line.

Typical applications of EDS:

- Qualitative material identification
- Microscopic phase identification
- Identification of inclusion content
- Identification of localized inhomogeneity.
- Contaminants identification and analysis
- Industrial forensic science and failure investigation
- Corrosion evaluation and identification of localized corrosion
- Elemental diffusion profiles
- Evaluation of thin coatings

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