ENERGY DISPERSIVE X-RAY SPECTROSCOPY

DESCRIPTION OF TECHNIQUE

Energy Dispersive X-Ray Spectroscopy (EDS or EDX) is a chemical microanalysis technique used in conjunction with scanning electron microscopy (SEM). (See Handbook section on SEM.) The EDS technique detects x-rays emitted from the sample during bombardment by an electron beam to characterize the elemental composition of the analyzed volume. Features or phases as small as 1 µm or less can be analyzed.

When the sample is bombarded by the SEM's electron beam, electrons are ejected from the atoms comprising the sample's surface. The resulting electron vacancies are filled by electrons from a higher state, and an x-ray is emitted to balance the energy difference between the two electrons' states. The x-ray energy is characteristic of the element from which it was emitted.

The EDS x-ray detector measures the relative abundance of emitted x-rays versus their energy. The detector is typically a lithium-drifted silicon, solid-state device. 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 preamplifier. 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.

ANALYTICAL INFORMATION

Qualitative Analysis - The sample x-ray energy values from the EDS spectrum are compared with known characteristic x-ray energy values to determine the presence of an element in the sample. Elements with atomic numbers ranging from that of beryllium to uranium can be detected. The minimum detection limits vary from approximately 0.1 to a few atom percent, depending on the element and the sample matrix.

Quantitative Analysis - 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. The accuracy of standardless analysis depends on the sample composition. Greater accuracy is obtained using known standards with similar structure and composition to that of the unknown sample.

**Elemental Mapping** - 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 are recorded simultaneously using image brightness intensity as a function of the local relative concentration of the element(s) present. About 1 µm lateral resolution is possible.

**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

- Foreign material analysis
- Corrosion evaluation
- Coating composition analysis
- Rapid material alloy identification
- Small component material analysis
- Phase identification and distribution

### Sample Requirements

Samples up to 8 in. (200 mm) in diameter can be readily analyzed in the SEM. Larger samples, up to approximately 12 in. (300 mm) in diameter, can be loaded with limited stage movement. A maximum sample height of approximately 2 in. (50 mm) can be accommodated. Samples must also be compatible with a moderate vacuum atmosphere (pressures of 2 Torr or less).