EDS system

- Most common X-Ray measurement system in the SEM lab.
- Major elements (10 wt% or greater) identified in ~10 secs.
- Minor elements identifiable in ~100 secs.
- Rapid qualitative and accurate quantitative analysis achievable.
- Relatively poor spectral resolution – 150eV at Mn Kα.
- Spectral interferences – peak overlap (eg., S Kα, Mo Lα and Pb Mα).
- Poor limit of detection ~0.5 wt%.
- Several manufacturers
  - CRF Oxford Instruments – INCA
  - CRF EDAX – Genesis
  - EVEX- NanoAnalysis
    Table top system
Elemental Analysis
Energy Dispersive Spectroscopy

• Mechanism of X-Ray formation and detection
  • Detectors
    Si(Li) – Silicon(lithium drifted) solid state detector
    SDD – Silicon Drift Detector

• EDS Signal Processing

• Wavelength Dispersive Spectroscopy
  • Detectors
    Multiple crystals

• WDS Signal Processing

• X-Ray Fluorescence

• Electron Backscatter Diffraction (Orientation Imaging Microscopy)™
X-Ray production

Two distinct formation processes during inelastic scattering.

Bremsstrahlung – X-Ray continuum – “braking radiation”.

- Formed by deceleration of the primary incident beam in the Coulombic field of the atom core (nucleus and tightly bound electrons).
- Energy range – Zero to incident electron energy.
X-Ray production

Characteristic X-Rays

- Incoming primary beam electron interacts with inner-shell electron.
- Bound electron is ejected leaving the atom in an excited state.
- Deexcitation of atom - outer shell electron fills vacancy.
- X-Ray or Auger electron is produced during transition.
- Characteristic energy of X-Ray is related to the energy difference between the two orbitals.
- EDS uses electron energy (eV).
- WDS uses electron wavelength ($\lambda$).
Quantitative EDS – Estimated by Calculations

• On board calculations written into the EDS software

• Intensity ratios - Standard vs Sample
  – Geometric Effects
    • Polished Bulk samples - ideal
    • Particles and Rough Surfaces
    • Thin Films and Foils

• ZAF corrections of Intensity ratios – Most Common
  – $Z =$ Atomic Number Factor
  – $A =$ Absorption Factor
  – $F =$ Fluorescence Correction
Find Best Conditions – Solvability Tool

- Why is accelerating voltage so important
- Controls depth of interaction between the electron beam and the sample
- Need to optimise generation and emission of X-rays from elements in all layers to be measured

Material: Fe
An EDS system is comprised of three basic components that must be designed to work together to achieve optimum results.

- **X-ray Detector**
  Detects and converts X-rays into electronic signals

- **Pulse Processor**
  Measures the electronic signals to determine the energy of each X-ray detected

- **Analyzer**
  Displays and interprets the X-ray data
Typical EDS spectrum
EDS detectors
Si(Li) – Silicon (Lithium Drifted)

- The **window** provides a barrier to maintain vacuum within the detector whilst being as transparent as possible to low energy X-rays.
- There are two main types of window materials.
  - Beryllium (Be) is highly robust, but strongly absorbs low energy X-rays meaning that only elements from sodium (Na) can be detected.
  - Polymer-based thin windows can be made much thinner than Be windows and therefore are transparent to much lower energy X-rays, many allowing detection of X-rays down to 100eV.

![Diagram showing the basic components making up the detector.](Image)

- The **Si(Li) crystal** is a semiconductor device that through the process of ionization converts an X-ray of particular energy into electric charge of proportional size. To achieve this a charge-free region within the device is created.
- Two main materials are used for the detecting crystal.
  - The most common is silicon (Si), into which is drifted lithium (Li) to compensate for small levels of impurity.
  - High purity germanium crystals (HpGe) are also used.
  - Si(Li) was the first material used in EDS detectors and remains the most common choice today.
  - HpGe offers performance advantages when measuring higher energy X-rays.
The field effect transistor, normally referred to as the FET, is positioned just behind the detecting crystal. It is the first stage of the amplification process that measures the charge liberated in the crystal by an incident X-ray and converts it to a voltage output.
Accuracy of EDS

- The accuracy of EDS spectrum can be affected by many factors. Beryllium windows in front of the SiLi detector can absorb low-energy X-rays (i.e. EDS detectors cannot detect presence of elements with atomic number less than 5, meaning that EDS cannot detect H, He, Li, or Be).

- Differing the over-voltage of the EDS will result in different peak sizes – Raising over-voltage on the SEM will shift the spectrum to the larger energies making higher-energy peaks larger while making lower energy peaks smaller. Also many elements will have overlapping peaks (e.g., Ti Kβ and V Kα, Mn Kβ and Fe Kα).

- The accuracy of the spectrum can also be affected by the nature of the sample. X-rays can be generated by any atom in the sample that is sufficiently excited by the incoming beam. These X-rays are emitted in any direction, and so they may not all escape the sample. (Absorption)

- The likelihood of an X-ray escaping the specimen, and thus being available to detect and measure, depends on the energy of the X-ray and the amount and density of material it has to pass through. This can result in reduced accuracy in non-homogeneous and rough samples. (Geometric effect)

(Source: Wikipedia)
How the EDS Detector Works

- The EDS detector converts the energy of each individual X-ray into a voltage signal of proportional size. This is achieved through a three stage process.
  - First - the X-ray is converted into a charge by the ionization of atoms in the semiconductor crystal.
  - Second - this charge is converted into the voltage signal by the FET preamplifier.
  - Finally - the voltage signal is input into the pulse processor for measurement.
  - The output from the preamplifier is a voltage ‘ramp’ where each X-ray appears as a voltage step on the ramp.
  - EDS detectors are designed to convert the X-ray energy into the voltage signal as accurately as possible. At the same time electronic noise must be minimized to allow detection of the lowest X-ray energies.
EDS detectors

SDD – Silicon Drift Detector – newer design

**Silicon drift detectors (SDDs)** are X-ray radiation detectors used in x-ray spectrometry (EDXRF) and electron microscopy (EDX). Their chief characteristics compared with other X-ray detectors are:

- high count rates
- comparatively high energy resolution (e.g. 140 eV for Mn K wavelength)
- Peltier cooling (no liquid N2 required)

**Working principle**

- Like other solid state X-ray detectors, silicon drift detectors measure the energy of an incoming photon by the amount of ionization it produces in the detector material.
- In the SDD, this material is high purity silicon with a very low leakage current.
- The high purity allows for the use of Peltier cooling instead of the traditional liquid nitrogen.

- In older detector designs, the collection electrode is centrally located with an external FET (field effect transistor) to convert the current into a voltage and thus represents the first stage of amplification.

- Newer designs integrate the FET directly into the chip, which greatly improves energy resolution and throughput. This is due to the reduction of capacitance between anode and FET, which reduces electronic noise.

(Source: Wikipedia)
• The major distinguishing feature of an SDD is the transversal field generated by a series of ring electrodes that causes charge carriers to 'drift' to a small collection electrode. (Anode)

• The 'drift' concept of the SDD (which was imported from particle physics) allows for significantly higher count rates.
Data within INCA Energy and Energy TEM is archived in a logical and easily accessible manner and can be directly viewed in the Data tree.

To access the Data tree, press the Data tab to the right of the Navigator tab.
This step in both Analyzer and Point and ID allows you to compare spectra acquired from different Sites of Interest and Samples from any of the Projects currently open.
Very high count rate maps

Texture analysis of fracture surface of a granite sample. This map, taken at a count rate of 400,000 cps with an X-Max 80mm$^2$ detector, shows clearly how it is possible to map the different chemical compositions of a rough surface to see how the textured surface and chemical composition of the surface relate to each other.

Source: Oxford Instruments
The EDS detector converts the energy of a single X-ray photon into a step of proportional size on a voltage ramp using a semiconductor crystal and FET-preamplifier.

Good analytical performance requires good resolution at low energies.

- Mn resolution (5.9keV) does not give a reliable indication of resolution at low energies because it is insensitive to ICC and does not separate noise and detector material components.
- F resolution (0.7keV) is more sensitive to low energy noise and is a better guide to low energy performance
- C resolution accounts for noise and is very sensitive to incomplete charge collection (ICC).

An excellent resolution at carbon implies good resolution at all energies.

The collection efficiency of X-rays is determined by the collection solid angle, not the area of the crystal.
Calculation of the solid angle of an EDS detector.

(a) The detector size and the geometry of the microscope control the distance between X-ray source and crystal.

(b) The solid angle is proportional to the active area of the crystal, rather than its actual area, and inversely proportional to the square of the distance.
WDS system

WDS differs from EDS in that it uses the diffraction patterns created by light-matter interaction as its raw data. WDS has a much finer spectral resolution than EDS.

WDS also avoids the problems associated with artifacts in EDS (false peaks, noise from the amplifiers and microphonics). **Microphonics** describes the phenomenon where certain components in electronic devices transform mechanical vibrations into an undesired electrical signal (noise).

In WDS only one element can be analyzed at a time, while EDS gathers a spectrum of all elements, within limits, of a sample.

- First commercialized for SEM in the 1970s
- High energy resolution – 5-10 eV
- Better detection limit ~0.1 wt%

Figure 15-12(A) Principle of operation of wavelength dispersive x-ray (WDX) spectrometer. X-rays generated by the specimen are “reflected” by the crystalline lattice into the detector. Very specific wavelengths of x-ray are reflected by different lattice spacings so different crystals are needed in the spectrometer to cover various energies of x-rays.

Source: Electron Microscopy, 1992, Bozzola and Russell
Oxford INCA Wave – WDS detector cutaway

- Detector
- Diffracting Crystal
- Signal from SEM
How is INCA\textsuperscript{W}ave different from other WDS spectrometers available for the SEM?

- **INCA\textsuperscript{W}ave** is a fully focusing spectrometer with a geometry similar to EDS. As long as the sample is in focus for EDS analysis, it’s in focus for WDS collection. 500um height variation - OK

- It uses the crystal to focus the beam into the detectors, thus it has no optics and is far less sensitive to sample height changes ensuring accurate quantitative analysis at all times.

- In comparison - **Parallel beam detectors** use X-ray channeling optics, to focus the X-ray signal into the detector. As a result they are very sensitive to any tiny changes in the sample position & the height of the sample.

- This means analysis is 100x more sensitive to sample position than EDS.

- If the sample is incorrectly focused, no WDS signal may be achieved at all and positioning the sample accurately becomes difficult and time-consuming.
WDS X-Ray detection - Oxford INCA Wave

Mapping
Accurate Maps Immediately
Easily and unambiguously determine the distribution of all elements in a sample even where peaks overlap (figure 4)

Figure 4  X-ray maps from a sample containing titanium and barium-rich phases. The Ti/Ba X-ray lines are overlapped when using EDS but resolved when using higher resolution WDS. Therefore the EDS maps incorrectly show phases containing both titanium and barium while the WD maps clearly distinguish between the titanium and barium-rich phases

Source: Oxford Instruments
X-Ray induced Fluorescence

- **X-ray fluorescence (XRF)** is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by bombarding with high-energy X-rays or gamma rays.
- The phenomenon is widely used for *[elemental analysis](https://en.wikipedia.org/wiki/Elemental_analysis)* and *[chemical analysis](https://en.wikipedia.org/wiki/Chemical_analysis)*, particularly in the investigation of *metals, glass, ceramics* and building materials, and for research in *geochemistry, forensic science* and *archaeology*.
- The technique is essentially EDS using an external X-Ray source directed at the sample, instead of an electron beam.
Additional Information sources

- **MICROANALYST.NET** - http://microanalyst.mikroanalytik.de/index_e.phtml
  - Information portal with X-ray microanalysis and EDX contents


- http://www.thermo.com/com/cda/landingpage/0,10255,921,00.html
Basics of EBSD

Electron Backscattered Diffraction

70 degree tilt from horiz
Basics of EBSD
Electron Backscattered Diffraction

• Crystallographic Information
  SEM Generated Diffraction Pattern (interference pattern)
  Kikuchi patterns
  Braggs Law:

Crystal Structure and Orientation  (Inverse Pole Figure)

Grain Boundary Misorientations
Electron Back Scattered Diffraction - EBSD or OIM™

Figure 1: Basic geometry and data of the EBSD experiment. A) Chamberscope image of the internal geometry of the SEM during an EBSD experiment. B) Raw EBSD pattern from silver metal. C) Overlay of indexed solution to the EBSD pattern in B.  

Source: Microscopy Today, 2010 March
Figure 2: High-quality EBSD inverse pole figure map of a magnetic alloy (raw data). There were 585,000 patterns collected for this image with 95.7% successfully indexed. Note that many of the unindexed pixels were at grain boundaries or at second phase inclusions (black). The color key for the image (stereographic triangle) is shown at right.

Source: Microscopy Today 2010 March
EBSD Spatial Resolution

- Interaction Volume
  - Spot Size
    - Current generally increases with increasing spot size
  - Current
    - ~5nA (1-10nA)
  - Voltage
    - Seems to effect depth more than lateral resolution
    - 5-30 kV
  - Material
    - Higher Z materials are better reflectors
  - Operating Conditions
    - Drift vs. Speed

“Average” Pattern
EBSD Applications

The different applications which are presented on this site have been grouped into the generalised fields of Metallurgy, Geology, Electronics and Ceramics together with a general section.

The presented range of applications is diverse and hopefully interesting. Some are clear problem solving examples, others involve more routine material characterisation and some are simply investigative analysis. Yet the power of the EBSD technique and the strengths of the HKL EBSD system are clear for everyone to see.

Metallurgy • Electronics & Ceramics • Geology • General

- EBSD analysis of TiAl alloys.pdf
- Cold rolling of magnesium alloys.pdf
- Preventing cracking of nuclear fuel cladding.pdf
- Phase analysis of duplex stainless steel.pdf
- Determining fcc-bcc orientation relationship.pdf
- Carbide and nitride precipitates in ferritic steel.pdf
- Friction stir welded aluminium.pdf
- Strain variations around crack tips.pdf
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