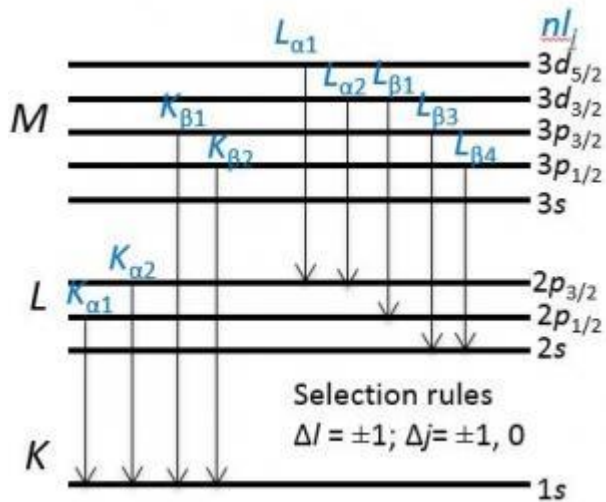


## EDS Principle



Energy Dispersive X-ray Spectroscopy (EDS or EDX) is a qualitative and quantitative X-ray microanalytical technique that provides information on the chemical composition of a sample for elements with atomic number ( $Z$ )  $> 3$ .

### Characteristic X-ray Generation

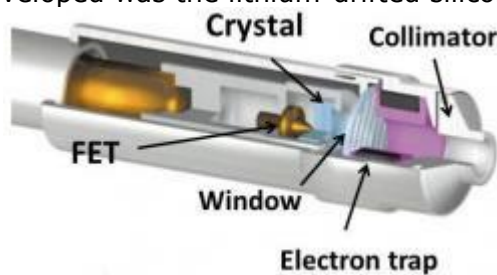
The atoms are ionized by the primary electron beam leading to holes generated on the core shells; following ionization the electrons from outer shells fill the holes and cause the emission of X-ray fluorescence lines.

The characteristic X-ray lines are named according to the shell in which the initial vacancy occurs and the shell from which an electron drops to fill that vacancy.

For instance, if the initial vacancy occurs in the K shell and the vacancy filling electron drops from the adjacent (L) shell, a  $K_{\alpha}$  x-ray is emitted. If the electron drops from the M shell (two shells away), the emitted x-ray is a  $K_{\beta}$  x-ray. Similarly, if an L-shell electron is ejected and an electron from the M-shell fills the vacancy,  $L_{\alpha}$  radiation will be emitted.

### EDS Detector

The detector is based on a semiconductor device, usually a crystal of silicon. The first detector developed was the lithium-drifted silicon or Si(Li) detector, which is now giving



way to the

Electron trap

silicon-drift detector or SDD.

A typical EDS detector is composed of

- A collimator to ensure that only X-rays generated from where the primary electron beam interacts with the sample will be collected.
- An electron trap to ensure that X-rays, but no electrons, enter the detector.
- A window to isolate the detector crystal, under high vacuum, from the chamber of the microscope. Older windows were composed of Be which did not allow low-energy X-rays ( $< \sim 0.9$  keV) to pass through it, but more modern windows are composed of polymers that will allow low-energy X-rays (down to  $\sim 0.1$  keV) to pass.

- A semiconductor crystal detector.
- Electronics to detect the charge recorded by the detector, convert it to a voltage pulse and pass it to the pulse processor.

#### Detector Operating Principle

- The energy of the incoming X-ray is dissipated by the creation of a series of electron-hole pairs in the semiconductor crystal.
- A high bias voltage is applied across the crystal and this causes electrons and holes to move to electrodes on opposite sides of the crystal, producing a charge signal which is passed to the pulse processor.
- The size of the signal is proportional to the energy of the incoming X-ray. For a silicon detector,  $\sim 3.8$  eV is used to generate each electron-hole pair ( $\sim 2.9$  eV for Ge). So for an incoming Ni K $\alpha$  X-ray of energy 7.477 keV, 1968 electron-hole pairs will be produced, and for an Al K $\alpha$  X-ray of 1.487 keV, 391 electron-hole pairs will be generated.
- By measuring the amount of current produced by each X-ray photon, the original energy of the X-ray can be calculated. An EDS spectrum is essentially a histogram of the number of X-rays measured at each energy.

To minimize electronic noise, the detector must be cooled. Si(Li) detectors are cooled to liquid nitrogen temperatures and are attached to dewars that require regular filling. Due to the greatly reduced anode noise contribution, the SDD can operate at  $-25^{\circ}\text{C}$  which can be obtained readily with Peltier cooling.



### Oxford EDS

#### 1. In SEM system:

- (a) Select the desired Accelerating Voltage: for EDS, the recommended accelerating voltage is 1.5 to 2 times the highest energy line in the spectrum. For example: the Cu K line appears at 8.04keV, so the proper accelerating voltage for a sample in which copper is the highest energy line would be 12 – 16kV. For unknown sample, 20kV is a good starting point, which will excite most X-ray lines from *most elements for analysis*.
- (b) Choose the Objective Aperture 3 for EDS.
- (c) Set the working distance (WD) to 10 mm.
- (d) Get a high quality sample image by fine alignments and adjustments.
- (e) **Make sure the IR camera is turned off** by minimizing or closing the IR camera window before collecting any spectra. Otherwise the deadtime will 100% during spectrum collection.
- (f) When collecting EDS spectrum, adjust the Spotsize so that the deadtime is about 30-40%.

2. Log in to EDS computer.
3. Open the INCA software by double-clicking the **[INCA]** icon on the desktop.
4. There are three analysis options for the program:
  - (a) **[Analyzer]** – allows for EDS acquisition and quantitative analysis but no imaging acquisition.
  - (b) **[Point & ID]** – allows image acquisition and site specific EDS analysis.
  - (c) **[Mapping]** – allows for EDS mappings and linescans using a data mining approach (i.e. a full spectrum is stored from each pixel)
5. Within each project file you may have multiple samples. For each sample, you may have multiple sites of interest, and each site of interest is an image with various associated spectra.
6. Following are basic instructions, but more detailed information can be readily obtained from the **[Show bubble]** help icon (upper right corner of the screen).
7. Image capture:
  - (a) Get high quality SEM image and select [Scan 2].
  - (b) Click the **[Image Setup]** → select the desired Image Resolution (512×384), Speed (medium) and Data (8 bit), and input the number of Frames (1). In the parentheses are recommended normal settings.
  - (c) Click the **[Site of Interest]** menu and click on the green button to acquire an image.
8. EDS acquisition:
  - (a) Click **[Acquisition Setup]** → set the desired Livetime, Process time (1-6, 5 and 6 for high resolution, 3 or 4 for normal spectra and mapping), Spectrum range (determined by the accelerating voltage), and Number of channels. A lower process time allows for faster collection with higher acquisition rates at the expense of energy resolution (i.e. wider peaks).
  - (b) Click **[Acquire Spectra]** → select region(s) of the sample to analyze. In Point & ID you can use the point, box, etc. tools, but in Analyzer mode there is no scan control so the system collects X-rays from the whole area.
  - (c) Click **[Confirm Elements]** to verify the peaks.
9. Quantitative analysis:
  - (a) Click **[Quant setup]** → set Processing Option (All elements), check Normalize Quantitative Results, Set Element List (Current spectrum).
  - (b) Click **[Quant]** to get the quantitative results, choosing different tabs to select different display methods.
10. EDS mapping:
  - (a) In the **[Mapping]** option, collect an image as per the image capture instructions

above.

- (b) Click [**SmartMap Setup**] → set Map or Linescan Resolution (256×192); Conditions: Process time(3), Spectrum range (0-20 keV), and Number of channels (2k); and Acquisition Time: choose fixed duration, Frame (20), map dwell (100µs), linescan dwell (2000µs).
  - (c) Click [**Element Setup**] to select the elements for mapping or linescan.
  - (d) Click [**SmartMap**] → select a region to map or draw a line to scan. Click the green button to begin acquisition.
  - (e) Click [**Element Maps**] or [**Element Linescans**] to show the mapping or linescan.
11. Save the project file to D:\data\. You can create your own folder **only** under this directory. To save any images, maps or EDS spectra, right click on them and select Export. For image format, please choose JPG for smaller file size. And EDS spectra can be exported as EMSA file (a text file) which can be plotted using software such as Excel or Origin. The linescan and mapping data can be exported as ".TSV" file (right click and select "Export Data Only", then select ".TSV" format) to be plotted using other software.