1. Signal Generation/Detection
The backscatter detector is a *solid state* detector:

- Incoming radiation is incident on a semi-conductor; this moves electrons around, which are collected and processed to form a digital signal

When the chamber camera is active, IR LED’s provide illumination

- The signal from these LED’s would interfere with the BSD detection, so the system automatically disables one when the other is enabled
- The chamber camera is crucial for protecting the hardware when driving the stage around – be aware that the signal hasn’t been paused prior!
The generation of an SE from an atom within the sample leaves a vacancy in its electron orbital. Electrons from higher orbitals will fill this vacancy as it is energetically favorable. As the electron jumps to a lower energy state, it emits the excess energy in the form of an x-ray photon. The energy of this photon is equal to the difference in energy between the initial and final electron shells. The energies associated with these transitions are unique and well tabulated.
Multiple different transitions (corresponding to multiple different X-ray “lines”) are possible
- **K-lines** represent transitions to the 1\textsuperscript{st} shell, L-lines are those to the 2\textsuperscript{nd}, etc
- \(\alpha\)-transitions represent those that occur between neighboring shells (2\(\rightarrow\)1, 3\(\rightarrow\)2, etc), \(\beta\)-transitions are those that are from two away (3\(\rightarrow\)1, 4\(\rightarrow\)2, etc), etc
- Example: an L\(\beta\) line represents the transition from the 4\textsuperscript{th} to the 2\textsuperscript{nd} shell
X-ray Detection

- X-rays are incident upon a collection electrode and channeled to the detector, where processing tabulates the data as number of counts per unit energy.
- Previous systems (Si-Li, left) suffered from count rate limitations.
  - Above 20,000 cps, the detector would flood, resulting in unreliable data.
- Newer designs (SDD, right) improve upon this.
  - “Drift rings” in collector channel X-ray signals at varying rates, allowing for greater incident counts.
  - >100,000 cps is routine, >1 Mcps is possible.
Like the BSD, the X-ray detector is also solid state
  - As an external detector, the camera will NOT be automatically disabled
  - If on, the large number of extra counts will greatly reduce energy resolution
  - Worse, the processor will assign the low energy IR signal to the ‘strobe peak’, a synthetic reference peak generated in the software at 0 kV
    - Results in incorrect assignments!
Resolution vs Counts

- Imaging resolution generally achieved by reducing counts
- High count rates, required for EDS statistics, comes with reduced resolution
2. ESPRIT Operation/Controls
ESPRIT Workspaces

- Each type of analysis has its own “workspace”
- CW from top left:
  - Spectra
  - Object
  - Line
  - Hypermap
- Additional non-EDS workspaces exist as well (e.g. particle analysis)
EDS Analyses: Spectrum Collection

- Collects a spectrum across the entire imaged area
- No inherent spatial information included
- FAST: ~20s / scan (qual.)
- Requires uniform chemical distribution
EDS Analyses: Object Analysis

- Rather than scanning the entire region, the beam will raster in user defined regions.
- Can characterize distinct phases separately.
- Multiple spectra collected via designated parameters.
- Still quick: small regions don’t take less time than full area!
  - Still rastering pixels at the same rate.
  - Caveat: denser regions more readily generate X-rays.

EDS Analyses: Line Scan

- Least utilized
- Collects spectra across user-defined line
  - Good for analyzing phase interfaces and non-discrete boundaries
- Entirely qualitative – “conc.’s” generated relative to peaks, not any calculated quantities
- New EDS detector (for F30) would include Quant. upgrade
- Profiles can be scaled relative to highest overall feature (‘universal’) or highest feature for that element along line (‘local’)
- ~5 min/scan
EDS Analyses: Hypermaps

- Far and away the most versatile – collects a spectra at every pixel in a defined region
  - Because the data exists in the full volume, all previous analyses are available as post processing
- Requires significant investment of time; assuming excellent count rate (>100 kcps)
  - 5-10 min for basic element maps
  - 15-20 min for more advanced
- NOT good for quant!
ESPRIT Workspaces

1. Sample setup
2. Standards setup
3. Microscope
4. Scan
5. Spectrometer
6. Report Manager
7. Project Manager
8. Workflow
9. Workspace I/O
10. Element I/O
11. EDS Workspaces
12. Image Analysis
13. Element Selector
1-3. Sample/Standards/Microscope

- No routine user applications
4. Scan

- Can adjust the quality of SE/BSE image imported into ESPRIT.
- Adjusting resolution is often good for mapping – unnecessarily scanned pixels leads to worse counts (statistics) in pixels of interest.
- If phases are large, user benefits from having lower resolution.

![Scan Configuration](image-url)
5. Spectrometer

- Gives count rate (ICR) and dead time information
- Dead time: percentage of time spent processing vs collecting data
  - Dead time not a bad thing! 40% is ‘ideal’
  - A function of count rate and pulse throughput
- Pulse throughput: X-ray sampling rate
  - Lower throughput: higher dead time (more processing) = less total counts
  - Higher throughput: more counts, but degraded energy resolution
- Maximum energy: defines the width of the energy channels
  - Max energy / 4096 channels
Pulse Throughput

- Below: 2 spectra collected for the same number of counts (10M total)
- The higher throughput required significantly less time (28s vs 80s)
- The lower throughput yielded finer energy resolution, allowing smaller features to be resolved
- **130 kcps is a very good 'general' setting**
- For less refined analysis, the higher count rate allows for faster collections
6-7. Report/Project

- **Report:** Data can be added to an automatically generated running report.

- **Project:** Data can be added to a project file for easy access to later processing. Note: all data in project occupies active memory – be wary about adding hypermaps! (very large)
8. Workflow

- Preview: Collect continually refreshing image/spectra
- Capture: Collect reference image using settings defined in scan
- Acquire: Begin collecting X-ray data
- Quantify: Quantify spectral data

<table>
<thead>
<tr>
<th>Spectra</th>
<th>Object</th>
<th>Line</th>
<th>Hypermap</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1.png" alt="Spectra Image" /></td>
<td><img src="image2.png" alt="Object Image" /></td>
<td><img src="image3.png" alt="Line Image" /></td>
<td><img src="image4.png" alt="Hypermap Image" /></td>
</tr>
</tbody>
</table>
9-10. Input/Outputs

- **Lower I/O buttons** control individual content in attached boxes
- **Upper I/O buttons** control entire workspaces
  - For instance, in an objects scan, the lower buttons save individual spectra or images. The upper button saves the reference image, applied objects, and collected spectra in one file

- **Make sure you’re saving the workspaces upon collection**
  - If it’s a series of spectra/object scans, it’s easy and makes post processing more convenient to add it to a running project file as well
11-12. Analysis/Workspace Selector

- Spectrum
- Object
- Line
- Hypermap
- Image Manipulation
- Particle Analysis

Switching to a new workspace doesn’t affect data in old workspace

Can’t switch workspaces when collecting
Image Manipulation

- Filter: Apply a number of image filters at user-defined levels
- Segmentation: Create false color images using intensity histogram. Ex:
  ![Image Example]
- Panorama: Stitch multiple images together
- Measurements: Make a number of different measurements of imaged features
Specialized CPU-aided image processing utility

- Particles are separated and tabulated via a sequence of filtering and binarization
- Each particle is characterized by a wide range of geometric quantities
- Distributions may be generated and correlated to the image
Variable Z

- EDS requires the detector be pointed directly at the sample
- For longer WD applications (wider field of view), EMC has the Variable Z adapter
  - Physical knob on the base of the unit which pivots the detector downwards
  - With sample in desired location, begin tilting downward – count rate should increase
  - Detector is pointed at sample where count rate is at a maxima
Variable Z

WD = 9mm

WD = 50mm
Spectrum Acquisition Options

- Counts-based (for statistics)
  - Automatic: # of counts in full spectrum
    - Fast (50k counts)
    - Precise (250k counts)
    - Exhaustive (1M counts)
  - User selected number of counts within a user-defined region (such as for a particular element/line)

- Time-based (for consistency)
  - Real (instrument) time
  - Live (collection) time
  - Manual
3. Brief EDS Processing
The heart of EDS analysis is based on the spectra

Spectral analysis available in all collection modes

Common capabilities

- Automatic, CPU aided, and manual peak identification
- Peak deconvolution aids in identification from overlapping features
- Standards-based & standardless quantification
- Many options for easy plot generation and exporting
Peak Identification

- Manual selection of elements
- Auto ID for well-resolved features
- Finder tool: highlight a region, and the software will list lines that fall within that region in order of ‘likelihood’: \(\alpha > \beta > \gamma\), distance from line from the center of the region, etc
Online Peak Deconvolution

Initial assignments appear to successfully account for all features present.
Online Peak Deconvolution

Reconstruction of elemental lines shows that Kβ signals (calculated from associated Ka lines) don't account for all detected counts.
Online Peak Deconvolution

Deconvolution indicates the presence of Mn and V.
Quantification

- Requires considerable planning prior
- Ultimately at the mercy of your statistics; many things can reduce statistics
- In short, region needs to be reasonably flat and chemically homogeneous
- Proper overvoltage is CRUCIAL
  - Voltage too low and lines aren’t properly ionized
  - Voltage too high and statistics decrease (greater background/interatomic affects)
Overvoltage

- Highest line: Ca (3.69 kV) – proper overvoltage ~8kV
- Spectra collected at 8kV and 15kV for 3 minutes each
  - Higher voltage yielded greater count rate (16M total counts vs 9M total counts)
  - Despite this, 8kV spectra gave remarkably better statistics
Individual/Composite Maps

Can generate individual and composite maps for any combination of elements
Elemental Heat Map

BSE Image

Standard Al Map
Maximum Pixel Spectrum

Reference BSE Image

Associated elemental map
Maximum Pixel Spectrum

**Net hypermap spectrum**
Represents the sum of each energy channel’s counts across ALL analyzed pixels – high spatial concentrations dominate

**Maximum pixel spectrum**
Indicates the presence of several additional elements, each of which may only exist in single-digit no. of pixels
Most features comprise significantly less than 0.1 wt% in the bulk, below the accepted detection limit of EDS.