MSE 528 - Scanning Electron Microscopy: Basic SEM Imaging - 1

Purpose:
This laboratory is intended to expose the beginning SEM operator with the steps involved in taking a micrograph.

Equipment:
JEOL JSM-5800 SEM with Thermo System Six (Noran) energy dispersive X-ray analyzer.

Time for this lab session: 2 hrs per group if lab is read in advance.

1. Identifying parts of the SEM

Two main parts of the SEM are the electronic console and the electron column. The console provides the switches and knobs for adjusting the focus, magnification and image intensity on viewing and photography screens. The column is where the beam is generated focused to a small spot and scanned across the specimen to create signals that control intensity of the image on the viewing screen.

Electron Gun - The source of electrons is located at the top of the column where electrons are emitted from a hot tungsten wire and accelerated down an evacuated column. The three gun components are the filament, the wehnelt, which controls the number of electrons leaving the gun, and the anode that accelerates the electrons to a selectable voltage between 1 - 30 kV. A vacuum is necessary because electrons can travel only short distances in air.

Electron Lenses -Two or three electron lenses are used to demagnify the electron beam to a small spot about 1 µm in diameter (100 nm, 1000 Angstroms) from a much larger (1000 times) crossover diameter inside the electron gun. The condenser lens is located closest to the electron gun and the final or objective lens is located closest to the specimen. The objective lens moves the smallest spot formed by the beam up and down in space to meet the specimen surface, which is a focused condition.

Scanning system -The image is formed by pushing the beam across the specimen surface in a regular manner in synchronism with a beam scanning within the cathode ray tube (CRT) on the console. Scan coils, used to push or deflect the beam, are located within the objective.

Experiment 1:
Take images at 500x, 1000x, and 2000x, save these images. Later open the saved files using the **Analysis software** and measure grain size.

Scanning Electron Microscopy: Qualitative X-ray Microanalysis (EDAX) -2

Introduction

The modern energy dispersive X-ray spectrometer (EDS) coupled with a computer based multichannel analyzer (MCA) provides a powerful analytical facility. The purpose of this laboratory is to introduce the student to the analytical capabilities of the EDS/MCA system, to provide practical experience in the acquisition of X-ray spectra and qualitative analysis of those spectra. Procedures for both qualitative and quantitative analysis will be examined.

Equipment

1. SEM with an EDS system. A conventional Be window Si(Li) detector with a 1024 channel MCA set to 10 eV per channel.
2. Picoammeter to measure beam and/or specimen current.

Specimen

Specimens should be metallographically polished (to 0.3 um grit) and unetched.

1. Al, Ni, Cu & Au pure element standards.
2. A 316 stainless steel alloy and sand sample, both of unknown composition.

Time for this lab session

Two hours

Families of X-ray Spectra

The analyst must be familiar with the appearance of typical X-ray spectra. In this section, we will record X-ray spectra for a number of sources and make use of the X-ray data display of the MCA to identify each X-ray peak. The EDS/MCA system is an ideal tool to study spectral energies and relative peak intensities.

Experiment 1: KLM spectra

Using a 20kV electron beam, record the spectra from each of the elemental samples for 60 seconds live time at 15% dead time. Identify all the peaks for the first two elements (Al & Ni) by looking up their energies using the "energy slide rule". Using the KLM markers of the X-ray system, identify the peaks for the last two elements (Zr & Au).

Questions

How can you recognize the K, L and M families of spectra? Do the software markers identify all the peaks that you found? What (if any) is the error in the spectrometer energy calibration? Why are there no Au Kα peaks in the Au spectra?

Qualitative Analysis Guidelines

1. It is dangerous to identify an element based on 1 peak.
2. Always accumulate sufficient counts to distinguish peaks from the continuum background.
3. EDS systems are prone to the appearance of artifacts when the count rate is high.
4. Be wary of peak overlap or peak obscuring.

Methodology

1. Start identifying peaks from the high-energy end of the spectra. Here separation of the peaks within a family is largest and are most easily resolved.
2. Try the K lines first - choose a large peak Kα and try to find the corresponding Kβ peak with a peak height ~10% that of the Kα line. If the Kα line is at the correct energy, the Kβ line should be within +/- 1 channel of the expected value.
3. Try the L-lines. If an L line is found, the entire L family should be there.
4. Once an element has been tentatively identified isolate all the lines (K, L and M) associated with that element.
   - The presence of Kα - Kβ pairs above 6 keV (iron) requires the presence of the L family at the lower end of the spectrum.
   - The existence of L lines above 5 keV (neodymium and above) requires the presence of the corresponding M lines.

X-ray family	**X-ray Intensity**
K	K_a(1),K_b(0.1)
L	L_a(1), L_{b1}(0.7), L_g(0.08), L_{b2}(0.2), L_I(0.04), L_h(0.01)
M	M_a(1), M_b(0.6), M_z(0.06), M_g(0.05), $M_{II}M_{IV}$(0.01)

5. Artifacts - The Si escape peak appears 1.74 keV below the main peak (Kα) due to partial absorption of the emitted X-ray by the Si atoms in the detector. Additionally, sum peaks may occur when large peaks at E_a and Eb, produce small (sum) peaks at $2E_a$, $2E_b$, E_a+E_b.

Qualitative analysis

Qualitative analysis is defined as the identification of the elemental constituents of a specimen by recognition of the characteristic X-ray peaks associated with those elements. With a conventional beryllium window EDS, elements with Z higher than Carbon can be directly observed. With a windowless detector, elements with Z down to boron can be detected. Qualitative analysis requires experience of both the characteristic appearance of X-ray peaks and the artifacts that can arise as part of the detection processes. The analysts must always apply common sense in identifying X-ray peaks and experience is gained by starting with elemental samples, progressing through binary compounds and so on. Experience of the possibility of additional peaks and escape peaks is also necessary - beware of reliance on automated (software) peak identification particularly where the elements identified are naturally rare.

Experiment 2: Qualitative Analysis of a Complex Spectrum

Record a spectrum from the 316 stainless steel and sand sample for a 100 sec live time, 10% dead time. Identify all the peaks, differentiating between major and minor constituents. Store the spectrum. Prepare the following report noting the energies of all peaks;

Specimen Identification:	Beam Energy = kV
Major constituents (> 10 wt. %):	
Minor constituents (1< wt. % < 10):	
Trace constituents (< 1 wt. %):	
Possible interferences:	

Questions

We have frequently used 30 to 60 second live times for spectra acquisition.
Why did we choose a longer live time for this analysis?

Quantitative Analysis

The ratio of X-ray intensity from an element in a sample of unknown composition to that of a standard is the basis of quantitative microanalysis. With proper attention to important user-selected parameters mentioned in this lab, the technique can yield detection limits of 0.1 wt% and errors on the order of ±3%.

The equation that forms the basis of quantitative X-ray microanalysis is the "K ratio".

$$K_{ratio} = \frac{\mathbf{I}_{specimen}}{\mathbf{I}_{standard}}$$

where **I** is the measured characteristic X-ray intensity for that element (corrected for background, peak overlap and dead time). The subscript "**specimen**" denotes the intensity of the element in the specimen and "**standard**" refers to the standard of known composition. The **K ratio** is the ratio of characteristic intensities measured on the specimen and standard. There exist "matrix effects", arising from the nature of the electron and X-ray interactions with matter, which modify the measured intensities and depend on the unknown composition of the specimen. A variety of approaches are used ("ZAF", "phi(rho-z)", "empirical") to calculate correction factors for these matrix effects. This laboratory will examine two methods: fully rigorous ZAF analysis using standards and DTSA's "Standardless Miracle". In addition, the lab will illustrate errors that can arise when user-selected parameters are incorrectly established.

When careful attention is paid to operating conditions and analysis procedures, fully rigorous analysis with standards can yield a relative accuracy of ±3% for most elements. For the EDS case, optimal conditions will be considered to be the following:

1. Element atomic numbers of 11 (sodium) and greater.
2. Concentrations greater than 5%.
3. No significant inter-element peak overlaps.

The precision of the concentration determination depends on the measurement statistics for the characteristic and bremstrahlung X-ray intensities. There are two key assumptions, often assumed as being obvious, which form the foundation of the X-ray microanalysis technique:

Fully Rigorous ZAF Analysis

The most effective strategy for EDS analysis is to record and archive spectra from standards (pure elements, simple compounds such as gallium-phosphide, alloys and glasses) under standardized electron beam and X-ray spectrometer conditions. Provided data collection conditions can be reproduced from day to day, these archived spectra from standards can be used indefinitely

Archived standard spectra may be used to process spectra from the specimen to extract characteristic intensities, automatically correcting for peak overlap and background. These spectra may even be used to calculate K ratios. The ZAF matrix correction method may then be applied to the K ratios to produce the final measured concentrations.

Experiment 3.1: Establishing Proper Working Conditions

The operating conditions for this lab will be 20 kV, 15 mm working distance, 0° tilt. . Choose a beam current that gives a dead time of 10-15% on the alloy. The live time should be set at 100 seconds. Record the absorbed (specimen) current with the picoammeter. Inspect the ZAF setup header to ensure that these parameters are selected. A specimen has been prepared for this lab that contains a Zr-Cu-Ni-AI alloy.

Experiment 3.2: K-ratios

Measure and record spectra for the pure element Cu, Al, and Ni standards at the operating conditions described above. Collect spectra from each of the pure element specimens and the alloy. Establish "windows" over the Kα peaks of both Ni and Al and record the net window intensity. Calculate the k-ratio from the net intensities.