**Scanning Electron Microscopy & Electron Microprobe Analysis**

**I- Basic Principles of Operation**

An electron beam is allowed to interact with the sample to produce a variety of effects which can be used in one way or another to help you identify/ analyze this sample. The interaction usually occurs under vacuum. The sample is always in the solid state, and is either a grain mount, whole sample (size a function of the size of the sample chamber), or thin section. The sample is usually polished (especially when quantitative data are sought), although whole rocks can be imaged to provide a “stereoscopic image”.

**II- Uses of an SEM/ EPMA:**

1. Imaging
2. Qualitative analysis (only if equipped with an “EDS”)
3. Quantitative analysis (only if equipped with an “EDS/WDS”)

**III- Differences between the SEM and the EPMA:**

Both instruments have the same basic principle of operation, and share many components. However, the SEM is optimized for imaging, especially when high resolution images are needed, whereas the EPMA is designed primarily for quantitative analysis. SEM’s are capable of 3-D imaging, and the user can tilt the samples, whereas in microprobes, the sample cannot be tilted. The EPMA therefore has several wavelength dispersive spectrometers which the SEM typically lacks.

**IV- Advantages of SEM and EPMA analysis**

1. Relatively simple and rapid preparation of samples
2. Techniques are non-destructive, samples can be analyzed over and over again.
3. Very fast; a full analysis for 10 major elements takes at most 5 – 6 minutes after standardization.
4. One of the best (if not the best) techniques for obtaining compositional and textural data simultaneously.
5. Imaging achieves resolutions of 100 – 10 nm depending on the type of image used.
6. Analyses are characterized by excellent precision (a fraction of a %) as long as the beam is stable and conditions of operation are optimized. Accuracy is typically ~ 1% for major element analysis.

**V- Geological Applications:**

1. Paleontology: identification of microfossils
2. Sedimentology: identification of porosity; clay minerals; and types of cement; textural interpretations.
3. Mineralogy: Mineral identification; compositional information, microstructures/ deformation.
4. Igneous and metamorphic petrology: Too many to list, but these include: compositional evolution of minerals, geothermometry and geobarometry, textural interpretations, microstructures in minerals and geospeedometry, P-T evolution of rocks and minerals, fluid – rock interaction, fO2 conditions of crystallization, and many more!
5. Geochronology: Recent advances in EPMA has led to the quanititative analysis of trace elements, and hence the ability to analyze for U, Th, and Pb in monazite. This has led to the development of a method of “chemical dating” of this mineral. Results are overall satisfactory, and the technique is quite powerful because it preserves textural relations, and because of its high spatial resolution.

**V- Sample preparation:**

*Polishing:* For quantitative analysis, the sample has to be polished to get rid of pits and cracks. Polishing is done using special laps and either alumina powder or diamond grit. The final grit size is typically 0.25 µm for best results.

*Etching*: Is carried out to convert compositional information to topographical differences, so that they can be recorded by SEI instead of BSEI (see below). This would allow the user to obtain better resolution. Etching is carried out by immersing the sample (usually unpolished) in acid, or suspending it over an acid bath to be attacked by fumes. The acid is selected according to the nature of the sample/ minerals contained (e.g. HCl is used for carbonates, HF for quartz, …. etc.). Etching is very useful for specific studies of surface textures of grains and sediments.

*Coating*: The sample has to be coated with a thin layer of conductive material such as gold or carbon. Carbon coating is preferred when using polished thin sections because it allows the user to carry out transmitted polarized light petrography without having to remove the coat.

**VI- Components of a typical SEM (Figs. 1 & 2):**

1. Electron gun (Fig. 3):

* Filament: W or LaB6 or field emission gun.
* Wehnelt cap: which is typically given a negative charge known as gun bias. The gun bias controls the brightness of the filament, as well as the “first crossover”.
* Anode plate: Controls the beam current intensity.
* Filament saturation: Fig. 4

1. Vacuum pumps: Rough pump: 10-2 torr, and a diffusion pump to 10-5 torr.
2. Condenser Lens: Controls the beam current, and demagnifies the electron image. Consists of 2 sets of coils that cause two more crossovers.
3. Objective aperture: controls the shape of the beam as well as the final beam current on the sample. Smaller apertures produce better resolution but a smaller beam current.
4. Objective lens: A third set of coils that control the spot size and demagnify the image. A defocused beam is obtained by applying a lower current to the objective lens.
5. Secondary electron detector: for SE imaging.
6. Backscattered electron detector: for obtaining BSE images.
7. Beam specimen stage/ sample holder: larger holders or compartments are available for the LV or environmental SEM’s.
8. Energy Dispersive Spectrometer
9. Wavelength dispersive Spectrometers (between 3 – 5 spectrometers set to measure specific wavelengths in microprobes only).
10. CRT and various PC controls.

**VII- Electron beam – Sample Interaction (Fig. 5a & b)**

1. Continuum (Brehmsstrahlung; Fig. 6)
2. Characteristic X-rays (Fig. 6). Note the importance of the fluorescent yield “ω” defined as the total # of ionizations which lead to the emission of X-rays. It is directly proportional to the average atomic number of the sample analyzed.
3. Elastic scattering: Is the phenomenon of large deflection of the electron beam by one of the nuclei of the atoms of the sample analyzed with very little loss of energy. A special type of elastic scattering results when the incident electrons are being deflected through angles > 90° to produce “backscattered electrons”. These high energy electrons are very useful for providing compositional information about the sample, since their production is a function of the average atomic number of the sample analyzed. Figure 7 shows the backscattering coefficient (defined as η = ) as a function of the average atomic number “Z”.
4. Secondary electrons: Are generated within a few nm from the surface (< 70 Å), and are characterized by a relatively low energy (< 50 eV). The number of secondary electrons generated per incident electrons from the original beam is typically 0.1 – 0.2.
5. Auger electrons: Produced from a depth of 1 nm within the sample. They are useful for surface microanalysis if a suitable detector is installed.
6. Cathodoluminescense (CL): Is the emission of long wavelength photons in the UV or visible regions of the spectrum. Their energy is typically a few eV. They are produced through an “electron – hole pair”, i.e. when electrons (in the compound) are raised to a higher energy level for a short while by the incident electron beam or the emerging characteristic X-rays, followed by their rapid return to their original state (hence producing these photons). CL is characteristic of some compounds with specific “activators” (typically a transition element impurity of some sort).

Note that the total beam current is equal to the sum of the backscattered current, the secondary electron current, and the absorbed current (known as sample current).

**VIII- Imaging**

*Optimum conditions for SE imaging:*

1- clean column

2- clean final aperture

3- perfect alignment

4- minimum vibrations

5- no or minimum external magnetic fields

6- small or no tilt (tilt angle < 18°)

7- good coating to prevent sample charging

8- perfect focus, appropriate magnification (avoid hollow magnification):

for 1 beam mag < 1000X, for 10-3 mag < 100,000.

9- optimum working distance: smaller working distance increases the resolution (but decreases the depth of field!)

10- small beam size for increased resolution: Field emission gun and LaB6 are superior in this respect.

11- smallest aperture leads to increased resolution

12- strong beam current increases SE signal, but may cause a larger beam size and therefore decreased resolution!

13- higher voltage (but be careful of damaging the sample!)

*Types of images*

1. *Secondary electron imaging (SEI):* A special type of scintillator detector (see below)is used to collect the secondary electrons, often using a positively biased grid to increase the signal strength (Fig. 8), hence yielding topographic information. Note that the greater the tilt angle of the sample, the lower the number of secondary electrons produced or detected.
2. *Stereoscopic images:* A form of SE imaging, obtained through changing the tilt angle, and taking a picture at each tilt angle. Pairs of pictures with some overlap can constitute a stereoscopic pair.
3. *Compositional information (BSEI):* The brightness of the backscattered image yields valuable compositional information; the brighter the image, the higher the average atomic number. However, the spatial resolution of a BSEI is worse than that of a SEI. To obtain a “combined” image (with some topographic information), two BSE detectors are used on the different sides of the incident beam (Fig. 9). The BSED is typically negatively biased to avoid collecting any secondary electrons.
4. *X-ray dot maps:* Needs an EDS detector, which is set for a particular energy peak for the element of interest. The sample is then scanned, and the concentration of the element in the sample appears as dots on the screen. X-ray dot maps always have a low resolution because X-rays have a weaker intensity!
5. *Absorbed Current Imaging (AEI):* Opposite to the BSEI, sometimes the signal is inverted to make them similar! AEI has the advantage of avoiding shadow effects.
6. *Electron Channeling patterns:* Yields crystallographic data. BSEI at low magnifications and angles close to the Bragg angles. Contrast enhancement becomes a necessity. Useful for determining if all quartz grains in a sample have the same orientation.
7. *Cathodoluminescence Imaging:* Useful if the sample has “activators”.
8. *Scanning Auger Imaging:* Surface analysis with high lateral resolution. Sample coating is a problem.

*The process of Scanning (Fig. 10)*

Two sets of coils: X & Y are used to raster the beam. The current is also varied in intensity over time. Note that the speed of rastering the beam is very important in controlling the quality of the image (slower speeds yield a better resolution).

*Magnification:*

Is controlled by the current provided to the condenser and objective lenses, and the CRT.

*Pixels:*

The pixel diameter is given as 100 µm/magnification.

*Depth of field:*

Maximum depth of field is obtained from the smallest aperture setting and the longest working distance.

*Problems with imaging:*

1- Astigmatism: solution: use the stigmators

2- Noise

statistical fluctuations

electronic noise in detector

Solution: Use slow scans; in case of BSEI, use a higher beam current.

*N.B: contrast enhancement emphasizes noise.*

3- Charging artefacts: solution: a good conductive coat.