PHYS 345 Laboratory:  
Scanning Electron Microscopy

Purpose: To become familiar with the operation and imaging techniques of a scanning electron microscope, and to perform an energy-dispersive X-ray microanalysis of both known and unknown samples.

Apparatus: OWU Scanning Electron Microscope and associated computer-controlled data acquisition and analysis software; various metal alloy samples; small (~ 1 cm³) sample of experimenter's choice; unknown sample

References:

A.R. Sampson, Scanning Electron Microscopy,  


Elastic Scattering of Primary Electrons and Secondary Electrons and Detection,  
http://mse.iastate.edu/microscopy/backscatter.html ,

Electron Microscopy and Scanning Electron Microscope (SEM),  
http://www.unl.edu/CMRAcfem/em.htm ,
http://www.unl.edu/CMRAcfem/semoptic.htm.


Scanning Electron Microscopy (SEM),  
http://accept.la.asu.edu/PiN/rdg/elmicr/elmicr.shtml.

Scanning Electron Microscope Lab at Ohio Wesleyan University,  
http://go.owu.edu/~semlab/.


Wikipedia description of a Scanning Electron Microscope,  

Introduction

Put simply, scanning electron microscopy is the science of image magnification and analysis using a beam of electrons and its interactions with a sample of interest. The use of electrons has a distinct advantage over the use of light in that the resolving power (the ability to distinguish fine detail) is increased by two orders of magnitude. The best resolving power achieved by modern light microscopes is about 200 nm, while a scanning electron microscope (SEM) has a typical resolution of 1–5 nm. In addition, an SEM can provide a magnification that is about 250 times greater than the upper limit of
the best light microscopes. These capabilities, coupled with other qualitative and quantitative techniques, have given scientists from a variety of disciplines a high-resolution imaging tool for both basic and applied research. For example, biological scientists use electron microscopy to diagnose diseases, study developmental aspects of cells, and visualize subcellular components and structures such as DNA. In materials science and solid-state physics, electron microscopy is a valuable tool in the determination of crystal lattice structure, performing elemental analysis, studying defects and impurities in materials, and measuring the width and surface depth of the junctions between p– and n– type semiconductors.

At the heart of the technique is the SEM instrument, which is conceptually similar to a reflecting light microscope or a hand-held glass lens. The first SEM was developed in 1942 and consisted of an electron source, three electrostatic lenses, and electromagnetic scan coils placed between the second and third lenses. A photomultiplier tube detected the scintillations on a phosphor screen caused by secondary electrons emitted from the samples. Significant changes to the electron optics were performed in 1956, when the electrostatic lenses were replaced with electromagnetic coils, and a double deflection scanning system was added. In 1960, the signal collection process was improved by replacing the original phosphor screen system with a light pipe coupled between the scintillator and photomultiplier tube, improving system efficiency. The combination of each of these improvements into a single instrument was performed in 1965, when the first commercial SEM, the Cambridge Scientific Instruments Mark I, became available. The SEM at Ohio Wesleyan is the Leo 435VP model and was brought to campus in 1996.

This experiment will allow you to become familiar with the components and operation of a SEM, and will give you the opportunity to perform both qualitative imaging and an X-ray microanalysis (resulting in the determination of the elemental composition) of a sample.

Theory

A simple schematic of an SEM is shown in Fig. 1. Electrons are produced from a heated filament and attracted to a positively-biased anode, producing a monoenergetic beam. This system forms the “electron gun” or “virtual source” of the instrument (see Fig. 2). A series of three electromagnetic lenses in conjunction with two apertures focus the beam on the sample (see Fig. 3). A set of coils, called the scan coils, sweep the beam across the sample using a magnetic field generated by a time-varying voltage introduced to the coils. The sweeping occurs in an orchestrated grid pattern called a raster, similar to the raster in a television receiver.

The same varying voltage that is sent to the coils is also applied to a set of deflection coils around the entrance of a cathode-ray tube (CRT). The magnetic field generated from these coils causes the deflection of a spot of light on the monitor of the CRT. Since the CRT deflection coils and the scan coils are driven by the same source, the pattern of deflection of the electron beam on the sample is exactly the same as the pattern of the deflection of light on the CRT monitor.
Figure 1. A schematic of a SEM.

Figure 2. A schematic of a typical SEM electron gun.

Figure 3. A schematic of the electron optics in a SEM.
The incident electron beam creates a complex series of interactions with the sample (see discussion below), causing the emission of electrons and X rays from the sample. Detection of the electron emissions causes a voltage signal to be produced from a detector, which is then amplified. This amplified signal is applied to a pixel grid on the CRT and modulates the intensity of a spot on the CRT monitor. In this way, bright (dark) spots on the monitor appear every time a relatively large (small) number of electron emissions are detected from the sample. Surface peaks on the sample result in large electron emissions, while surface valleys yield reduced electron flux. Thus the SEM image consists of thousands of lighted pixels of varying intensity on the CRT monitor that correspond to the topography of the sample.

Figure 4 indicates the various types of emissions from a sample that can be detected following the interactions that occur between the primary electron beam and the sample. Of these, secondary electrons form the primary means of the formation of an SEM image. Secondary electrons are produced mostly by inelastic collisions between the electrons of the primary beam and those in the weakly-bound conduction band of the sample. By convention, they are defined as electrons emitted from the sample with a kinetic energy less than 50 eV.

Backscattered electrons result from the elastic scattering of beam electrons from the atomic nuclei of the sample. The energies of backscattered electrons range from 50 eV (using the same convention as that applied to the secondary electrons) up to the initial kinetic energy of the primary electrons in the incident beam, due to possible multiple scattering in the sample.

When beam electrons induce ionization of an inner-shell electron in the atoms of the sample, X rays characteristic of the element can be released from the sample (and later detected) when an electron from an upper shell fills the vacancy in the ionized shell with an energy equal to the energy difference between the two discrete states \( E_x = h\nu = E_i - E_f = \Delta E \). Alternatively, the X-ray energy may be transferred to another atomic electron, which leaves the sample as an Auger electron with a kinetic energy determined by \( \Delta E \) minus the energy necessary to overcome the ionization energy and the binding energy (or work function) of the sample. Both Auger and secondary electrons are highly susceptible to elastic and inelastic scattering and can leave the sample only from a very thin surface layer on the order of a few nanometers thick, thus making them a sensitive probe of the surface features of the sample. However, they can be generated by backscattered electrons originating from a deeper region of the sample surface, in addition to the narrow beam of primary electrons entering this thin surface layer.

Other electron beam/sample interactions include electron-beam-induced surface current (EBIC) and cathodoluminescence (CL), both byproducts of inelastic scattering with the primary electron beam. EBIC results from the charges liberated from the collisions, while CL refers to the emission of light resulting from electron transitions across energy band gaps in a sample.
Figure 4. The primary interactions that take place between the incident electron beam and the sample in scanning electron microscopy.

**Experimental Apparatus and Techniques**

The OWU SEM apparatus is shown in Fig. 5, with the major components labeled. Interior views of the vacuum chamber are displayed in Figs. 6 and 7. The sample platform and vacuum thermocouple (for measurement of vacuum pressure) are shown in Fig. 6. Most of the other hardware shown in the figure relate to the rotation and translation of the sample platform.

Figure 7 shows the various detectors used in the SEM. The secondary electron detector is of the Everhart-Thornton type (see S. L. Flegler et al.), consisting of a CaF scintillator with a surrounding Faraday cage used to both attract secondary electrons as well as to shield the electrons from the high voltage applied to the scintillator itself. When in use, the backscattered electron detector is inserted concentric to the primary electron beam (hence the hole in the center of it). It has a relatively large surface area to assist in the collection of the rather high-energy backscattered electrons. The X-ray detector is a lithium-drifted silicon [Si(Li)] crystal detector. The detector is kept at liquid
nitrogen temperature in order to reduce leakage current (electronic noise in the crystal resulting from free charges), and to prevent redistribution of the lithium as well as possible electrical breakdown in the crystal induced by the applied bias voltage due to decreased resistance at higher temperatures (true of all semiconductors in general).

Figure 6. Internal view of the SEM vacuum chamber, showing the sample platform.

Figure 7. Internal view of the SEM vacuum chamber, showing the various detectors used and the tapered injector of the primary electron beam.
Experimental Procedure

Familiarize yourself with the operation of the SEM. Make sure you are trained on the operation, control, and data acquisition techniques by the OWU Scanning Electron Microscopist.

Collect magnified images and X-ray spectra for the known sample, the sample that you supplied, and the unknown sample supplied by your instructor. (Note that conducting samples provide better images than non-conducting ones, since conducting samples provide a path to ground for the incident electrons, reducing surface charging of the sample which would appear as extraneous bright spots on the image.) Be sure to record all of the relevant SEM parameters for each image (magnification, beam current, and primary electron beam energy). Place a scale marker on each image you record to give a sense of its size. Record X-ray spectra using the electron beam energy recommended by the Scanning Electron Microscopist, with the primary electron beam placed in at least three different locations on each sample.

When performing an X-ray analysis of a sample, it is important to select the electron beam operating conditions that optimize accuracy and precision. For example, increasing the accelerating voltage will improve the X-ray count rate and peak-to-background ratio and therefore the precision and sensitivity, respectively. However, increasing the beam accelerating voltage will also increase the absorption of lower-energy X-ray lines. A correct determination of the accelerating voltage and beam current depends on the elements under investigation and the spatial resolution required. Ideally, we would like to select X-ray lines which are similar in energy for each of the analyzed elements in order to obtain similar excitation volumes from each element. A good rule of thumb for properly exciting X-ray transitions is to choose an electron beam energy that is about a factor of two larger than the largest expected X-ray energy (“edge” energy) from a particular element. For example, the edge energy for nickel, 8.33 keV, determines the minimum acceptable beam energy of about 16 keV which will adequately excite Ni K_\alpha radiation. For more information on this topic and a thorough introductory discussion on the methods of X-ray microanalysis in scanning electron microscopy, see S. L. Flegler et al.

Procedure quick reference

All of the SEM control functions can be selected from a menu of icons (or drop-down menus) in a Windows-based program. The following is a list of the most commonly used functions:

- Vacuum status window
  - Use “Vent” button to vent vacuum chamber to air pressure (valves to vacuum pump system shut and dry air introduced to vacuum chamber)
  - Use “Pump” button to evacuate vacuum chamber of air. (“Good” vacuum obtained when chamber pressure ~ 7 × 10^{-5} torr.)
- To start/stop primary electron beam: click on traffic light icon
• To alter brightness of image: click on sun/moon icon, then adjust by clicking and
dragging with left mouse button
• To change magnification/focus: click on magnifying glass icon, then adjust by
clicking and dragging with the left mouse button
• To change energy of primary electron beam: click on lightning bolt icon
• To alter position of sample in monitor: use joystick control of sample platform
• To perform X-ray analysis: run LinkISIS program
  o Set focus to 19 mm
  o Improve focus of sample on monitor using joystick “z” control
  o Electron beam energy can be typed in directly

Data Analysis and Discussion

Perform a qualitative study of the images produced by the three samples. Discuss
the general surface features and any variations in the features that you see, including any
possible stresses, fractures, impurities, or other defects. When a non-conducting sample
is used, some images may appear particularly bright in spots, as if a flashlight were
shining on them. Explain why this happens in terms of the electrical properties of the
sample material.

Determine the total background-corrected counts of each clear peak in one
representative X-ray spectrum for each sample. This is the total number of counts
subtended by a peak minus the number of (constant) background counts near the same
peak. What statistics should be used to calculate the random error associated with this
measurement? Calculate the random statistical error for the total background-corrected
counts for each peak based on the answer to this question. Calculate a “peak-to-
background” ratio (ratio of net peak counts to background counts) for each peak in each
sample spectrum that you analyze. Note that the bigger this ratio, the better. Why?

Perform an elemental analysis of all three samples that you observed with the
SEM. Using your X-ray spectra, describe the elemental composition of each sample, and
determine what the unknown sample is.

In your discussion of the physics of the SEM, provide the answers (with details on
how you obtained the answers) to the following questions:

(1) Assume that the electromagnetic lenses of the SEM have focusing properties
similar to that of thin glass lenses. If the three lenses of the electron optics
system each have a focal length \( f \) and are separated by a distance \( L \), what is the
effective focal length of the three-lens system?

(2) A small electron spot of diameter \( d_0 \) at the sample is produced by successive
demagnification of the first crossover image (see Fig. 2) of diameter \( d_c \) by
means of the three-lens system. Provided that each intermediate crossover
image is formed at a (relatively large) distance \( D \) in front of the next lens, the
subsequent demagnified image will be near the lens focus, at a distance
\((1 / f - 1 / D)^{-1} \approx f\), and the total demagnification of the system results in a
spot with the geometric diameter $d_0$ given by

$$d_0 = \frac{f_1 f_2 f_3}{L_1 L_2 L_3} d_c = m d_c ,$$

(1)

where $m \ll 1$ is known as the demagnification, and $L_1$, $L_2$, and $L_3$ are the separation distances between the source and the first lens, the first and second lenses, and the second and third lenses, respectively. If $f_1 = f_2 = f_3 = 5$ mm, $d_c = 30$ μm, and $L_1 = L_2 = L_3$, what is the maximum value of the demagnification in order to achieve $d_0 \leq 10$ nm? What then must be the minimum length of the electron optical column?

(3) The primary beam of electrons from the SEM source can undergo elastic scattering from the nuclei in the sample. Assume that this elastic scattering follows classical Rutherford scattering from an unscreened nucleus (see the *Experiments in Modern Physics* text or any textbook on modern physics). (Note that this treatment neglects the intrinsic spin of the electron and spin-orbit effects during scattering.) Determine the relative probability for scattering through angles $\theta \geq \alpha$ by calculating the partial cross section $\sigma(\alpha)$ for angles $\theta \geq \alpha$, given by

$$\sigma(\alpha) = \int_0^{2\pi} d\phi \int_\alpha^\pi \frac{d\sigma}{d\Omega} \sin \theta d\theta ,$$

(2)

where $d\sigma / d\Omega$ is the differential scattering cross section. Plot $\sigma(\alpha)$ as a function of $\alpha$ ranging from 0 to $\pi$ and note any singularities that occur in the graph. (Any singularity that occurs is a consequence of the long-range Coulomb field of the unscreened nucleus, but would disappear if screening were considered.)

(4) In principle, the SEM could be used to determine fundamental properties of the crystal lattice structures of the solids under investigation, since the electrons in the primary beam can be considered as waves that probe the lattice and interfere with each other upon diffraction from the scattering planes of the crystal. Discuss a possible experiment that could be done with the SEM to measure one or more intrinsic structure features of a particular crystal lattice.