NE 226 L

Characterization of Materials

Title Page

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1. Introduction

Scanning electron microscopy (SEM) is one of the most versatile methods available for the analysis of solid materials and the production of high-resolution images of sample surfaces. In SEM, an electron beam is rastered across the surface of a sample over a rectangular area which interacts with the sample surface producing a number of different signals. These signals can be analyzed to provide useful information about the sample such as morphology, surface topography, composition, and crystallography.

2. Questions

1. Describe the interaction of a high energy electron beam and the resultant production of x-rays, backscattered electrons and secondary electrons. Where in the solid are the different electrons and photons produced? From where in the solid do they escape?

When an electron beam hits matter, the beam broadens due to strong elastic scattering effects. At the same time, inelastic interactions cause electrons to lose energy. The energy of the electrons will be completely transferred to the sample if the sample is thick enough. Because of these various elastic and inelastic processes, the overall result of the electron beam interactions with the sample will be a pear-shaped (or teardrop-shaped) interaction volume (Figure 1). Because the electron beam is high in energy, the electrons will penetrate the sample relatively deeply.

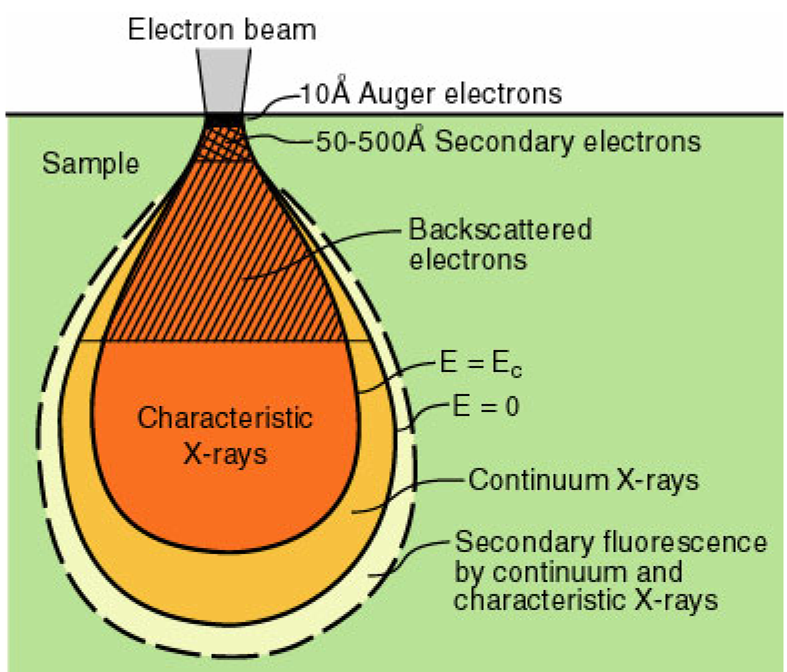

Figure 1 Interaction of electrons with matter

Electrons in the conduction or valence band can escape with a minimum amount of energy (the work function E_W of the material). The excited electrons move towards the surface of the sample undergoing elastic and inelastic collisions until it reaches the surface, where it can escape if it still has sufficient energy. One of the major reasons for coating a non-conductive specimen with a conductive material (e.g. gold) is to increase the number of secondary electrons that will be emitted from the sample.

Backscattered electrons are the electrons that are elastically reflected back upon collision with a large and positively charged nucleus. As such, these electrons are produced from a little deeper into the surface of the sample and are ejected from the top surface of the specimen at high angles. The number of backscattered electrons produced from a material depends strongly on a sample's average atomic number, Z.

X-rays, being composed of photons, can be produced from anywhere within the pear-shaped interaction volume. X-rays are produced when an electron from the sample is excited to a high energy state by an incident electron. Relaxation of this electron back to its ground state results in the emission of a photon whose energy is in the x-ray range.

2. Compare imaging using backscattered and secondary electrons. What differences might be observed? Why? Which electrons are more useful for surface imaging? Why?

Because of the low energy of the secondary electrons–which are essentially electrons that were originally in the conduction or valence bands–they can only escape from the sample if they are generated close enough to the surface. Consequently, recording the energy of interaction of the secondary electrons can only give us topographic information about the surface structure.

Backscattered electrons (BSEs), on the other hand, provide compositional information about the sample. BSE imaging takes advantage of the fact that heavy atoms with a high atomic number Z are much stronger scatterers of electrons than light ones and therefore cause a higher signal. We can use BSE imaging to detect crystalline areas, defects and grain boundaries. We can also perform phase and particle size analysis from BSE imaging. Detection of small clusters or even single atoms of a heavy metal in a matrix of light elements (Z-contrast) may be obtained from BSE imaging.

Secondary electrons are more useful for surface imaging. For one, the resolution using secondary electrons is much better than when using backscattered electrons. Secondly, the shallow depth of production of detected secondary electrons makes them very sensitive to topography.

3. What information does EDX give? What is the minimum electron gun voltage that should be used?

Energy-dispersive X-ray spectroscopy (EDXS) gives us qualitative and quantitative information about the elements present in the sample. Most importantly, EDX provides laterally resolved information about the sample composition. The EDX spectrum contains characteristic peak positions that correspond to the possible transitions in its electron shell. The more number of transitions possible, the more number of peaks will be present.

For EDX spectroscopy, a minimum electron gun voltage of 20 kV (energy of the electrons will then be 20 keV) must be used to have enough energy to excite electrons to higher states which upon relaxation will produce x-rays with wavelengths between ten and one thousand picometers.

4. Draw a diagram and describe the main parts of a SEM.

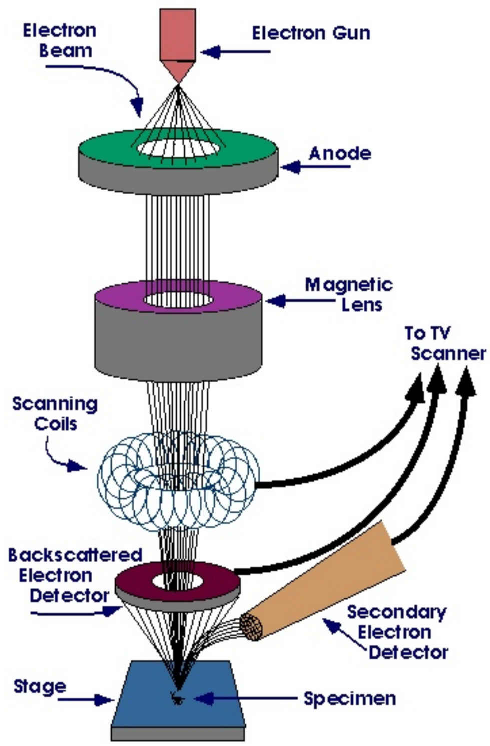

Figure 2 Basic components of a scanning electron microscope

The SEM is an instrument that produces a largely magnified image by using electrons instead of light to form an image. A beam of electrons is produced at the top of the microscope by an electron gun. The electron beam follows a vertical path through the microscope, which is held within a vacuum. The beam travels through electromagnetic fields and lenses, which focus the beam down toward the sample. Once the beam hits the sample, electrons and X-rays are ejected from the sample and are detected by the various detectors placed around the sample. The scanning coils are essential for scanning the sample in raster pattern.

5. Describe how a sample surface is scanned with the electron beam in an SEM. What is rastering?

The electron beam passes through pairs of scanning coils in the objective lens, which deflect the beam horizontally and vertically so that the beam scans in a *raster* fashion over a rectangular area of the sample surface. This is much like how conventional CRT TV's and monitors work.

Rastering is the process of scanning a surface in a pattern of parallel lines guiding the electron beam starting from the top left and ending at the bottom right.

6. What is the difference between a Field Emission SEM and the Tungsten filament source used in the SEM shown in class?

In a typical SEM, electrons are *thermoionically* emitted from a tungsten cathode and are accelerated towards an anode. Tungsten is used because it has the highest melting point and lowest vapour pressure of all metals, thereby allowing it to be heated for electron emission.

Alternatively, electrons can be emitted via a process known as field emission. Field emission is a form of quantum tunneling in which electrons pass through a barrier in the presence of a high electric field. This phenomenon is highly dependent on both the properties of the material and the shape of the particular cathode. One remarkable advantage of using field emission for generating electrons is the method's dramatically higher efficiency in terms of less scatter of emitted electrons and faster turn-on times.

3. References

[1] Q. Xie, F. McCourt, *Nanotechnology Engineering NE 226 Lab Manual*, University of Waterloo, Waterloo, pp 61-67 (2007).