MY4200 Lab #3: Specimen/Beam Interaction

Introduction

The operator of the scanning electron microscope (SEM) has available a range of operating parameters that affect the nature and intensity of the signals created by the interaction of the electron beam and the specimen. In addition, different types of specimens interact with the beam differently. If the operator is familiar with the nature of the specimen, operating conditions can be chosen to optimize performance of the SEM for the information required.

High resolution in secondary electron images (SEIs) can be accomplished at either high or low voltage operation. Low voltage limits the volume of interaction with the sample, although it often increases the aberration effects. High voltage is preferred in high-resolution imaging of targets made of heavy elements as for such targets the interaction volume is small and aberration effects are reduced.

Backscattered electrons escape the sample with little or no loss of energy, and are a function of average atomic number in the region on the specimen interacting with the beam. Backscattered electron images (BEIs) are optimized with a high current and provide information about compositional variation in the sample.

In some cases, nonconductive samples can be imaged without coating by balancing the current in the beam with electron emission by the sample.

Since changing one operating parameter can often be detrimental to another, the user must learn to optimize parameters for each sample to produce the best image possible.

Experiments

Each group will acquire SEIs and BEIs requiring very specific conditions for optimum quality and resolution. In addition, the group will determine the diameter of the probe under different condenser settings. All images in this exercise will be acquired at a working distance of no more than 15 mm and using the 50 μm aperture.

1. High resolution imaging

Images: A high resolution SEI of gold nanoparticles at three different accelerating voltages (35, 20, and 5 kV) will be obtained at the highest magnification the students can obtain using the tungsten filament cathode. The experiments will be carried out with short working distance (8 mm) and strong condenser lens.

Determination of probe diameter: It is important to know the probe diameter that is produced at different voltages and at different condenser settings in the SEM being used, since probe diameter is the primary limiting parameter in resolution. Students will scan the beam in line mode across the edge of a Faraday cup and determine the diameter of the beam by measuring the region between the metallic edge of the cup (white) and the cage (black) on the resulting line scan. Probe diameter will be determined at three condenser settings (6, 10, and 14) using the 50 μm aperture at 35, 20, and 5 kV accelerating voltage.

2. Backscattered electron imaging

A multiphase rock sample will be analyzed using BEIs for the purpose of identifying the number of phases present in the sample. This will require careful adjustment of signal strength.

3. Effect of sample charging on quality of the image

Students will image an uncoated specimen of an alumina at a voltage that is less than the voltage referred to as "crossover point" (Goldstein et al. 2003), to obtain a charge-up free image. Short working distance will be used and the experiment will start with 1 kV. The accelerating voltage will be raised and images will be collected to see the effect alumina charging on the quality and resolution of the image. The ultimate goal is to obtain resolution in a range of 100-200 nm.

Report

The report should include all images that were acquired, along with a complete description of the conditions under which each image was acquired. The discussion should address why the parameters that were used were chosen, the effect of the chosen parameters, and explain how the interaction of the beam with the specimen produced the images with the desired information. Also, discuss the information that was obtained in the images.
Introduction should include three subsections briefly reviewing: high resolution imaging (section 5.1 in the textbook), compositional contrast (section 4.4.2), and charging effects in nonconducting specimens (sections 15.1 and 15.2).

Reference

J. Goldstein, D. Newberry, D. Joy, Ch. Lyman, P. Echlin, E. Lifshin, L. Sawyer, and J. Michael, 2003, "Scanning Electron Microscopy and X-ray Microanalysis," Kluwer Academic/Plenum Publishers, New York, pp. 89-91 and 656.