Ge 116 Winter 2017

Lab project Week 1: Scanning Electron Microscope, part 1 (instrument use and imaging)

Report due Tuesday January 10. Electronic submission is acceptable, so is paper.

This exercise is a combination of learning to use the instrument and actually analyzing your sample. Many steps are things to do rather than questions to answer. Products that should be turned in are shown in **boldface**. Make yourself directories within Users/GPS/Ge116 on both the SEM computer and the EDS-EBSD computer to store your results.

*Note: Sometimes students in this class already have some experience on one of the instruments being taught. If, any week this term, the assignment feels too basic or redundant to you, please ask and we will design a more advanced exercise!*

A. Required reading for instrument check-out

All new SEM users are required to **read the following three documents**:

<http://www.gps.caltech.edu/facilities/analytical/sem.html>

<http://www.gps.caltech.edu/facilities/analytical/sem_user.html>

<http://www.gps.caltech.edu/facilities/analytical/sem/LEOSEM_CIT_v4.pdf>

Please read these *before* your lab session this week.

A. Sample preparation- This has been done for you so we can start right in, but you should know what is involved in preparing a sample for SEM work…

The rock was sawn into wafers and cored to make 1” diameter rounds. One side was mechanically polished to optical mirror finish, to at least 0.25 micron grit. For high-quality SEM work additional steps included:

1. *Final vibro-polish*: The vibrating polisher in the Analytical Facilities lab uses a colloidal silica suspension as abrasive. This gives a finish good enough for EBSD work next week.

2. *Carbon coat*: An insulating material like a rock needs a thin coat of conductive material on the surface to avoid charging artifacts when an electron beam is incident on the surface. For SEM use we place a ~12 nm coat of carbon on the surface using an evaporative coater. Then we will wipe it off for EBSD next week. Then again we will recoat it with a thick coat for Electron Probe analysis in two weeks.

B. Basic instrument operation

1. *Start-up, vacuum system, sample loading*: Start the SEM software, sign-in as user asimow, password asimow. Get your sample ready to mount on the SEM stage with C tape to provide ground path from carbon coat to instrument stage. **Invent and document (e.g. with a sketch or mobile phone photo) a coordinate system that breaks the circular symmetry of your sample so that you can load it in a consistent orientation from week to week and instrument to instrument**. Vent the chamber. Insert your sample. Pump to vacuum (learn about vacuum system diagnostics, high vacuum and variable pressure modes). For now, use high vacuum mode. Learn about sample chamber layout, chamber camera viewing, basic instrument safety (This machine is NOT fool-proof; you can break it; please don’t).
2. *Electron column, beam conditioning*: Learn how to get an electron beam, choose accelerating potential and beam current, align the electron optics, focus the beam at given working distance, and stigmate the beam. For starters, use 10 kV accelerating potential, a 30 m aperture (do not select high-current mode), and an 8 mm working distance.
3. *Stage and scanning controls*: In the course of focusing, you will learn the steps necessary to find and focus on regions of interest on your sample: stage navigation, scanning (scan rates and raster sizes), focus and stigmation, brightness and contrast, and zooming.

C. Electron imaging

1. *Secondary electron image*: Secondary electrons are most sensitive to sample topography. Our SEM has several secondary electron detectors; we will learn something of the strengths and weaknesses of each, but we will begin with the SE2 detector for this task. We will verify that our sample is flat and well-polished and that the beam condition steps above are correctly done by taking SEI images.
   1. **Capture a well-focused survey SEI image of your sample at lowest possible magnification with suitable brightness and contrast**.
   2. Choose an imperfection on the surface (a pit or crack) and zoom in on it. **Capture the highest magnification SEI image of this feature at which you can get good image quality (at least 1000x, but see if you can get 50000x, or even 100000x!)**. **Estimate the spatial resolution you are able to achieve (smallest distinctive feature or separation between features).**
   3. **Repeat step (b) using the In-Lens SEI detector and the same target. Is your spatial resolution better or worse?**
2. *Backscattered electron image*: Operating conditions: 15 kV acc. voltage, 120 m aperture, 20 mm W.D. Backscattered electrons are most sensitive to mean atomic number of the sample material. With optimized contrast it is easy to distinguish variations between adjacent elements in the periodic table at moderate concentrations. There are trade-offs between imaging parameters (accelerating potential, aperture, working distance) that we will explore somewhat. Our instrument has two backscattered electron detectors; learn about the strengths and weaknesses of each, but use the AsB detector for this task.
   1. Go back to low magnification, optimize brightness and contrast to distinguish among the minerals in your sample and, using the mapping function of AZTec software on the EDS/EBSD computer, **capture a mosaic of BEI images as a navigation map of the general mineral texture of your whole section**. Use 512 pixel resolution to save time. In AZTec you select three points on the boundaries to define a circle and then the software will propose a tiling of rectangles to cover it. Be sure to choose a brightness and contrast that will work for the whole section [Note: you may as well capture EDS data, at 256 pixel resolution, while you are doing this (so it will be co-registered with the BSE image coordinate system), but we will not look at it until next week]. Adjust the imaging parameters so that you can grab the whole map in under an hour. After capturing the frames, click *montage* to combine them into a single mosaic image.
   2. **Pick 3 areas of interest and capture moderate magnification BEI images of these areas. Note the location of these areas on your overall map.** We will return to these areas over the course of the quarter; you can pick areas that seem to have most of your major minerals, have an abundance of trace minerals, an area with neat BEI patterns or zoning, etc.
   3. *Do this at least three times on different major mineral targets*: zoom in on a single grain of some mineral, optimize brightness and contrast to reveal any zoning that might be present within the grain. Design imaging parameters for maximum Z contrast and **capture a moderate-magnification BEI of the grain**.
   4. *Also do this at least three times on different accessory mineral targets*: zoom in on a single accessory mineral or inclusion, design imaging parameters for maximum spatial resolution and try to obtain **the highest magnification BEI image at which you can get good image quality**.

3. When you are done, retrieve your sample, store it in a labeled container in the dessicator in the SEM lab, and return the machine to standby properly. Transfer your image data to yourself (by FTP, dropbox, e-mail, memory stick, whatever).

D. Interpretation (i.e. work on these questions after you are done using the machine)

1. *Mineralogy*: Based on backscatter contrast, **how many different minerals can you identify in your sample?** **Rank the minerals by typical backscatter brightness** (noting that some may be zoned and cover a range of brightnesses) and **characterize each distinct mineral by typical shape and size (estimate the mean size for each phase)**. You may at this stage apply petrological insight, thin-section, or hand-sample observation to attempt to identify some of the minerals, but we will get more diagnostic information later so this would just be preliminary.

*2. Texture*: Based on your BEI navigation map, describe the texture of the rock. **Is there a shape-preferred orientation apparent on the plane of your section? Defined by what textural features?** Are there any other notable textures, such as variations in size, overgrowth or replacement textures, symplectites, etc? Given that you may not yet have identification of the minerals that make up the texture, **summarize the textural features of your sample (mineral size, anisotropy, relations between minerals, etc.)**.

*3. Areas of Interest:* **On a copy of your BEI map, locate and mark the 3 areas of interest. Bring a copy of your BEI map, marked with areas of interest, as well as BEI images of the areas themselves to the next lab session** (on laptop or paper).