

## Introduction to VP SEM – Imaging and EDS

The purpose of this lab is to introduce you to operation of the SEM in “variable pressure” mode. Two aspects will be introduced using specimens that are NOT conductively coated:

- (1) imaging of uncoated insects, observing (a) pros/cons of the BSE and the ESED detectors and (b) optimizing depth of field for these 3D objects, and
- (2) acquisition of EDS spectra on various minerals present in a polished rock section (banded iron formation: quartz, ankerite, magnetite) and observation of (a) Duane Hunt Limit and (b) the skirt effect.

Each student should get 20-30 minutes on the instrument. Save acquire several images/spectra, saving in 777 folder on the computer.

1. VP-SEM Imaging: The first sample is a 1” stub with sticky carbon tape, and upon it are mounted several Weeks Hall volunteers (house flies, etc). Using what you have learned about operating the Hitachi S3400N, properly insert the specimen into the chamber. Pump down in VP-SEM vacuum mode to 30 Pa. Select initial settings you think might be beneficial for imaging: \_\_\_ HV, \_\_\_ Filament, \_\_\_ Gun Bias, \_\_\_ Probe Current, \_\_\_ Aperture setting

Notice that under Image in the right hand column, “SE” is not accessible. Why?

The default image in VP-SEM is BSE. Leave it there to start.

Locate an ‘interesting area’ and then make any necessary adjustments in order to optimize (1) depth of field and (2) objective lens focus. You may wish/need to (3) change the gas pressure and also (4) change the scanning mode from one long 20 second acquisition to 16 averaged rapid acquisitions.. As the class progresses, write down “lessons learned” about these concepts and adjustments to the SEM. You will summarize these in a paragraph afterwards.

Depth of field: when you start, you will be setting the sample to the ‘normal’ 10 mm working distance. Focusing upon one fly, at this working distance, can you have the whole fly in focus? \_\_\_\_\_ Adjusting the objective lens, what do you estimate the height of the fly to be? \_\_\_\_\_ mm

As you zoom in on an area, do you observe part of the image getting white/whiter? \_\_\_\_  
What is this?

Why does it occur upon zooming in at higher mag?

For all changes made, observe the same field of view in both BSE and ESED modes.

If/When you lengthen working distance, what is the impact on the images?

If/When you increase the gas in the chamber, what is the impact on the images?

Recall that images can be acquired both in one long slow 20 second capture, or 16 very fast captures that are averaged. When would the later be good?

Also, the BSE Detector has some settings you need to be aware of. "Normally" it is set for Gain of 3, but for very low currents you want to set to 4 (HR=high resolution with low currents).

Also you can operate the BSE with the diodes mathematically added/subtracted (Composition vs Topography), AND you can add an off axis BSE diode for "Topo" with enhanced shadowing effect.

If you make other adjustments to the SEM (E0, probe current, apertures), note them and comment upon the effect upon the images.

2. EDS in VP-SEM: We will be investigating a small (2-3 mm high x cm wide) piece of banded iron formation (which contain typically quartz and an Fe-rich phase, magnetite/siderite/ankerite/hematite). Position the sample in the chamber with the arrow pointing up (toward the wall). Today you will be using the Thermo Fisher EDS system, which is located on the right NEC monitor.

First, set the vacuum to **high vacuum**, "SEM" , in the Conditions menu. When it is ready turn on the HV (15 kV is good).

You need now to do is get an image with the Hitachi software.... Do that now...notice any problems with the image? \_\_\_\_\_ Any ideas why?

The icon on the desktop is "NORAN System Six" (near top left). First person will start it up. Note the file structure: you must always operate in a "project" which is a green folder, to have full functionality. Always click the + button to the left of the master folder you are using (here, G777), which then shows what folders are inside it, etc. If you click on a folder, then you are at the 'root' level of that folder and not inside another

folder. If you want to create a new folder (strongly recommended that all your work is kept in distinctive folders, usually by a date or topic). Click OK to exit this window.

In the top menu, locate the yellow “Edit Acquisition Properties” button (to left of the blue ‘cactus’ icon). Here is where important parameters are:

Live Time and Time Constant. Click on and Investigate the possible ‘suggested’ settings and write down the microseconds and possible count rates here:

Auto	_____usec	_____ cps
Slow	_____usec	_____ cps
Classic	_____usec	_____ cps
Mapping	_____usec	_____ cps
Fast Mapping	_____usec	_____ cps

The instructor will explain what “auto” means. Write down some notes here so in your final writeup you will be able to explain it.

For today, let’s leave TC set to Auto. Set the **Live** Time to 30 seconds

We usually operate in Point & Shoot mode (second option on left vertical menu).

OK, now acquire an image with Noran: click the “strat column” icon at the top (“Acquire an averaged electron image”). This is a *LOW* resolution image; wait until control is returned to the Hitachi before proceeding. Now “point and shoot” at the **darkest phase** you see in the BIF. Now while we are waiting, look at the bottom right of the Noran monitor display. First: green bar is counting down....DT is some value...and SR is stored counts, which means the count rate. What is the DT? Because we set the TC to “AUTO” it is selecting the smallest TC it can have to try to keep the deadtime under 50%. OK, when it is finished, go back to the Hitachi and reset the vacuum to SEM (high vac).

Click the X button in top menu and then the LOG scale. What is the Duane Hunt limit? \_\_\_\_\_ Are you surprised? \_\_\_\_\_ Why or why not?  
\_\_\_\_\_ We will return to this shortly.

Now let’s go to the proper vacuum setting: First turn off the high voltage, then set vacuum to VP-SEM at 30 Pa. When it is ready, turn on the HV. Find a dark grey phase and set up an image in Hitachi, then go to Noran SS and point and shoot the grey phase.

Inspect the spectrum. One of the useful (but dangerous if used improperly) is the “AutoID” function. What are the highest peaks? \_\_\_\_\_ and \_\_\_\_\_ Any guesses what this mineral might be? \_\_\_\_\_

What is the next highest element apparently present? \_\_\_\_\_ Does this make sense? \_\_\_\_\_ Increase (stretch) the height of the spectrum, and list the other peaks that are "AutoID'd".

The instructor will apply "black magic" and generate a "standardless analysis" of this point on the sample. Write down the results:

Do these numbers make sense? \_\_\_\_\_ Do they show you that it is foolhardy to expect standardless EDS in VP-SEM to provide correct quantitative results? \_\_\_\_\_

You should acquire an image of a different phase (mineral) in this sample and use the NSS software to acquire several spectra, and identify the peaks.

For your assignment, write up two paragraphs describing (1) what factors are involved with imaging in VP-SEM mode and (2) what issues are involved with EDS spectra acquired under VP-SEM conditions.