Geology 777 Lab 4 Week of Feb 18, 2014 Name_____

# 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 ~15 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: 15 HV, ___ Filament, ___ Gun Bias, 50 Probe Current, Aperture setting = 0 (Always check the aperture setting before you start!)

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 both High Vacuum and in VP-SEM modes: We will be investigating a small rock specimen: either a piece of banded iron formation (which contain typically quartz and an Fe-rich phase, magnetite/siderite/ankerite/hematite), or iron oxide-cemented sandstone. (If there is an arrow on the sample, 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?

Start up the "NORAN System Six" EDS software on the right monitor

In the top menu, locate the yellow "Edit Acquisition Properites" button (to left of the blue 'cactus' icon). Here is where important parameters are:

Live Time -- set to 60 seconds -- and Time Constant – set to auto.

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 either the **brightest** or the **darkest phase (different students choose different ones)** 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 _____% and SR is_____cps.

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? _____
_____ Are there any spectral artifacts?
_____

Now let's go to the VP-SEM 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".

Now use "Spectral Match" to auto ID the mineral: list the top three matches

1 _____ 2_____ 3_____

Click thru the top three match results and compare their spectra. How useful do you think this feature is? What might be some limitations?

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.

What is the equation for the "skirt" radius (make sure you use proper units)?

Now fill in the blanks: Z of gas (assume nitrogen) ___
L (working distance)=___ mm keV used ____
Pressure ___ Pa Temperture ___ K

For your assignment, write up several paragraphs describing (1) what factors are involved with imaging in VP-SEM mode, (2) what issues are involved with EDS spectra acquired under VP-SEM conditions, including (3) what you calculate the skirt radius to me in microns. Include images where they demonstrate points you make.

Versions: 10/4/09 10/6/09 2/20/12 2/18/13 3/6/14