Physics 7230
Laboratory 4: Quanta 600 Environmental SEM: Low vacuum mode and charging

Introduction:
This laboratory is designed to help you collect SEM data on non-ideal samples that are non-conductive. Today you will be working on the FEI Quanta 600 FEG ESEM. This variable pressure SEM has high, low and environmental vacuum modes. Using these variable vacuum modes, one can counteract charging issues that occur when visualizing non-conductive specimens.

Samples:
1. Uncoated sandstone chip
2. Filter paper

Objectives:
1. Proper startup and vacuum level checks
2. Specimen exchange
3. Alignment and working distance determination
4. Image capture
5. Changing vacuum modes

Operating Conditions
1. SEM: 10, 5 kV; Spot 3.5 Aperture #6; 12 mm WD.
2. Electron Detectors: Everhart-Thornley SE and BSE; Large Field Detector (LFD, SE) low vac mode Quad BSE detector.
3. Imaging: 1024 X 884; 40 ms dwell time
1. Why must we gently close the specimen exchange chamber door?

2. What are the vacuum levels associated with high, low and environmental modes? How are these vacuum levels achieved?

3. What are at least two ways of imaging/compensating for a non-conductive sample?

4. What is the difference between a single secondary electron image and an integrated multiple secondary electron image?

5. How would one determine the “plateau point”?
PART I. SANDSTONE ROCK
Operational Parameters
SEM: 10 kV; Spot 3.5, Aperture 4; 12 mm WD, BSE &LFD detectors mounted
Imaging: 1024 X 884

Start-up
1. Log in book. Record date and time
2. PC should be on
3. Press the START button on the toolbar at the top of the desktop.
4. Select Quad 4 (chamber scope). Click anywhere inside the Quad 4 to select it.
   1. Note: The information field will be highlighted Blue to indicate when a Quad is selected.
5. Make sure Quad 4 is Live. If the PAUSE icon is in the upper left corner, click on the Pause icon in the upper field.

*****NOTE: Always view the sample exchange with the IR camera active!*****
Specimen exchange
1. Sample holder should be well below the 10mm marker.
2. Click the VENT button. Wait until the venting sound stops, ~ 1 min.
3. Open the CHAMBER DOOR. Wait until the venting sound stops then easily pull the door out with the handle at the bottom.
4. Insert the SANDSTONE sample, secure with allen wrench.
5. Make sure Quad 4 is still active.
6. **Gently close door!! While watching the chamber scope, slide the door in slowly, making sure the sample is below the 10mm mark. Stop when it rests against the o-ring and then very gently push it closed.

***** If the door is closed to hard and fast, the resulting pressure could damage the thin Be window on the EDS detector!!*****

7. Select HIGH VACUUM. Vacuum condition is selected under MODE.
8. Click on the PUMP button. It is located at the top-right side under VACUUM
9. Wait for the SPECIMEN CHAMBER to turn green. At the bottom there is an icon of the electron optical column. When the vacuum reaches ~4X10⁻⁴ torr, the chamber will go from orange to green. Also the BEAM button will undimmed.
   a) Note: The gun/column half should always be green. If not, please get help.

Initial Start-up
1. Under the STAGE menu, engage CENTER POSITION.
2. Click the BEAM ON button.
3. Under most SCAN Menu, go to PREFERENCES
4. Go to Databar
   a. Make sure the data field contains: High Voltage, Horizontal Field Width, Working Distance, Detector Type, and Pressure
5. Go to Presets
   a. Add 200X TO THE MAGNIFICATION PRESETS.
6. Select HIGH VOLTAGE and SPOT located under COLUMN. (10 keV, Spot 3.5).
7. Select Quad 1 ETD (SE) and Quad 2 BSE (A-B. topo) & make it active.
8. Open the Detector Settings Window and drag it in Quad 3.
9. Select the SCAN RATE and IMAGE RESOLUTION.
   a. Scan rate is selected with the up/down buttons between the turtle (slower) and the rabbit (faster). Set this at 0.3 µs.
10. Set image resolution to 1024 x 884.
Acquire Image and Set Working Distance (“Link Stage”)

1. Obtain an SE image by unpausing Quad 1
   ****NOTE: image will be charging so get the best you can****
2. Adjust brightness/contrast and focus
3. Increase magnification to 2000x and refocus
4. Link the Forward Working distance to the Stage.
   
   ***** Make sure the working distance read at bottom of active Quad matches the Z height coordinates (~60 mm)****
5. Decrease the mag.
6. Set the sample at WD at 25 mm.
   a. Type value in Z coordinates.
   b. Click on “Go To” button.
7. Increase magnification to 2000x and refocus
8. Link the Forward Working distance to the Stage.
9. Decrease the mag.
10. Set the sample at WD at 12 mm.
    a. Type value in Z coordinates
    b. Click on “Go To” button.
11. Increase magnification to 2000x and refocus
12. Link the Forward Working distance to the Stage.
13. Adjust brightness and contrast.

Perform Alignments and Adjust Astigmatism

1. Align the lens.
2. Correct for astigmatism (using Sigmator X&Y).
3. Zoom down to ~100x and activate Quad 2 BSE
4. Adjust brightness and contrast.
**SE IMAGING A NON-CONDUCTIVE SAMPLE IN HIGH VAC MODE**
1. Go to 200X.
2. Set Scan Rate to 10 µs.
3. Adjust the brightness/contrast using the Videoscope, signal in the lower half.
4. Take first SE image – label it *sandstone 200x ET SE*, save in a new folder (Lab LV)
5. Set scan speed to 100 nanoseconds
6. DO NOT ADJUST brightness and contrast
7. Next to the PAUSE button is SELECT FILTER MODE
   a) Select integrate
   b) Select Number of Frames – 64.
   c) A warning box appears, just hit OK
8. Take 2nd SE image – label it *sandstone 200x ET SE int*
9. Go back to SECT FILTER MODE, and select Live

**BSE IMAGING A NON-CONDUCTIVE SAMPLE IN HIGH VAC MODE**
1. Switch ET from SE to BSE
2. Set scan speed to 10 µs
3. Adjust brightness and contrast
4. Quad 2 should be active, BSE topo mode (A-B)
5. Adjust brightness and contrast
6. Take both images simultaneously.
   a. Label Quad 1: *sandstone 200x ET BSE*
   b. Label Quad 2: *sandstone 200x SS BSE topo*
7. Switch Quad 2 to BSE Z contrast mode (A+B)
8. Adjust brightness and contrast,
9. Take another image, *sandstone 200x SS BSE Zcon*

**IMAGING A NON-CONDUCTIVE SAMPLE IN LOW VAC MODE**
1. Turn the BEAM off by depressing the Beam button
2. Set CHAMBER PRESSURE to 0.3 torr.
3. Select LOW VAC Mode
   a. Select no Accessories and OK
   b. Wait for the green light
   c. Quad 1 should now be set to LFD (Large Field Detector) SE
4. Turn on BEAM
   *****VERY IMPORTANT – SEE WARNING ON THE SEM CHAMBER DOOR******
   a. Never go above 70% contrast with the LFD!
   b. It will cause electrical arcing between the detector and the sample and shut the system down.
5. The image with the LFD will be very bright. Adjust by:
   a. Turn down the contrast to 50%
   b. Set the Enhance mode to 50% (Enhance mode digitally expands the contrast)
   c. Set brightness accordingly
6. Set the scan rate to 10 µs
7. Adjust the Enhance and brightness using the Videoscope.
8. Take another micrograph, *sandstone 200x SS SE LFD 0-3*
9. **Optimizing the LFD signal:** As you increase the pressure, the number of electrons will increase the output signal, plateau, and then fall. We want the **plateau point**.
   a. Turn Videoscope back on
   b. Do not adjust contrast or enhance during these steps.
   c. Increase the Chamber pressure .4 torr (read the pressure at the bottom)
   d. Lower brightness back to where it was.
   e. Continue to increase the pressure by 0.1 torr, adjusting the brightness until the brightness doesn’t increase or decreases and set the pressure at the maximum point.
10. Take another micrograph, *sandstone 200x SS SE LFD 0-x* (in torr)
11. Go to the BSE Quad and take a BSE image. **sandstone 200x SS BSE Zcon LV**
Exchange Samples
1. Make sure Quad 4 is UNPAUSED to view the inside of the specimen chamber.
2. VENT the specimen Chamber
3. Get assistance to remove the BSE detector and to insert the low keV cone.
4. Put in the filter paper sample
5. Go back to High Vacuum mode
6. Hit Pump
PART II. FILTER PAPER

Operational Parameters
SEM: 5 kV; Spot 3.5, Aperture 6; 12 mm WD, LFD detectors mounted
Imaging: 1024 X 884

B. Initial Set-up
1. Increase magnification to 2000x and refocus
2. Link the Forward Working distance to the Stage.

***** Make sure the working distance read at bottom of active Quad matches the Z height coordinates (~60 mm) ****

3. Decrease the mag.
4. Set the sample at WD at 25 mm.
   a) Type value in Z coordinates.
   b) Click on “Go To” button.
5. Increase magnification to 2000x and refocus
6. Link the Forward Working distance to the Stage.
7. Decrease the mag.
8. Set the sample at WD at 12 mm.
   a) Type value in Z coordinates
   b) Click on “Go To” button.
9. Increase magnification to 2000x and refocus
10. Link the Forward Working distance to the Stage
11. Zoom back to 500X

C. Imaging at high vacuum and low keV
1. Set scan to 10 µs
2. Adjust brightness and contrast focus
3. Take an image – paper 500x HV SE
4. Switch to ETD, BSE mode.
5. Adjust brightness and contrast focus
6. Take an image – paper 500x HV BSE
7. Switch back to ETD, SE
8. Zoom up to 10KX
9. Adjust brightness and contrast focus
10. Take another image – paper 10Kx HV SE
11. Why is it jumping around?
12. Under select filter mode, do another integration
   a) Set scan rate to 100 ns!
13. Save image as paper 10Kx HV SE int
15. Turn Beam off

D. Imaging at low vacuum and low keV
1. Set CHAMBER PRESSURE to 0.2 torr.
2. Select LOW VAC Mode
   a) This time, set accessories to Low KeV cone.
3. Wait for the green light
4. Quad 1 should now be set to LFD (large field detector) SE
5. Turn on BEAM

**********VERY IMPORTANT – SEE WARNING ON THE SEM CHAMBER DOOR**********
a) Never go above 70% contrast with the LFD! It will cause electrical arcing between the detector and the sample and shut the system down.

6. The image with the LFD will be very bright. Adjust by:
   a) Turn down the contrast to 50%
   b) Set the Enhance mode to 50%
   c) Set brightness accordingly
   d) Note: The Enhance mode digitally expands the contrast

7. Set the scan rate to 10 useconds

8. Adjust the Enhance and brightness using the Videoscope.

9. Take another micrograph, paper 10kx SE LFD 0-2

10. Optimizing the LFD signal. As you increase the pressure, the number of electrons will increase the output signal, plateau, and then fall. We want to find the plateau point.
    a) Turn Videoscope back on
    b) Do not adjust contrast or enhance during these steps.
    c) Increase the Chamber pressure .3 torr (read the pressure at the bottom)
    d) Lower brightness back to where it was.
    e) Continue to increase the pressure by 0.1 torr, adjusting the brightness until the brightness doesn’t increase or decreases and set the pressure at the maximum point.

11. Take another micrograph, paper 10Kx SE LFD 0-x (in torr)

E. Shutdown
1. Turn Beam off
2. Set WD to 20
3. Hit Vent
4. Remove sample
5. **Get assistance to remove the low keV cone and to insert the BSE detector.**
6. Set the vacuum back to High Vacuum
7. Close door and select Pump
8. When light is green, select the Stop button to exit.