

Laboratory 4: Quanta 600F Environmental SEM - Low vacuum mode and charging

Sept. 26-30th, 2016

Due: 1 week from lab

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. Lincoln Head Penny (conductive)
2. Uncoated sandstone chip (non conductive)
3. Challenge Sample: Filter paper (if time remains for more experienced Quanta Users)

Objectives:

1. Proper startup and vacuum level checks
2. Specimen exchange
3. Alignment and working distance determination
4. Image capture
5. Changing vacuum modes
6. Visualizing secondary and backscattered electrons.
7. Finding optimal large field detector settings for secondary electrons in Low Vac Mode on a non-conductive sample.

Operating Conditions

1. **SEM:** 10 kV; Spot 3.0, Aperture #6 (30 μm) ; 12 mm WD.
2. **Electron Detectors:** Everhart-Thornley for secondary electrons (ETD/SE – biased at 250 Volts), Everhart-Thornley for backscattered electrons (biased at - 150 Volts) and Large Field Detector (LFD, SE) in low vacuum mode solid state diode for back-scattered electrons (BSED);.
3. **Imaging:** 1024 X 884; 10 μs dwell time

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Questions to be answered

1. Describe the differences in back-scattered signals seen with using the ETD biased (-150 Volts) versus with the solid-state diode back scattered detector set in Z-contrast versus topo modes. Why do these images look different? (3)
2. What are the vacuum levels associated with high, low and environmental modes? How are these vacuum levels achieved? (2)
3. What are at least two ways of imaging/compensating for a non-conductive sample? (2)
4. What is the difference between a single secondary electron image and an integrated multiple secondary electron image? (2)
5. Why must one stay below 70% contrast in low vacuum mode? (2)
6. Why does the electron signal (brightness in large field detector) increase when allowing more water vapor gas into the chamber? Please describe the phenomenon seen here, including the interaction of the primary beam with the water vapor gas and cite appropriate references. (4)
7. What happens when we reach "charge balance"? Please describe what happens at the beam specimen interface and the resulting image. Why do we wish to find this charge balance? What are the consequences if we image on either side of charge balance? (3)

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PART I. LINCOLN-HEAD PENNY

Operational Parameters

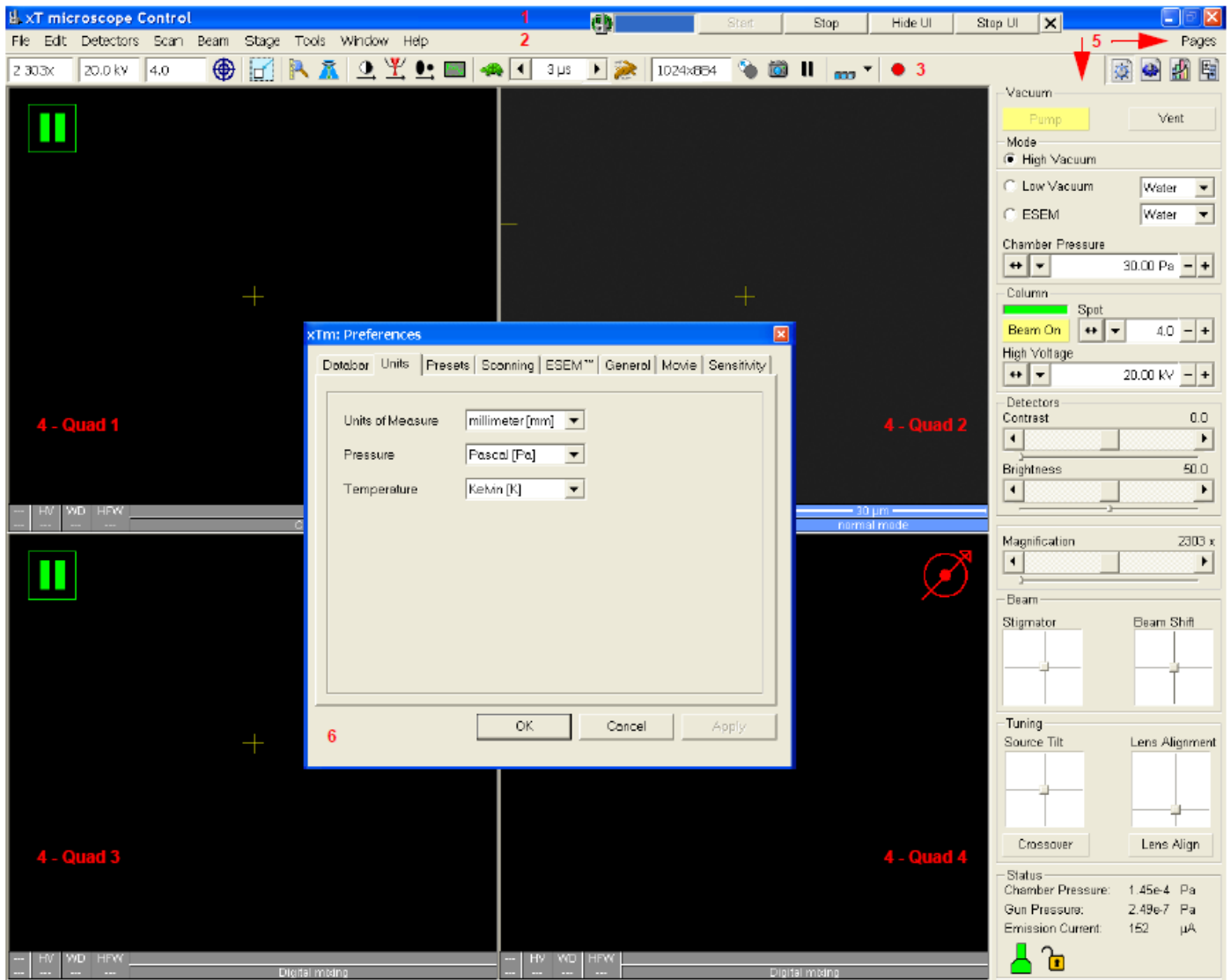
- **SEM:** 10 kV; Spot 3, Aperture 6 (30 micron); 12 mm WD, Everhart Thornley (ETD-SE & ETD-BSE), Solid State BSED (Z-contrast & topo) detectors
- **Imaging:** 1024 X 884 pixels, 10 μ s dwell time

Start-up

1. Log into FOM.
2. Middle and Right monitors should be on.
3. Select Quad 4 (chamber scope). Click anywhere inside the Quad 4 to select it.
 - Note: The information field will be highlighted Blue to indicate when a Quad is selected.
4. 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!**

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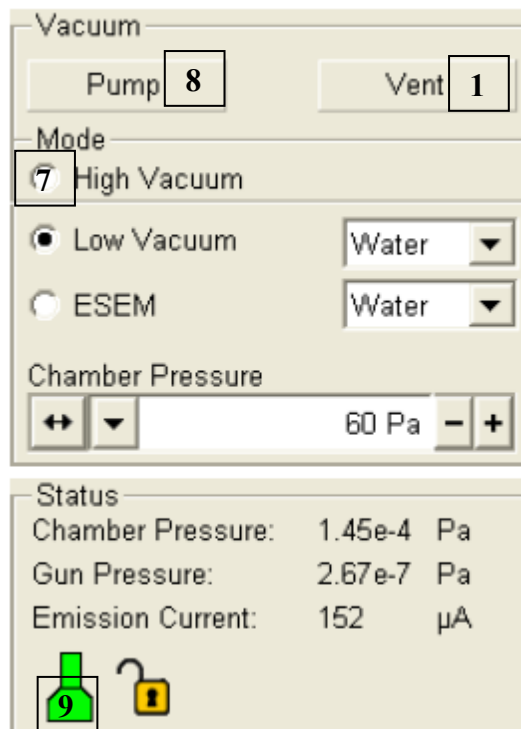
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Specimen exchange

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 LINCOLN-HEAD PENNY sample, secure with silver allen wrench (will not tighten like a normal screw, it slips).
5. Make sure Quad 4 is live (not paused).
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.

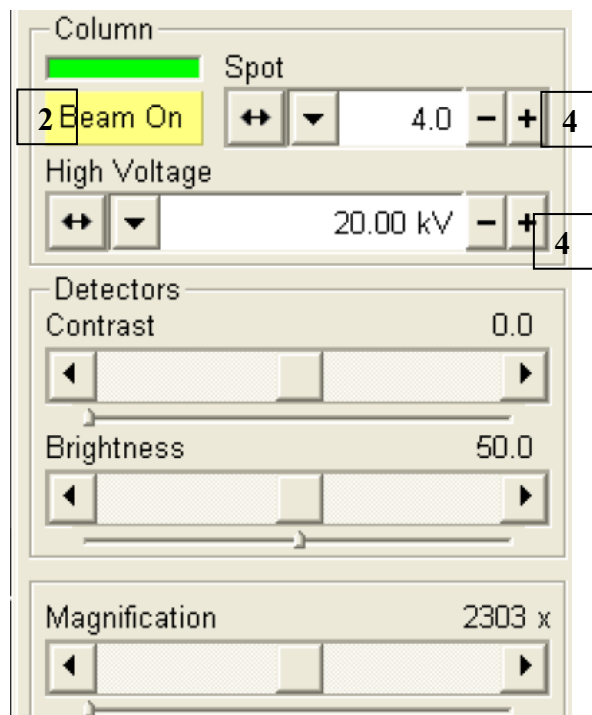
****** NOTE: If the door is closed to hard and fast, the resulting pressure could damage the thin Be window on the EDS detector!!**

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 $\sim 4 \times 10^{-4}$ torr, the chamber will go from orange to green. Also the BEAM ON button will enable.
 - a) Note: The gun & column should always be green. If not, please get help.



Initial Start-up

1. Under the STAGE tab, engage CENTER POSITION.
2. Click the BEAM ON button.
3. Under the SCAN tab, go to PREFERENCES
 - a. Go to Databar and ensure the data field contains:
 - i. High Voltage
 - ii. Horizontal Field Width
 - iii. Working Distance
 - iv. Detector Type
 - v. Pressure
 - b. Go to Presets
 - i. Add 200X to the MAGNIFICATION PRESETS.
4. Set HIGH VOLTAGE to 10 kV & SPOT to 3
5. Select upper left quad (Quad 1) to show ETD (SE) and upper right quad (Quad 2) to show BSED topo
 - a. Select quad and enable detector under DETECTOR SETTINGS tab
6. Unpause to make active.
7. 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 300 ns.
8. Set image resolution to 1024 x 884.



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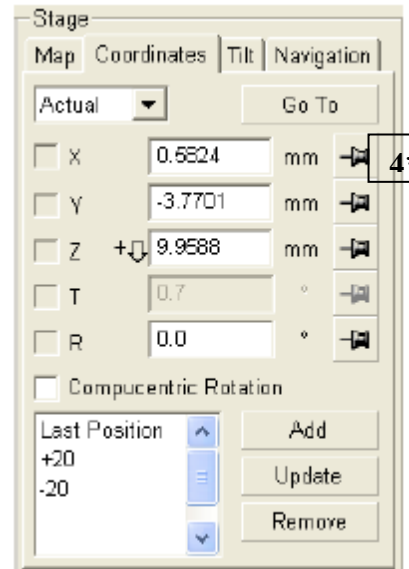
Acquire Image and Set Working Distance (“Link Stage”)



1. Obtain an SE image by unpausing Quad 1
2. Adjust brightness/contrast (can use knobs or autoBC), double click the Quad 1 image to navigate.
3. Increase magnification to 10Kx and refocus
4. Define Z-height by “**linking**” the Forward Working distance to the Stage Z under COORDINATES tab.

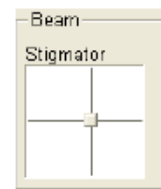
***** **Make sure the working distance read at bottom of active Quad matches the Z height coordinates (~60 mm)**

5. Decrease the magnification
6. Move the sample closer to the objective lens by setting the sample at WD of 25 mm.
 - a. Type 25 mm in Z coordinates.
 - b. Click on “Go To” button.
 - c. Hover mouse cursor over “Go To” which now says “Stop” and click if sample comes too close to stage
7. Increase magnification to 10Kx and refocus
8. Again, LINK the Forward Working distance to the Stage Z (see step 4).
9. Decrease the magnification
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 10Kx and refocus
12. LINK the Forward Working distance to the Stage Z (see step 4).
13. Adjust brightness and contrast.



Perform Alignments and Adjust Astigmatism

1. Zero Cartesian Coordinates (Stigmator, Lens Align, Crossover, Beam Shift) using right click on each coordinate and selecting “ZERO”
2. Align the lens.
 - a. Find a feature with sharp contrast (like a dust particle).
 - b. Go to high mag (~100Kx)
 - c. Enable LENS ALIGNMENT
 - d. Try to minimize movement
 - i. Note movement direction
 - ii. While holding down left mouse button, drag the cursor slowly in the direction of the movement
3. Correct for astigmatism (using Sigmator X&Y on hand panel).
4. Zoom down to ~100x and activate Quad 2 BSED
 - a. At bottom of Quad 2, should read BSED.
 - b. If not, go to DETECTORS tab and select BSED
5. Adjust brightness and contrast.



SE IMAGING A CONDUCTIVE SAMPLE IN HIGH VAC MODE

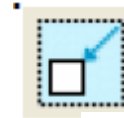
1. Go to 200X.
2. Navigate to middle of columns in Lincoln Memorial – find Abe sitting in his chair.
 - a. Holding middle mouse button and dragging allows you to navigate around.
 - b. Also double clicking a feature will place it in the center.

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3. Once you find Abe, center him in your image.
4. Zoom in 10Kx and select reduced area for focusing/astigmatism correction
5. Set Scan Rate to 10 μ s.
6. Adjust the brightness/contrast
7. Take first SE image – label it **Abe 200x ET SE**, save in a new folder (LastName_Lab4).
8. Set scan speed to 300 nanoseconds
9. DO NOT ADJUST brightness and contrast
10. Next to the PAUSE button is “SELECT FILTER MODE”
 - b) Select “INTEGRATE”
 - c) Select Number of Frames – “16”.
 - d) A warning box appears, just hit OK
11. Take 2nd SE image – label it **Abe 200x ET SE int**
12. Go back to “SELECT FILTER MODE”, and select Live

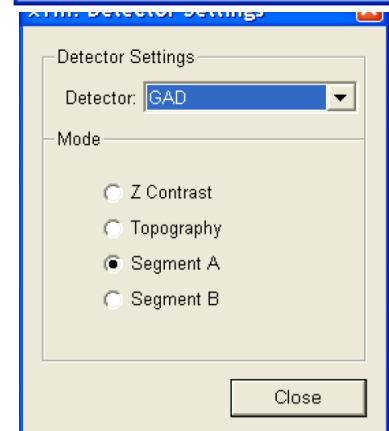
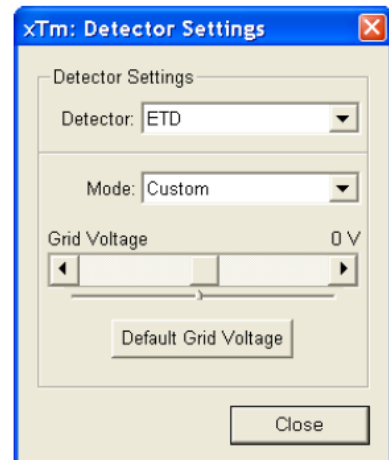


BSE IMAGING A CONDUCTIVE SAMPLE IN HIGH VAC MODE

1. Switch ET from SE to BSE in Quad 1.
2. Set scan speed to 10 μ s
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: **Abe 200x ET BSE**
 - b. Label Quad 2: **Abe 200x SS BSE topo**
7. Switch Quad 2 to BSE Z contrast mode (A+B)
8. Adjust brightness and contrast,
9. Take another image, **Abe 200x SS BSE Zcon**

Exchange Samples

1. Make sure Quad 4 is UNPAUSED to view the inside of the specimen chamber.
2. VENT the specimen Chamber
3. .
4. Put in the sand stone sample.
5. Go back to High Vacuum mode
6. Hit Pump



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PART II. SANDSTONE

Operational Parameters

- **SEM:** 10 kV; Spot 3, Aperture 6 (30 micron); 12 mm WD, Everhart Thornley (ETD-SE & ETD-BSE), Solid State BSED (Z-contrast & topo) detectors
- **Imaging:** 1024 X 884 pixels, 10 μ s dwell time

Repeat the procedure “Link stage” and find your sample of interest. Adjust the alignment as needed.

SE IMAGING A NON-CONDUCTIVE SAMPLE IN HIGH VAC MODE

1. Go to 200X.
2. Set Scan Rate to 10 μ s.
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”
 - e) Select “INTEGRATE”
 - f) Select Number of Frames – “64”.
 - g) A warning box appears, just hit OK
8. Take 2nd SE image – label it **sandstone 200x ET SE int**
9. Go back to “SELECT FILTER MODE”, and select Live



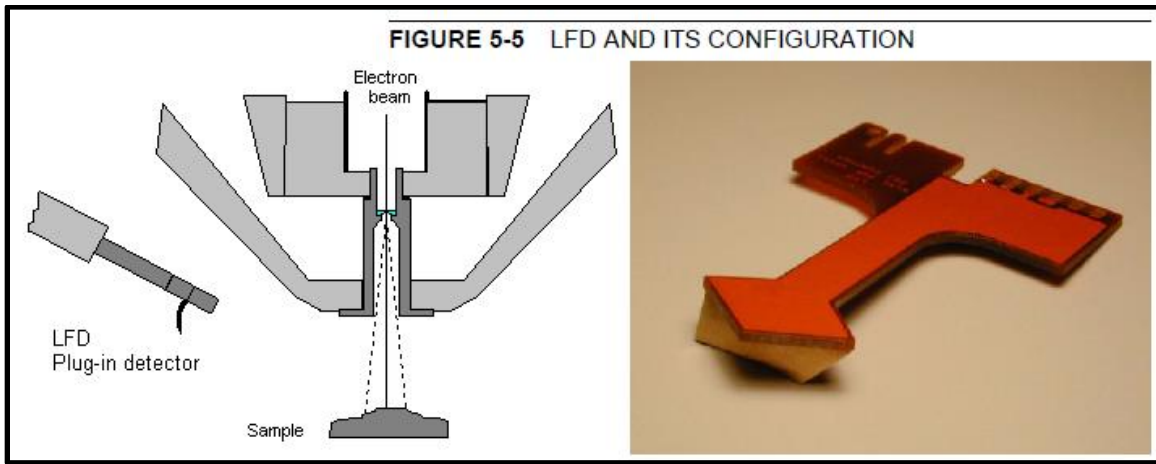
BSE IMAGING A NON-CONDUCTIVE SAMPLE IN HIGH VAC MODE

10. Switch ET from SE to BSE
11. Set scan speed to 10 μ s
12. Adjust brightness and contrast
13. Quad 2 should be active, BSE topo mode (A-B)
14. Adjust brightness and contrast
15. Take both images simultaneously.
 - a. Label Quad 1: **sandstone 200x ET BSE**
 - b. Label Quad 2: **sandstone 200x SS BSE topo**
16. Switch Quad 2 to BSE Z contrast mode (A+B)
17. Adjust brightness and contrast,
18. Take another image, **sandstone 200x SS BSE Zcon**

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IMAGING A NON-CONDUCTIVE SAMPLE IN LOW VAC MODE

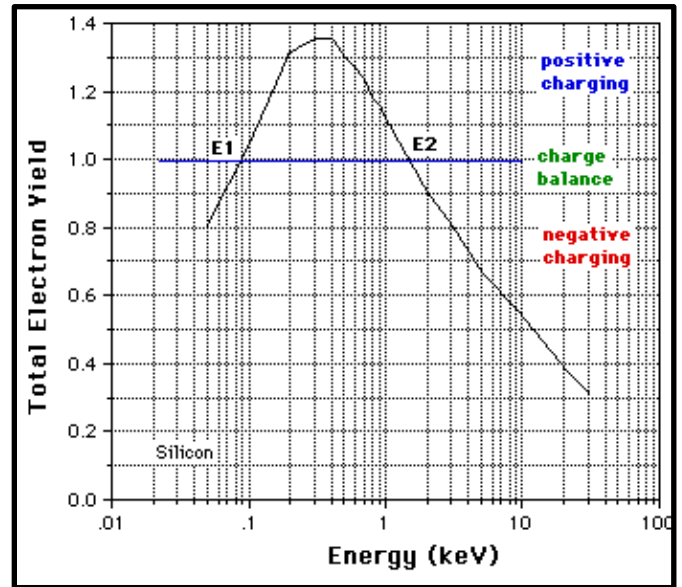
1. Turn the BEAM off by depressing the "BEAM ON" button
2. Set CHAMBER PRESSURE to 10 Pascals.
3. Select LOW VAC Mode
 - a. Select no Accessories and OK
 - b. Wait for the green light
 - c. Quad 1 should now be set to LFD (Large Field Detector) SE
4. Turn on BEAM
 - a. *******VERY IMPORTANT – SEE WARNING ON THE SEM CHAMBER DOOR**
 - b. Never go above 70% contrast with the LFD!
 - c. 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 90 % (Enhance mode digitally expands the contrast)
 - c. Set brightness accordingly.
6. Set Mag to 200x

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7. Set the scan rate to 10 μ s
8. Adjust the Enhance and brightness using the Videoscope.
9. Take another micrograph, **sandstone**
200x SS SE LFD 0-3
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 **charge balance**.
 - a. Turn Videoscope back on, ensure your signals is contained within in the lower half of the videoscope
 - b. Do not adjust contrast or enhance during these steps.
 - c. Increase the Chamber pressure by 10 Pascal increments (read the pressure at the bottom). Let equilibrate, by acquiring 3 10 microsecond scans
 - d. Lower brightness to where your signal is contained within the lower half of videoscope.



11. Continue to increase the pressure by 10 Pascal increments, adjusting the brightness until the brightness doesn't increase or decrease.
12. Take another micrograph, **sandstone 200x SS SE LFD xPa** (in Pascal)
13. 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. .
4. Put in the filter paper sample
5. Go back to High Vacuum mode
6. Hit Pump

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PART III. 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 μ s
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
 - a) Why is it jumping around?
11. Under select filter mode, do another integration
 - a) Set scan rate to 100 ns!
12. Save image as paper 10Kx HV SE int
13. Go back to Live in the Filter Mode.
14. Turn Beam off

D. Imaging at low vacuum and low keV

1. Set CHAMBER PRESSURE to 26 Pascal.
2. Select LOW VAC Mode
 - a) This time, set accessories to Low KeV cone. (Do we need to use the low Kv cone if the vacuum in the lower IGP doesn't deteriorate?)
3. Wait for the green light

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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 40 Pa(read the pressure at the bottom)
 - d) Lower brightness back to where it was.
 - e) Continue to increase the pressure by 10 Pascal increments, 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 xPa (in Pascal)

E. Shutdown

1. Turn Beam off
2. Hit Vent
3. Remove sample
4. **Get assistance to remove the low keV cone and to insert the BSE detector.**
5. Set the vacuum back to High Vacuum
6. Close door and select Pump