SEM : Quanta FEI

- Log into computer
- Open xT microscope server
  - Wait for the three buttons to turn green
  - Hit "Start"
- Log-into microscope with same log-in as the computer
  - Home stage
  - Note any error message and clear message screen
  - Minimize server window by double clicking in blue bar

General Notes:

- Vacuum Status:
- 

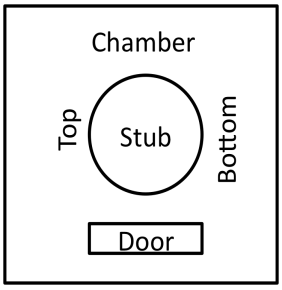
Green: PUMPED to the desired vacuum mode
Orange: TRANSITION between two vacuum modes (pumping / venting / purging)
Grey: VENTED for sample or detector exchange
- Short cut keys:
  - +/- = change magnification
  - * = rounds off magnification to even number
  - F2 = takes picture
  - F3 = videoscope
  - F5 = maximizes/minimizes window
  - F7 = reduced area focus
  - Left click = focus
  - Left click + shift = stigmate
  - ESC = cancel button, will stop whatever the active command is
- Saving and retrieving files
  - Save all files on the left computer in your user account
  - Do not put anything in the right, microscope computer as this will void the warranty
- On first start up – go to tools → preferences → general → lower z upon vent → data bar → date → move to top

General order of operations for the SEM

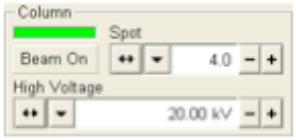
1. Insert sample/pump down
2. Rough focus samples
   - Link to z
   - Align microscope
3. Map samples
4. Fine focus and image
   - Fine stigmata each image
5. Return all settings to normal and remove sample

SEM Operation

1. **Load sample**
   - always wear gloves when touching anything to go into the chamber
   - "Vent"
     - "Yes" – you want to vent
   - Insert sample, and screw down
   - Hold door shut and "Pump"
   - Wait for icon to turn completely green
   - Change the sample height
     - Press and hold the scroll button and move the mouse up to move the stage to ~10 mm. (for a flat sample on the standard sample 25 mm stub = 55 mm on z)
     - After your initial focus and z-height alignment, it can be moved closer



2. **Rough Focus and Alignment**
   - Good conditions to start = 10 kV and spot size = 1-2
   - Click on window that you want to view samples (bar will turn blue) and unpause ( )
   - "Beam On"



   - Rough focus the image (should be around WD = 10mm)
   - Link Z ( ) →
     - Tells the instrument where the stage height actually is.
     - Re-link Z often during imaging
   - Lens Align
     - This should be done at a mag greater than your normal working mag
     - Want this to blink in and out, not move up and down/side to side
   - Crossover
     - Spot size should be centered over the X
     - We do not have control over this feature, but it should be checked after changing spot size or kV

3. **Map Samples**

4. **Fine focus and Imaging**
   - After focusing sample and re-linking z, can move sample closer to gun for higher res imaging
   - Sigmation = shift and right click
     - "Focus" removes the direction
     - "Stigmate" sharpens image

- Think of focus and stigmata as working together: focus then stigmate then refocus and restigmate until you are happy with the quality of the image
- Generally, your shouldn't have to make significant changes in the stigmation

- To use reduced area screen use: 
- To move image, press and hold scroll button and move mouse the direction you want to go
- Double click will center object that you double clicked
- Scroll Brightness and Contrast buttons to adjust/use videoscope () to aid
- Use reduced area to fine focus and stigmata 
- To take picture, hit  or F2 and save files when prompted in your folder
- Use () to change the scan speed or the arrows

5. **Return all settings to normal and remove sample**
   - "Beam On" = turn beam off (wait for valve release)
   - Vent
   - Remove sample and any accessory, return to high vacuum mode if necessary
   - "Pump" – wait for () to turn green
   - Log out of microscope screen
   - File → Exit out of microscope screen
   - If no one else is coming:
     i. Turn off server → "Stop" → wait for blue bar to go away
     ii. Exit out of server (right click on blue bar)
     iii. Log off computer

- **Low vac mode:**
  - Vent
  - Install low vac accessory on the end of gun
    - it will gently push in like a button
    - use CCD to verify that it is pushed in evenly
  - Change to "low vac"
  - Change chamber pressure to 0.6 to 0.68 Torr to start
  - Pump
  - Choose "low vac" accessory option
  - Vacuum will pump, then purge, then pump again before () turns green
  - Can change the vacuum pressure during imaging to optimize
  - Start at 8 kV

Sputter Coater

1. Turn on gas tank
2. Turn on sputter coater (switch on back)
3. → System start
4. → Screens
5. → Manual Sputter set up
    a. → Turn on mechanical pump (flip valve on pump to make sure it is on)
        Wait until pressure is down to 40 mTorr
    b. →Open gas valve
        Adjust gas pressure to 50-70 mTorr using valve
6. → Set point
    Should be 35-40
7. Turn on thickness monitor
8. →Rotation on
9. →Power on
    Let run until desired thickness reached (no more than 10 minutes)
10. →Power off
11. → close gas valve
12. → turn off mechanical pump
    Turn valve to closed
13. Turn off gas
14. Turn off sputter coater
15. Turn off thickness monitor

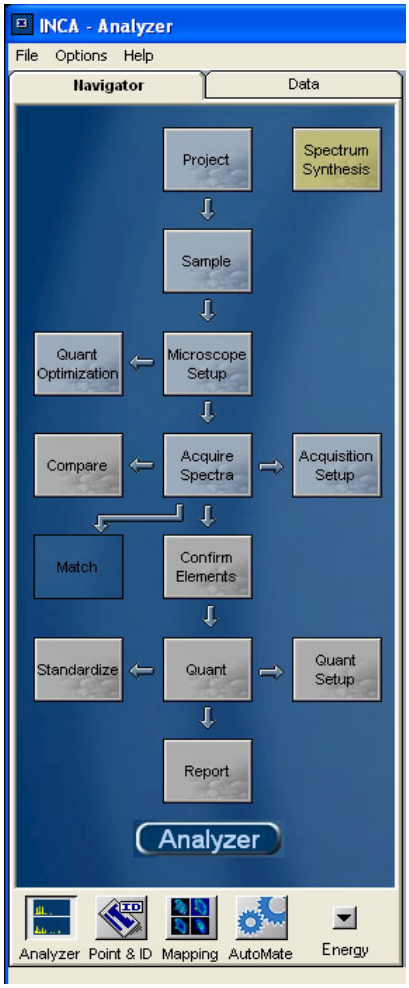
Oxford INCA EDX

***Use uncoated Samples for EDX

1. Start the SEM as normal
   - 10 mm is the optimum working distance for the geometry of the detector during EDX measurements
   - Use a KV of 2x that of the highest energy you hope to see (found on slide rule or mouse pad by the computer)
   - Start with a spot size of ~4
2. Focus and stigmata on your sample at this conditions
3. Pause the CCD camera
4. Open the INCA program on the support computer
   - INCA can be operated under three different areas. Each area has its own flow chart to follow during analysis

   (1) Analyzer – gathers information from the whole field of view
   (2) Point and ID – can choose where you want to get information
   (3) Mapping – can map the spectra
5. Image the area of interest
6. Log off SEM as usual and close INCA program

** Analyzer **



➔ Project
- Name project or start a new project by choosing the thumb tack icon

➔ Sample
- Name your sample or start a new sample by choosing the thumb tack icon
- Each project can have multiple samples

➔ Microscope Set-Up
- Run the cycler
- Input rate > 2k. This can be adjusted by increasing the spot size
- Dead time should be <40%
- Choose a process time between 1-6. 6 is the longest process time and will give the highest resolution; 1 is the shortest and will have the lowest resolution. 4-5 is a good place to start. This can be adjusted to influence the dead time

➔ Acquire Spectra
- Each sample can have multiple points of interest
- Choose an appropriate acquisition time and acquisition time and kV range

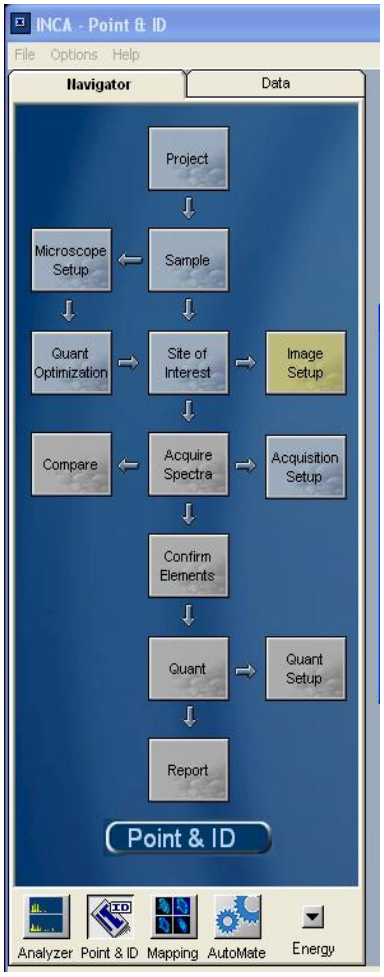
➔ Confirm Elements
- Confirm the elements that many be present in your Sample

➔ Quant
- Identify the correct elements

→ Quant set-up
- "Transfer" the elements from your spectra or choose the element of interest from the drop down menu

➔ Report
- Choose report format you want, export, and save the report as a word document
- Repeat for each spectra

**Point and ID **



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➔ Site of Interest
→Image Set-up
- Adjust resolution
- Focus on your sample area under the microscope
- Record image on INCA (green circle)

➔ Acquire Spectra
- Each sample can have multiple points of interest
- Choose an appropriate acquisition time and acquisition time and kV range

➔ Confirm Elements
- Confirm the elements that many be present in your sample

➔ Quant
- Identify the correct elements
  → Quant set-up

- "Transfer" the elements from your spectra or choose the element of interest from the drop down menu

➔ Report
- Choose report format you want, export, and save the report as a word document
- Repeat for each spectra