Environmental Scanning Electron Microscope

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Sample preparation:

1. **Powdered sample:**

    - Place the carbon tape on the stub.
    - Spread the powdered sample on butter paper.
    - Invert the carbon tape on butter paper and allow the sample to stick.
    - **Blow the excess sample with the blower**.

2. **Cross sectional sample:**

    - With the help of forceps hold the sample in between the screws.
    - Tighten the screws with the help of screwdriver.
    - Make sure that the **sample is not moving** and in position before placing the sample inside the system.

3. **Block samples/ Powdered block samples:**

    - If powdered block samples are to be observed, they may not stick to the carbon tape as loose powder will keep coming out.
    - Stick such a sample on a small piece of **aluminum foil** with the help of fevistick.
    - Later treat this as a whole sample and **stick the aluminum foil** (with the sample) **on carbon tape**.
    - **Make the sample surface conducting by placing a small carbon tape from top of the sample to the stub.**

4. **Odd samples:**

    - Those samples that do not stick at all, are odd in shape can be stuck on the stub with the help of carbon tape.
    - Stick the carbon tape on the stub.
    - **Cut another small piece of carbon tape and make it circular.**
    - Place this on the stub. This will look like a well.
    - Place the sample inside this and stick the carbon tape properly.

Note:

- **Please make sure that the sample is sticking properly and then only load inside the system.**

Coating the samples:

Auto mode:

1. Place the sample inside the coater.
2. Switch **ON the coater** from below. It will show 000.
3. Select the desired **mA**. It can only be 10 or 20.
4. **Press the button near Pa to switch over to seconds.**
5. With the help of up n down keys select the desired seconds.
6. Press again the button to switch over and see the vacuum.
7. Once the Vacuum is acquired and it goes **below 20 a beep sound will be heard**.
8. Press **start in the auto mode.**
9. This **start will keep on blinking till coating is done**.
10. Vacuum will go till 5 and then break. Pa will show Vacuum 000. Vacuum will be regained. **This process will be repeated 2-3 times**.
11. All this time the start will keep on blinking.
12. Once the desired pressure is acquired **plasma** will be released (blue/purple haze). **Start button will be red**. Count down of seconds can be seen on screen.
13. Once coating is done, start will not show any LED **(it will stop blinking).**
14. Turn off the coater from below.
15. Let the vacuum get released and remove the samples.

Manual coating:

- Samples with moisture or block samples may give problem with coating in auto mode. Vacuum in the auto mode does not go down easily and sample coating may take a longer while. For this we can go for the manual mode of coating.

- Initially the sample is placed in auto mode only. If it takes too long then follow the below.

- U have already selected the desired mA and seconds, pressed start in the auto mode and Vacuum is moving very slowly then:

    1. Press **stop in the auto mode.**

    2. The start button will stop blinking.

    3. Press the **mode button to switch over to the manual mode**

    4. Press **control gas.**

    5. **Vacuum** will be released can be seen as **000.**

    6. Vacuum will be acquired again. This Vacuum will move very slowly and reach a point from where it will not move quickly.

    7. Now press **start in the manual mode**.

    8. Plasma will be released and coating will be done.

    9. Once coating is done, turn off the coater let the vacuum release and remove the samples.

Scan rotation/ Compucentric rotation:

Compucentric rotation:

- Compucentric rotation is done to rotate the entire stage.
- Compucentric rotation **should not be done**.
- Press **only F12** for this option.

Scan rotation:

- If the sample has vertical or horizontal features then u can adjust the sample with the help of scan rotation.
- Press **CTRL+ F12**.
- A scan rotation circle will appear.
- **Make sure that scan rotation is written on top.**
- Adjust the arrow and rotate the sample.
- Make the **scan rotation zero before going to next sample.**

Turning the microscope OFF:

- System may not communicate properly due to many reasons. You may observe that the system is hanged and not taking commands.
- **This may occur with the EDX Digital Controller is turned ON.**
- **U may observe that the stage is not moving and Z axis is not moving.**
- U may have to turn OFF the microscope and restart it.

Kindly do the following:

1. **Vent the system** or make sure N2 pipe is connected to the system.
2. Minimize the XT microscope control / User interface
3. U will observe XT microscope server.
4. Click on **Stop UI.**
5. User interface will shut down, **UI state: Stopped.**
6. Click **stop.**
7. All the **bbox items will turn from green to black**.
8. Server state : stopped
9. Once the server state is stopped, **Gun & vacuum, optics and motion will be black. Imaging will always be green.**
10. Click on **shut down**.
11. It will ask: Do u really want to shut down? Click **yes**
12. **Green LED light of the microscope will be turned OFF**.
13. Right click where XT microscope server is written(Purple blue line)
14. Click on **Exit XT microscope server**
15. IT will ask: Do u really want to exit: click **yes.**
16. Now turn off the PC as usual.
17. U will have to turn off the support PC also.

Turning ON the microscope:

1. Turn **on the microscope**. (Green LED will glow)
2. Turn **ON the server and support PC.**
3. Passwords for both the PC:

   Login: supervisor
   Password: supervisor or **Login: support Password: support**

4. Click on **XT microscope server**
5. Once **Gun & vacuum, optics, motion, Imaging are green then press start**.
6. It will **automatically show the user interface.**
7. **Login/ Password: supervisor, supervisor or support, support**
8. System will ask to do a **home stage rotation**.
9. Remove the pause on the stage screen, open the microscope door and click yes.
10. Once the home stage is done system is ready for analysis.

Imaging:

- Make sure that the sample is sticking properly.
- Adjust the Working distance (WD) of the sample according to sample height.
- If the sample requires **greater depth of field** keep the **WD greater** than 12mm for better focusing.
- For taking **EDAX** make sure that the sample surface is at 10mm-12mm WD. Lesser than 10mm may give error.
- **Digital contrast /Brightness:** can be reduced or increase by 1 or 2 points only. If greater/lesser values are used will create noise.

Low vacuum mode:

- Going from High Vacuum mode to Low Vacuum mode turn off the HV before changing the mode.
- In low vacuum mode adjust **enhance to 65 and below**. It will create noise above that.
- On **changing spot size enhance will change**. Adjust accordingly.

ESEM mode:

- Make sure that the **stage is in centre position.**
- **Do not do home rotation once the pettier stage is attached.**
- Make sure that u have selected the correct pressure level using the slider bar. **(above 600pa)**
- Before pumping go to **Tools- Preferences-NO PURGE. Click OK**.

Back scattred mode:

- For changing from ETD to Back scattered mode or low vacuum to back scattered mode. Go to – **Detector- Solid state BSE.**
- **Changing of spot size may result in higher contrast values**. Adjust accordingly.
- Slow scan in this mode will give sharper images.

Note:

- **Never leave the system Pumped in Low vacuum or ESEM mode**.

EDAX: Normal EDAX, EDAX with Image, Maps/Line

For taking Normal EDAX:

Please check the following:

- Switch On the EDAX digital controller from behind.
- Kindly check that the EDAX detector is cooled. (**Bias 1-green , power-green**)
- If Bias 1 is red ---then the detector is not cooled.
- If the support PC is switched On from before hand then the EDAX controller will not communicate. And will give the **error message "Failed to initialize HW COM object " "Security key missing or not detected"**
  Kindly **restart the support PC** for connecting with the EDAX digital controller.
- On doing this u may observe in the user interface that the **stage is not moving**. (vertical z axis is locked). **Kindly restart the entire microscope**.

Working:

1. Sample should be at **10mm working distance**. If its greater than 12mm or less than 10mm (9.7mm) then u will not get the correct Dead time(DT%) and Counts per second(CPS).

2. The **CPS** should be around **900-1200. DT% below 40**

3. Please check the following or change to:

   Analyzer: Det 1
   Preset 50
   Live
   Amp time:100us

4. On clicking peak ID if **no peak identification** is done, it means that the system is not communicating. Kindly **restart the support PC**.

5. Kindly delete the background.

6. Save the quant in word doc.

7. Kindly confirm the sample folder before saving.

8. Save the graph in **.spc and .bmp format**.

For taking EDAX with Image:

Please check the following:

- Switch On the EDAX digital controller from behind.
- Kindly check that the EDAX detector is cooled. (**Bias 1-green , power-green**)
- If Bias 1 is red ---then the detector is not cooled.
- If the support PC is switched On from before hand then the EDAX controller will not communicate. And will give the **error message "Failed to initialize HW COM object " "Security key missing or not detected"**
  Kindly **restart the support PC** for connecting with the EDAX digital controller.
- On doing this u may observe in the user interface that the **stage is not moving**. (vertical z axis is locked). **Kindly restart the entire microscope**.

Kindly note that:

- **High magnification** images will **not be saved** in this format as we require changing the spot size to 3.5 and below. Thus a very poor quality image will be saved.
- **Request the student to save the image separately and take a normal EDAX.**
- For taking EDAX we require **spot size 4.5 and above.**
- Kindly note while **adjusting the DT% and CPS** changing the spot size may result in **slight shift of the sample region.**
- Kindly collect the image after adjusting the DT% and CPS or **recapture the image**.

Working:

1. Sample should be at **10mm working distance**. If its greater than 12mm or less than 10mm (9.7mm) then u will not get the correct Dead time(DT%) and Counts per second(CPS).

2. The **CPS** should be around **900-1200. DT% below 40**

3. Please check the following or change to:

   Analyzer: Det 1
   Preset 50
   Live
   Amp time:100us

4. Click on **Image option**. (Spectrum/image/maps/line)

5. Kindly put the **Scan mode on EXTERNAL**.

6. Click on collect Image. (If u get an image with error –check the scan mode)

7. **Auto LUT**- on the right hand can be used to change the contrast on the acquired image.

8. U can select the **entire area or selected area, spot, arbitrary**- according to the student's choice.

9. **To delete the earlier graph**-- Below the image – click on the second option. The graph will go but the elements will remain. There is no option to remove this.

10. To collect the graph- check the DT% and CPS and click on the first option below the image.

11. The **Lsec** in this case will **stop before** reaching **50**. **Its ok**.

12. **There is no option for deletion of background**.

13. Kindly click on peak ID and make the changes according to the sample.

14. For the **quant** reading click on **"Q" below** the image. A box with quant will appear near the image.

For taking Maps:

- Kindly follow the instructions and **take EDAX with image**.
- Note that very **high magnification** mapping data is **not possible** as the change in spot size will result in poor image quality at high magnification.

Working:

1. Kindly follow the instructions and **take EDAX with image first.** To define the elements.

2. Click on **Maps/line option**. (Spectrum/image/maps/line)

3. Click on the **multiple grid option**.

4. Note that: Maps will be collected on the identified peaks. If any changes are to be done can be **changed by PEAK ID**.

5. If u want to **change** the frames. **Right click on frames**. And then select the other options.

6. After all the above setting select : **Collect Maps**.

7. Kindly select the folder and the name for the maps.

8. These maps will be **save automatically**.

9. The mapping process will start with a **black background**. To **change** the color to **white- Go to DISPLAY –REVERSE PELLET.**

10. During this time the **DT% will keep on changing. ITS OK**.

11. Please check that the on going frame/no of frames **(2/32, 16/32…)** at the base.

12. Once the mapping is done 32/32 frames. **The data will be displayed in a different format**. Kindly wait till all frames are done.

13. For doing **overlay** of the data acquired: click again on **multiple grid option**.

14. On doing so **only image** will be displayed in the **first quadrant**. All the other **quadrants will appear white. Its OK. Data is not lost.**

15. Next select the Image and the desired quadrant with **“CTRL+ click**”. U can choose multiple quadrants also.

16. After selection go to **PROCESS--COLOR --Substitution overlay.**

17. Overlay will be displayed in new quadrant. This overlay is saved automatically.

For taking Line analysis:

- Kindly follow the instructions and **take EDAX with image**.
- Note that very **high magnification** line data is **not possible** as the change in spot size will result in poor image quality at high magnification.

Working:

1. Kindly follow the instructions and **take EDAX with image first.** To define the elements.

2. Click on **Maps/line option**. (Spectrum/image/maps/line)

3. Click on the **Single grid option**.

4. Note that: Line analysis will be done on the earlier identified peaks. If any changes are to be done can be **changed by PEAK ID**.

5. Below Maps option please select the option of **diagonal red line with arrow**.

6. Draw a desired line on the image.

7. Depending upon the length of the line the points will change. (seen above the dwell ms)

8. Longer is the line higher are the points.

9. After this click on **collect line**.

10. Kindly choose the folder to save the data.

11. This data will be saved in **word format.**

12. Click **YES** on **Line scan collection**.

13. A small green cross will move across the drawn line.

14. The **image** with the drawn line and the **word sheet** is **automatically saved**.

15. Below the maps option there is another option **red,green graph with diagonal white line.**

16. Click on red, green graph with diagonal white line option. **Line scan ROI count** will appear with colourful graph.

Note that:

- **If this window is not maximized then a pixilated image will be saved.**

Filament Blown:

- If the filament has completed certain hours it may be blown (mostly 80 hrs).
- U will observe that **Emission current is Zero-0**

- **Filament current – Blown?**
- For any change in the voltage there is no change in the above.
- Kindly contact the person incharge and inform.
- **Turn OFF the HV as usual at 500v.**
- Remove the samples as usual
- Pump the system in High vacuum mode.

Leaving the system for next day:

1. A/C Switch over
2. Turn the power switch OFF for the other A/C
3. N2 gas cylinder closed , loosen the cork on regulator
4. N2 gas pipe disconnected from the microscope
5. EDX Digital Controller OFF
6. Support PC (EDAX PC) shut down.
7. Stage in centre position and taken below
8. System pumped in High vacuum mode.
9. Internet PC shut down.

Change of Password:

- Passwords need to be changed after a certain time.
- System will ask that: **password expires in certain days**. Do u want to change it?
- Say **yes**.
- Login n password remain same: **supervisor n supervisor**

Wehnelt cleaning:

- After removing the blown wehnelt place the wehnelt on the ceramic base.
- With the help of the black round screwdriver loosen the screws.
- Rotate the wehnelt cap and remove the cap.
- U will observe that the cap has turned black-blue.
- With the help of the silver plate like tool unscrew the ring.
- Remove the used filament and discard it.
- Pack the base of the wehnelt in aluminum foil and keep aside.
- With the help of cif, cotton buds and tooth picks clean the filament cap and the ring.
- Clean the cap hole properly.
- Make sure that no cif is sticking on the ring threads. (as it may damage the ring threads later)
- Sonicate the cap and ring in beaker. **Do not cover with aluminum foil**.

    1. 15 mins- Acetone
    2. 15 mins –acetone
    3. 15 mins IPA

- Total time: **45 mins**.

- Dry the cap and ring under IR for less than 5 mins and then do the filament adjustment.

Adjusting the filament in the wehnelt:

- Place the wehnelt base on the ceramic base.
- Place a new filament in the wehnelt.
- **Match the notch on the filament and the wehnelt**.

- Place the ring on the filament and with the help of silver plate tool tighten the ring.
- Match the **hole on the wehnelt base and cap**. Place the cap on and **rotate to lock.**
- Observe the filament under the microscope and do the centering. Tighten the screws with the help of black round screwdriver.
- **Adjust the height** of the filament. The triangular arrow on the filament base should be near the base ring.

Changing the Wehnelt:

1. **Vent** the system.
2. **Open the gun cap**.
3. Message will appear on the **server screen gun cap open**.
4. Remove the earlier wehnelt.
5. **Match the notch (with dot) on the wehnelt with the notch in the gun cap.**
6. Close the gun cap.
7. Say clear and hide to the messages on server screen.
8. Click on **wehnelt changed**.
9. Place the **standard sample** in the system and pump the system in **high vacuum mode.**
10. Click on **Reset timer**.
11. Change the **filament voltage**. Slide the voltage bar and make it **0.00V**
12. Select **HV-- 20kV**. There will be no change in the emission current as HV is not ON. 20kV is selected as it is a in between kV.(10kV-30kV)
13. **On HV**.
14. Now observe that-
    - **Store is highlighted green.**
    - **Emission current --0mA**

- **Filament current --0.1 A or 0.0A**

15. **Limit voltage is ticked and 0.00V**

16. Select quadrant 1 and remove pause.

17. Press F5

18. **Remove the tick on limit voltage**

19. Make **contrast 50 brightness less than 45**. (so that we get good contrast and brightness later while taking images)

20. **Autobias is always ticked on.**

21. Click on **crossover to observe the crossover.**

22. Now slowly **increase the limit voltage.**

23. Filament current will keep on increasing. There will be **no change** in **emission current**.

24. Once **filament current** is above **2.00A change in emission current** will be observed.

25. Adjust the contrast to observe the crossover.

26. The filament needs to be **saturated**. So keep on **increasing the limit voltage** and adjust mostly with **change in contrast**. If any **blank black spaces** are observed it means that the filament is **not saturated**.

27. **Emission current at 20kV will reach above 100** and **limit Voltage** will be at **around 1.90V**.

28. Mostly all the filaments get **saturated above 1.90V and below 2.10V**. Keep checking with **change in contrast for any blank black spaces (filament not saturated)**.

29. Once the filament is saturated. **Click on limit voltage.**

30. Click on **store**.

31. Check the **autobias value**.

    - If this value is **above 120** then the filament is placed far away in the wehnelt such a alignment will not give good contrast n brightness to the images.

- **Filament height needs to be reduced**
- **Remove the wehnelt and adjust the filament height (reduce the height)**
- U may have to center the filament again.

32. If it is observed that the **crossover for the filament is cutting on the edges** then filament is not in centre.

    - Use the **accessories (Kaan)** to adjust the crossover and see a **complete elliptical shape crossover.**
    - If the accessories (kaan) are moved too quickly then **vacuum may break.**
    - System error: vacuum pressure too high.
    - Wait till the **vacuum status is ready**.
    - Start from 500v slowly going to 20kV.
    - At 20kV see the crossover and adjust slowly with the accessories (Kaan) to see the complete crossover.

33. **Click** again on **store**.

34. **Click on crossover to observe the standard sample** (glass microspheres coated sample).

Alignment for the filament:

1. **Click on crossover to observe the standard sample** (glass microspheres coated sample).

2. Place the sample at **10 mm WD**.

3. Do auto contrast brightness. And focus the sample.

4. Go to 3rd page (Option). **Stigmator-- right click. Make stigmator zero.**

5. Now go to the alignment page.

6. **1st: Tetrode alignment**.

    - At each kv- click on xover- bring in centre and store.

7. **2nd: Gun alignment**

    - Keep magnification up to **100X**.
    - Change the **resolution 512X442**.
    - Image will not appear good (pixilated). But the adjustment will be done faster.
    - **Scan speed – fastest**.

8. **3rd: Condenser alignment**

    - Change the **resolution back to 2048X1768**
    - **Stigamator should be zero**
    - **Bring recognizable feature in the centre (+).**

9. **4th: Final lens alignment:**

    - Change the resolution to **1024X884**

- Press start.
- Click on **AVERAGE option** (Blue squares) and select number of **frames 32.**
- Keep the **magnification low**.
- Rotating spheres will be observed and centre with non rotating spheres.
- **Adjust the image with non rotating spheres**. (finding the centre of non rotating spheres)

10. **5th : Stigmator alignment**:

- Change the resolution to **2048X1768**
- Click on **LIVE option** (Blue squares)
- Make sure that the **sitgmator is in centre** on options page.
- Before pressing start **adjust the focusing**.
- **While doing the stigmator alignment do not do focusing**.
- Magnification should be **above 4000X**

Water level for Low Vacuum Mode:

- Kindly check the water level for Low Vacuum mode and ESEM mode **each month.**
- Water level should **not be below black line.** Then water needs to be changed.
- **System should be vented.**
- Remove the clay from crock.
- Remove the crock and **remove all the water**.
- Add the fresh distilled water. **Level should be above the yellow label**.
- Fix the crock and cover all the edges with clay to avoid enter of air.
- Place the flask back in position.
