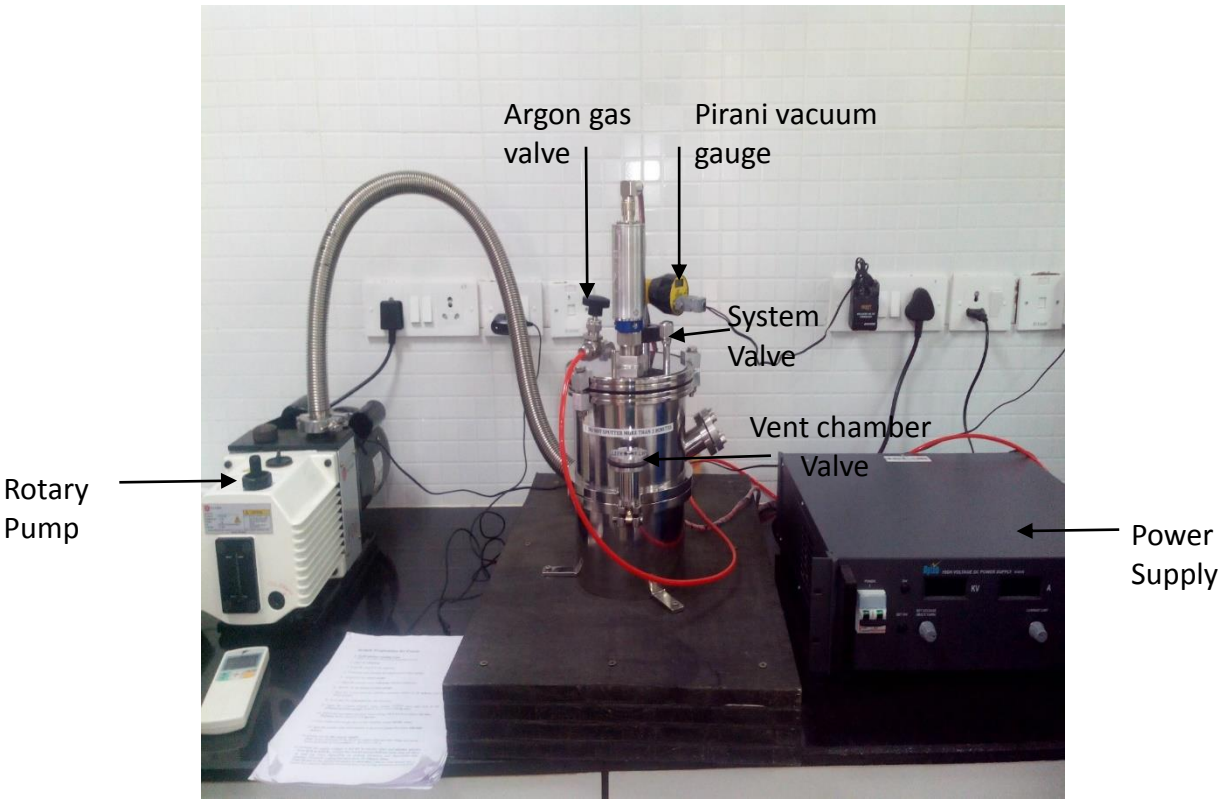
Sample Preparation for FEG-SEM

➢ Gold sputter coating Unit

(**Note:** It is mostly used for non-conducting samples)



1. Open the **chamber.**
2. Load the sample in the chamber.
3. Closed the vent chamber & system (steel) valve tightly.
4. Switch ON the **rotary pump.**
5. Open the vacuum valve (**chamber valve**) completely.
6. Switch ON the **pirani vacuum gauge.**
7. Wait for 5 min until the chamber pressure comes to **20 mTorr.** (see the pirani gauge)
8. Now pass the **Argon gas** into the chamber.

9. Open the cylinder primary valve (using cylinder key) and look at the **cylinder pressure gauge**, refill it if it is below **10 kg/cm2**.

10. Adjust the secondary pressure valve (using black knob) to adjust **the line Pressure to** the value of **1.5 kg/cm2**.

11. Now control the Ar gas flow in the chamber using **needle valve**.

12. Open the needle valve till pressure in the pirani gauge becomes **450-500 mTorr**.

13. Switch ON the **DC power supply**.
(**Note:** Before switching ON the DC power supply make sure that voltage and current Knobs are at **zero** (or min) **position** i.e. @0.07kV, 0.00 A

14. Increase the supply voltage to **0.4 kV** in smaller steps and **plasma** appears from **0.24 to 0.34 kv**, will get the current around **0.01A**at same time we have to start our timer depending on coating thickness and deposition rate required. Normally coating has been done for **15secto 20sec**.
(**Note: Do not** use the chamber and sputter for **more than 3 min** at a time because due to current flow, chamber target gets heated and there is no cooling mechanism incorporated)

15. After completion of the sputtering process, minimize the supply voltage and Current.

16. Switch OFF the DC power supply.

17. Close the needle valve of Ar gas.

18. Close the vacuum chamber valve.

19. Open the system steel valve.(To release the sample chamber pressure)

20. Open the vent chamber valve.

21. Open the chamber and unload the samples.

22. Close the chamber.

23. Close the pirani gauge power supply.

24. Close the rotary pump.

25. At the end close the cylinder pressure valves. (Secondary and primary)

➢ Sample holders Used

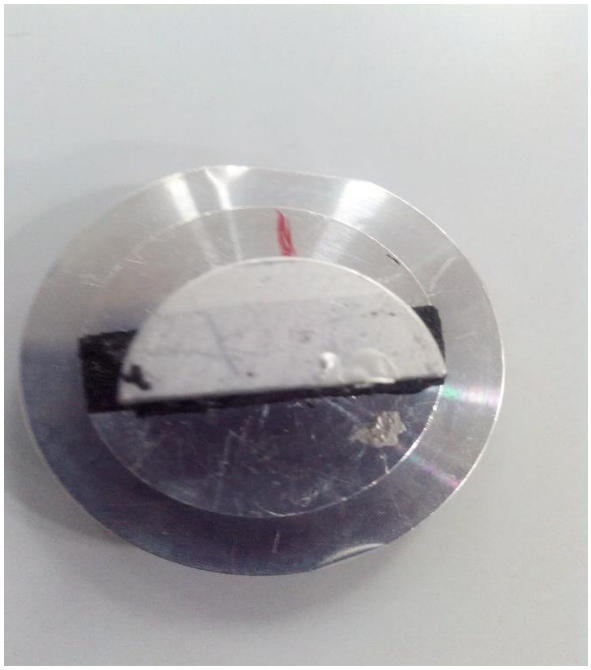
1. Surface/tilt view: sample holder has facility to mount 9 stubs of diameter 1 cm.

Carousel (9x9)

2. Cross-sectional view
   Wafer section: custom made sample holder with a slit of 2 cm available for mounting the samples with same thickness.

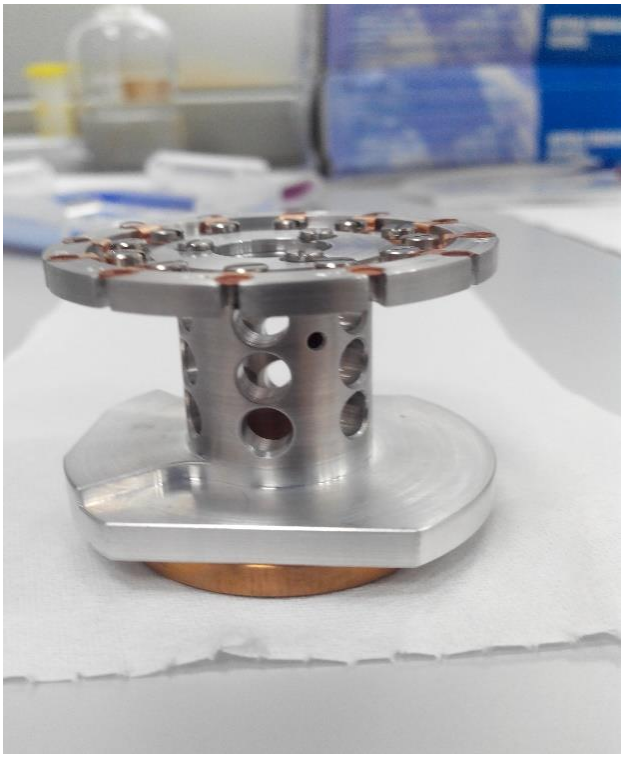
Wafer section

3. Single stub: custom made sample holder with a slit of >2 cm available for mounting the samples with different thickness.



Single stub

4. STEM mode: samples holder has facility to mount maximum 12 samples (Copper grids 3 mm diameter as used in TEM analysis)



Grid

Biological samples, high vapor pressure/degassing, powdered*, dispersion Samples are not allowed.

(Note:* Powdered samples are done on case by case basis. Only powders with nano particles (< 10nm) and properly suspended in carbon black and well dried can be allowed).

Sample/Substrate Size:

(2 mm x 2 mm x Z) to (10 mm x 10 mm x Z) [for surface imaging]
(4mm x 4 mm x Z) to (8 mm x 8 mm x Z) [for cross-sectional imaging]

Where, Z is the variable substrate thickness (it can vary from 200 microns to 2 mm, depending on the common substrate types like: Si / glass substrates etc. But it should not be more than this range; if it is, consult with operator /SO before loading the samples.

➢ Sample preparation on sample holder& loading precautions

1. Check/know about the sample nature and its history, it should not be the kind Of sample listed in the **NOT allowed** samples. If having any doubt, consult SO /Operator/ Faculty in charge.

2. **Samples** should be of **specified size** as given in the SOP.If it is not so, ask the user/sample owner to cut it. If user asks you to do so, do it very Carefully.

3. Always wear clean **gloves**, **hair net**, **safety goggles** and **face mask** while cutting or preparing the samples.

4. Chose the appropriate sample holder for loading the samples (surface/cross-Section).

5. **Surface and cross-sectional samples can't be loaded at the same time on the same holder.**

6. For surface analysis use 9 stubs sample holder. Here, maximum 9 samples can be loaded at a time **only** on the stub area but with the **same** sample height/thickness. If it is different, load the samples in the different runs because while moving from one sample to another, samples/sample holder may get collide with the detectors.

7. Use the good quality carbon tape to stick the samples/substrate on the stubs.

8. Always use **clean** stub and new carbon tape to load the sample.

9. Powdered sample should be loaded with extra care. Unscrew the stub from the holder and separately load/put the sample on to the stub already pasted with carbon tape.Take very small amount of sample i.e. the ball of the ball point pen onto the **Glassine weighing paper** already kept on the smooth and hard **sample preparation table.** Use the same paper and rub against each other in order to convert powdered sample into very fine particles (< 10 nm).Take the top paper containing little amount of powdered sample visible to you. Take the stub already pasted with carbon tape of smaller dimension (<5mm× 5mm) and stick it to the paper containing fine sample particles in a very small amount. Firmly hold the stub pasted with sample using **proper** tweezer and blow the loosely bonded sample particles using N2 Gun. Then only fix it on to the sample holder.

10. Load the samples on the stubs in sequential **order** and note down their Specific **position** on the sample holder so that there should not be any confusion in its recognition after loading into the chamber.

11. Don't put unnecessary stubs on the sample holder not containing samples because this enhances the chance of vacuum chamber contamination.

12. Samples should be freshly cut across the edge to be observed in cross-Sectional mode.

13. While loading the cross-sectional samples into the slit of the sample holder, Keep the sample out of the top surface of the sample holder **NOT** more than2 mm because samples may collide with the BSD/Inlens detectors during stage movement. Also, this helps in reducing the sample charging effect.

14. If there are more than one cross sectional samples of same substrate thickness needs to be loaded at the same time; they should be at the **same** surface **level** and not more than the 2 mm out of the slit from the sample holder surface. Samples with different thickness **can't** be loaded at the same time and should be done in different runs.

15. If the sample is some conducting films deposited on the non-conducting substrate then just use the **silver/carbon paste** at the edge of the sample/substrate in **minute quantity** to provide an electrically conducting path from the sample to the metallic stub.

16. If the complete sample with substrate is non-conducting or semiconducting,

then apply 2-3 nm gold coating as well as conducting silver/carbon paste.

17. Follow the **specified procedure** for **gold coating metal sputtering unit**.

18. Before gold coating as well as after application of the conducting Silver/carbon paste, dry the sample under IR lamp for ~5-10 min.

19. Now load the samples into the chamber immediately after drying.

20. For samples to be analyzed in a **tilted** condition should be loaded on the 9 stub sample holder as used for surface imaging. The sample area/edge which has to be viewed in a tilted condition, should be loaded on the **outer** stubs (**not** the center stub) keeping the edge/area of interest at the extreme end from the centre of the sample holder and radially outward as **shown in figure**. There is specific procedure for positioning and stage movement to do **fast** and **safe** imaging, learn it properly from the SO/operator and follow the same. Figure: sample loading scheme for tilt operation Sample edge/area to be observed

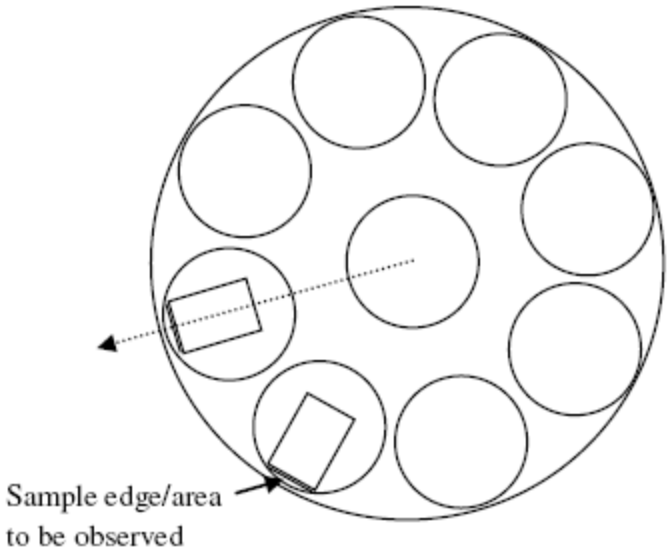
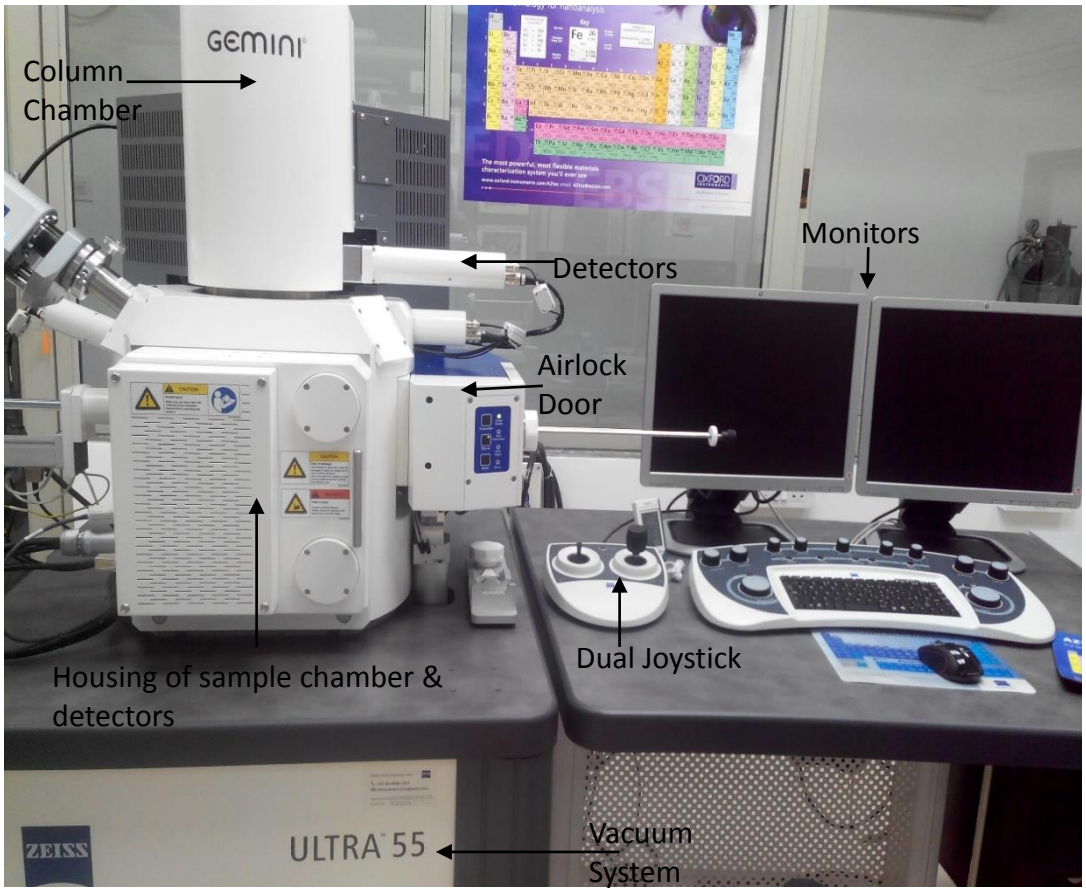


Figure: sample loading scheme for tilt operation

SOP OF FEG-SEM ULTRA-55



1. Check all the **facilities** required by the system

a. Status of the UPS
b. N2 cylinder: Switch on the N2 supply to the system using cylinder key and check:
i. Cylinder pressure (> 10 kg/cm2) [if<, inform Tech staff to **replace it**]
ii. Line pressure (1 - 2 kg/cm2)
c. AC is on and set at 23 degree C.
d. Status of de-humidifier and relative humidity.

2. Check for any notice about the system present for the next user.

3. Wear **clean** gloves, hair net.

4. System always remains in the **standby mode** (yellow button ON) under idle condition in which vacuum pumps are running and keeping the system/column vacuums at specified levels.

5. **Switch ON** the system using **green** button present at the front of system control panel.

6. Wait for the computer system till it completes the **boot up**. Then Log on window will open in that write the password.
   Password: **zeiss**

7. Start the **Smart SEM** Software by double-clicking on the s/w logo and put your **login** username/password required.

   Username: **Sagarmitra**
   Password: **ECEL**

8. Take the appropriate **sample holder** and put it on the clean lint free cloth/paper and **load** the samples on the appropriate **stubs** of the sample holder as per the observations to be done i.e. surface/cross-section.

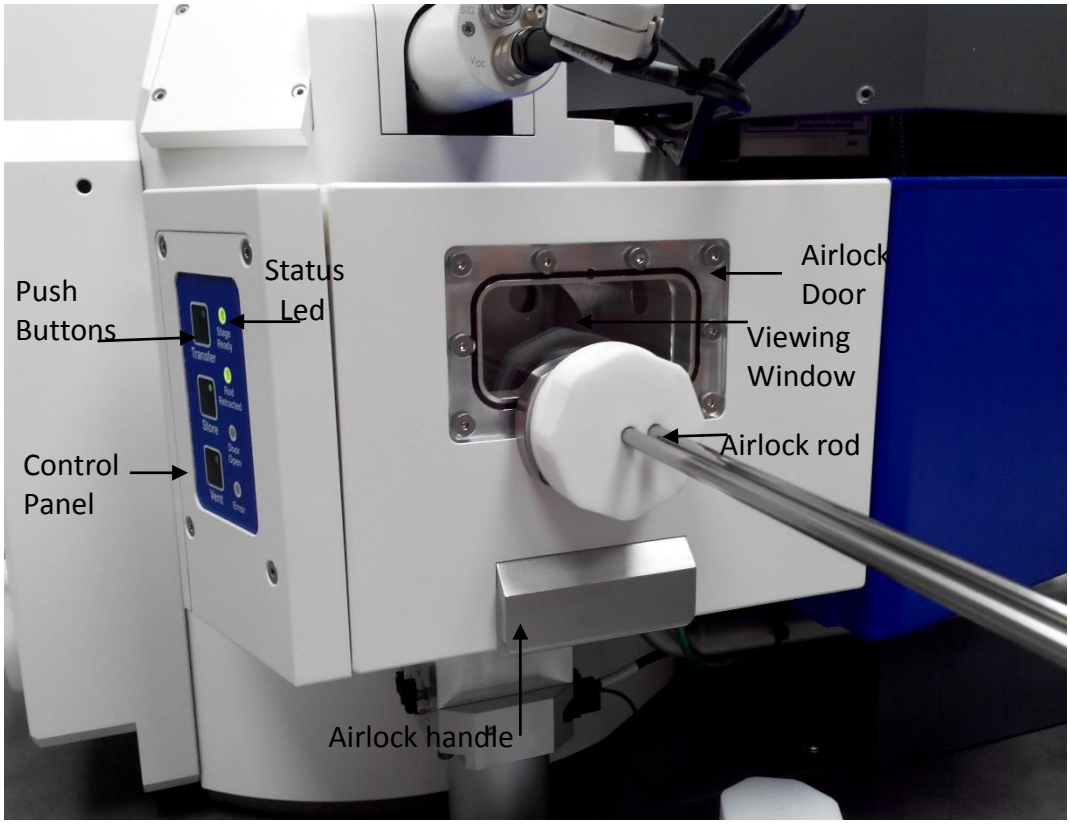
9. Follow the **recommended sample preparation** and **loading procedure** (like: cutting, loading on to sample holder, blowing by N2 gun, drying under IR lamp, gold coating etc.). Non-conducting/Semi-conducting samples should be necessarily gold coated with particular thickness depending on sample and analysis.

10. Do all the sample preparation and loading process on the **sample preparation table** in specified room.

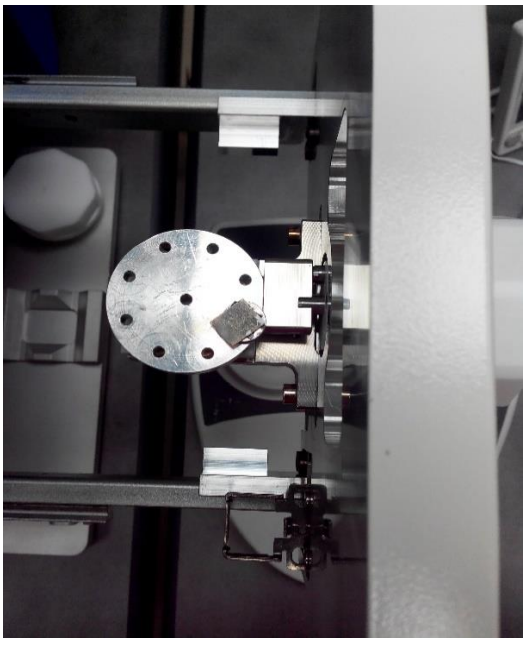
11. Sample height/size should be under **specified range** otherwise it may hit the beam column/BSD detector and severely damage the system during z height Change/ rotate/tilt operations. While loading the samples into cross-sectional Sample holder, keep the samples out of the top surface of the sample holder **NOT** more than 2 mm.

12. While loading the samples on stubs for surface imaging or in a slit of cross-sectional sample holder for cross-sectional imaging, **all samples should be only on the stub area and of equal height.**

13. After mounting the samples on the stubs/sample holder and for loading into the chamber:

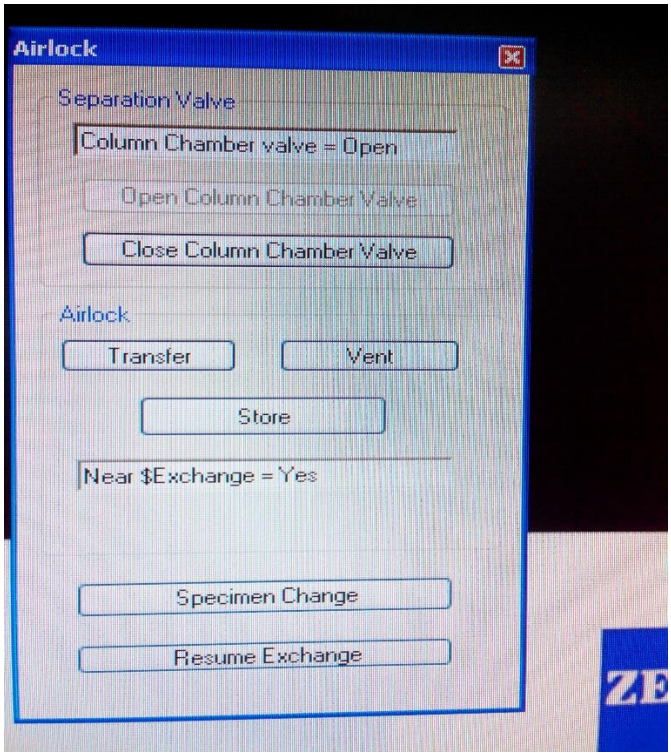


1. **Open** the load lock chamber then Screw the sample loading rod with the Sample holder (sample loading rod should be retracted so that LED for **rod retracted** remains green) then **Close** the load lock chamber.

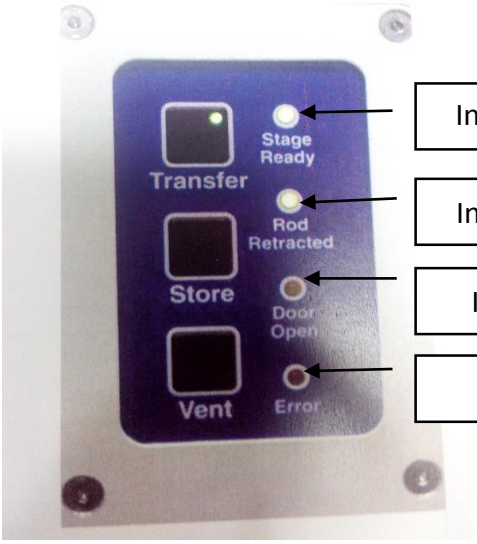


2. Press **Store**.

3. Close the column chamber on display monitor 1 (Airlock window).



4. Press **Transfer** when load lock chamber gets pumped out.



Indicates that stage is in the position ready to transfer

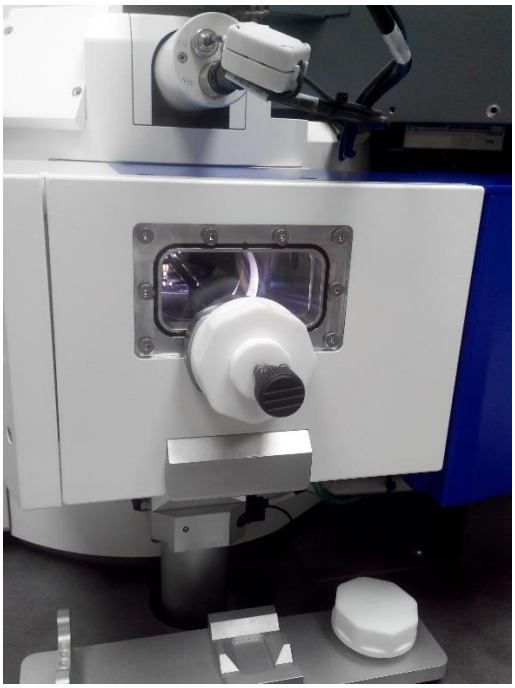
Indicates that the airlock rod is completely withdrawn

Indicates that chamber door or airlock door is open

Indicates an error

5. **Rod insert**

Chamber valve opens and now shift the sample holder inside the chamber and properly load/slide it onto the provided base (sample holder should be Completely shifted till the stopper notch provided on the sample loading base) while sample loading, move the sample loading rod straight inside Without bending it

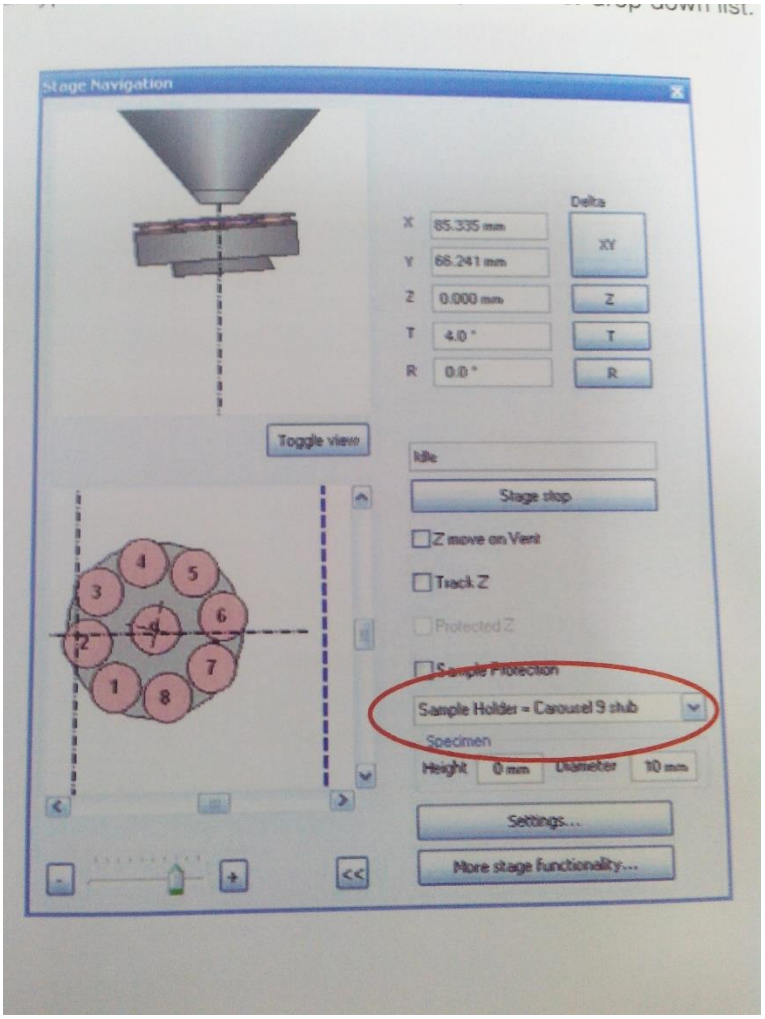


6. **Rod retracted.**

After inserting the rod **completely** unscrew the sample loading rod from the sample holder and take it straight away out of the chamber till **Rod retracted** position comes

7. Press **Store.**
   Now the sample loading is complete. Then wait till **system vacuum** comes **10^-6** and **gun vacuum 10^-9**(It shows on display monitor 1 in gun window).

14. Go to the **Stage navigation map in menu bar** and **select** the same type of **Sample holder** that has been loaded inside the chamber. This navigation map you can see on display monitor 2.

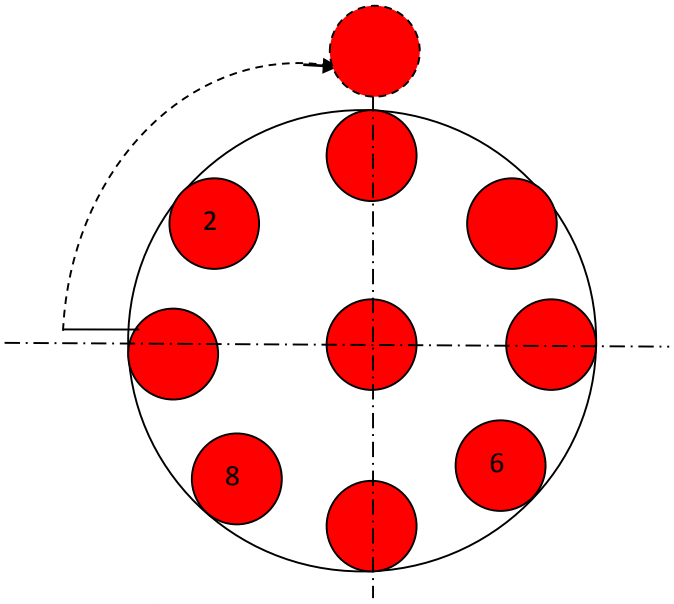


1. Double clique on the particular sample position which you want to Observe, using stage navigation map. Observe the sample holder movements in **TV mode** (sample holder move to the given position and the cross wire in the navigation map resembles the position of sample below the **Column/objective lens**).

2. Surface scan.

   1. Select carousel(9x9)

2. Double clique on centre position (9th stub).Using X-Y direction joy stick rotates the knob till 1st will come exact above to 9thstub. Then double click on 1st stub of centre so in that way 1st sample will focus with gun to get better scanning image. For other samples scanning used the same rotate knob & do double click on their centre position.(position shown in below figure)

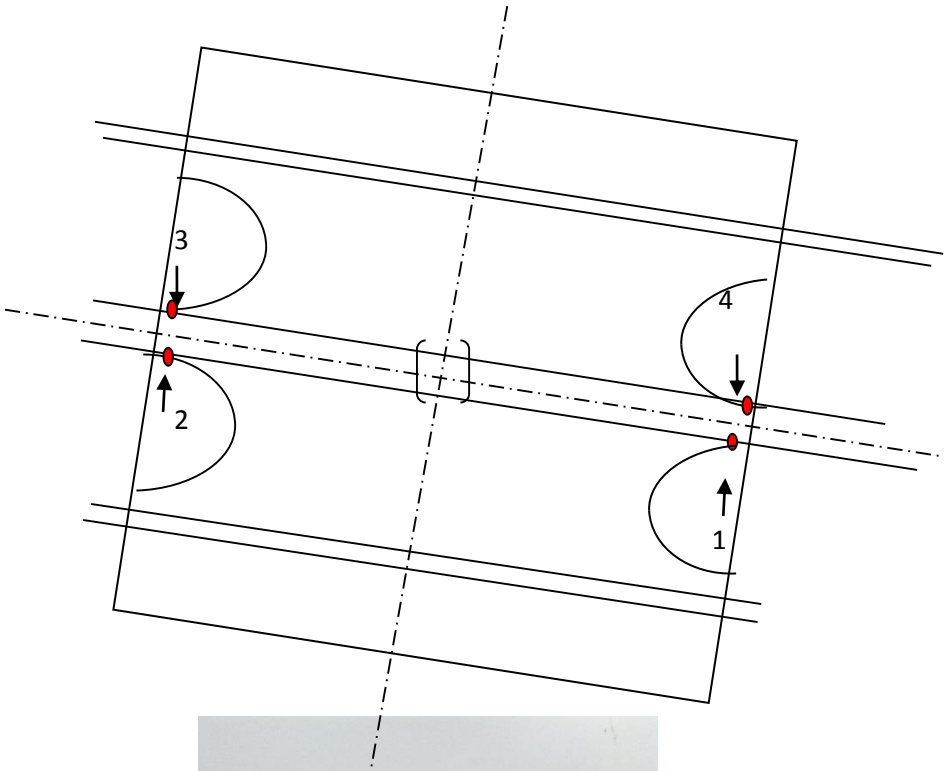


Carousel (9x9) Position in Stage Navigation Window

3. Cross section Scan

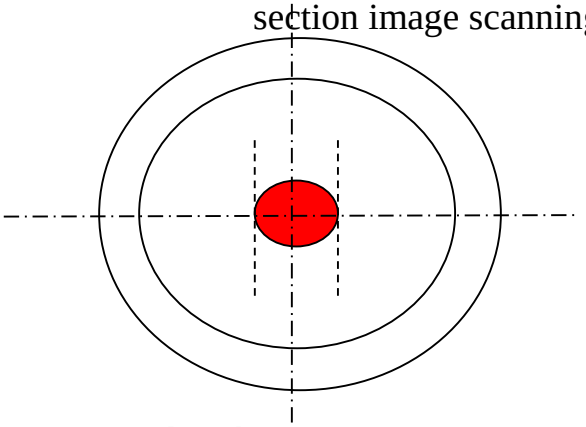
1. Wafer section

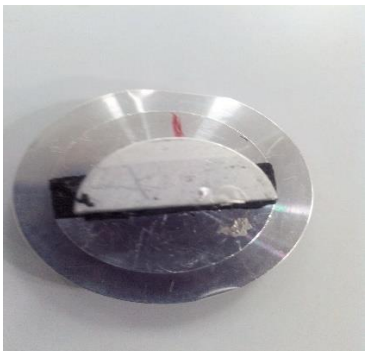
- If sample size in between 1-2mm then use wafer section for cross section image scanning.
- We can place 4 samples in this holder.
- Double clique on centre position. Then using X-Y direction joy stick it can be placed at position 1, 2, 3 &4.
  (Note: Put the arrow mark where you want to see the cross section area on sample & with reference to the number (shown in sample holder) find out edge scanning area. In the diagram it shows red circle mark to understand the position area.



Wafer section Position in Stage Navigation Window

2. Single stub
    - If sample size in between >2mm then use single stub for cross section image scanning.





Single stub Position in Stage Navigation Window

15. Reduce the **gap** between the sample/sample holder and column/objective lens Using **joystick** (Z-movement only) **slowly** and in **smaller steps** but should have a visibly **safe gap** of > 1 cm for avoiding collision with the detectors. (**Note:** do all these stage movements in **TV mode** and very consciously.)

TV mode (Showing distance between sample & gun)

Dual Joystick

16. Check the **System vacuum** and **Gun vacuum** which should be equal to ~10-6 mbar and ~2×10-9 mbar respectively or better than that.

17. Open the column chamber in display monitor 1

18. Set the **EHT** voltage (say ~5 kV, depends on sample material) and Switch ON the EHT (don't do multitasking while switching ON/OFF the EHT)

19. Select the **Inlense detector in signal menu.**

20. Select the **Normal/Image mode**

    1. Adjust the brightness/contrast and observe the sample features at lowest magnification locate the surface feature and focus it. Increase the magnification (i.e5-10 k) and focus it observe the **Working Distance** (**WD**) in properly focused condition (WD tells you the distance of the sample from the **objective lens/ Inlens detector.**

    2. It should **not be less than 3.0 mm** in **Inlens/BSD Detectors.**
       (Note: Standard WD is 3.4mm for surface scan)

    1. It should be ~**7-8 mm inSE2/EDS detectors).**

    2. Go to the TV mode and adjust the Z-height (WD) as recommended for the Particular detector using joystick (Z-movement only). Do it in smaller steps, slowly and very carefully.

21. Go to the **Normal/Image mode**

    1. Observe the sample at lower magnification (~50X)in '**pixel avg mode**' of **Scanning** with appropriate **scan speed 4**and locate the surface feature, try to focus it.

    2. Increase the magnification and keep focusing it &observe the sample at required magnification.

    3. Do **aperture alignment** and **stigmation corrections** if required

    4. Shift the sample position a little bit using **ctrl+tab centering feature** because sample features get changed due to continuous exposure of electron flux (Use the joystick or centering feature for aerial movement across the sample surface at higher magnification only but not at lower

magnification because the use of joystick or centering feature at lower magnification may shift the sample much and hit the detectors)

3. Do **Line Integration for noise reduction** under **Scanning** with preferred scan speed (normally scan speed 5 is good enough but samples which get heavily charged, do the fast scanning otherwise sample features will be lost)

4. After Line Integration the image automatically **freezes** then save the Image using **'Save**' or '**Save as**' options to save the image in particular folder.

5. Again go to the **pixel avg mode** and follow the same procedure for next SEM image.

22. Use the **Navigation map** and **joystick** (X/Y movements) to carefully move to the next sample in **TV mode** only and do SEM imaging as usual.

23. If there is a need of tilting the sample, then first go to TV mode and increase/adjust the gap between sample holder and the BSD/Inlens detector so that it should not collide during the tilt/rotate operation.

24. In case of SEM imaging of **tilt** and **cross-sectional** samples, there are **Specific Ways/instructions** to load the samples on stubs/sample holder, positioning **and** movement to do **fast** and **safe** SEM imaging. So **learn** and **follow** those.

➢ Tilt operation:

1. The samples can be tilted from -40 degree to +70 degree using 9 stub surface sample holder and following **recommended procedure** in TV mode only (i.e. 70 degree in one direction and 40 degree in opposite direction from the neutral (tilt=0) position.

2. The tilt operation should be performed well within the limit and in smaller steps (**Not** more than 5 degrees/step), simultaneously maintaining the safe distance from the detectors in TV mode only.

3. There is a **recommended procedure** for sample holder **positioning** and **movement** to do tilt/cross-sectional samples. **Learn** it properly from the trainer and strictly follow the same.
4. Tilt operation should not be used in cross-sectional mode (if necessary,

only ± 1-2degree tilt is allowed to make the observation edge of the sample perfectly normal to the electron beam in TV mode only)

5. During the **tilt position**, **do not** use joystick (for x/y movements) because it can severely damage the detectors. Here, X/Y movements translate itself into complex Z movements.

25. Reduce/adjust the EHT voltage (kV), if the sample charging (image drift/ Loss Of sample feature) takes place.
(**Note:** change the kV in smaller steps in TV mode and **don't** go beyond **20 kV)**.

26. If there is a need of **EDS analysis** of the sample while doing SEM imaging, Increase the gap between the sample and column/objective lens so that the WD is in the specified range (~7-8 mm), increase the EHT voltage (15-20 kV) in TV mode and follow the **specific procedure** for EDS analysis of the sample.

27. **Closing the Ultra -55 procedure**

1. After completion of the imaging **turn OFF** the **beam/EHT.**

2. Move the stage/sample holder safely down/away from the detectors using joystick (Z movement only) in TV mode

3. Maintain all the **home settings** to original ones (like: tilt=0, rotation=0, EHT= 5 kV, sample holder to 9×9 in navigation map etc.) what you had purposefully changed.

4. Make the entry of the all the required fields (Name, start/end time, number Of samples, nature of the sample, system vacuum & gun vacuum during the SEM imaging and any comments etc.) in the **log book**.

5. Now unload the samples: Go to the **Stage. Store/Recall** → double clique On $**exchange** (sample moves to the exchange position). Follow the same sample loading procedure in **opposite** sequence.

6. Close the column chamber.

7. Press transfer on system then insert the rod rod &screw the knob tightly.

8. After tightening retract the rod.

9. Press store.

10. Press vent & then open the sample chamber.

11. Remove the samples from the sample holder and put the sample holder in its box.

12. Then open the column chamber on display(gun window).

13. Close the EM server and exit the programme (software).Log off SEM s/w and shutdown the system .Put the system in the '**Standby**' mode by pressing the **yellow** button on the front of the system control panel. Ensure that LED's on the load lock chamber corresponding to **Stage ready** and **rod retracted** is **green**.

14. 'Switch **OFF**' the **N2 Supply** to the system using cylinder key if there is no next user for SEM imaging.

15. 'Switch **OFF**' the unnecessary tube lights, empty the bucket of the de-humidifier and check for the problems/faults to the facilities for the system, if any lock the door of the room.

EDAX (Energy dispersive X-ray analysis)

1. In order to perform EDS analysis for elemental analysis of the sample, while doing SEM imaging:

   i. Go to the **TV/camera** mode.

   ii. Reduce the z-height (change the WD to ~7-8 mm in focused condition) using joystick (Z-movement only).

   iii. Increase the EHT voltage (kV) in smaller steps from 5 – 20 kV depending on sample and its analysis.

2. Go to **image/normal mode**

   i. Focus the image move to the particular location/feature of the sample.

   ii. Do the aperture alignment and stigmation corrections if required.

iii. Scan the image in **pixel avg mode** at required magnification and scan speed.

3. Starting steps of **EDS**

i. Switch ON the PC for EDS analysis

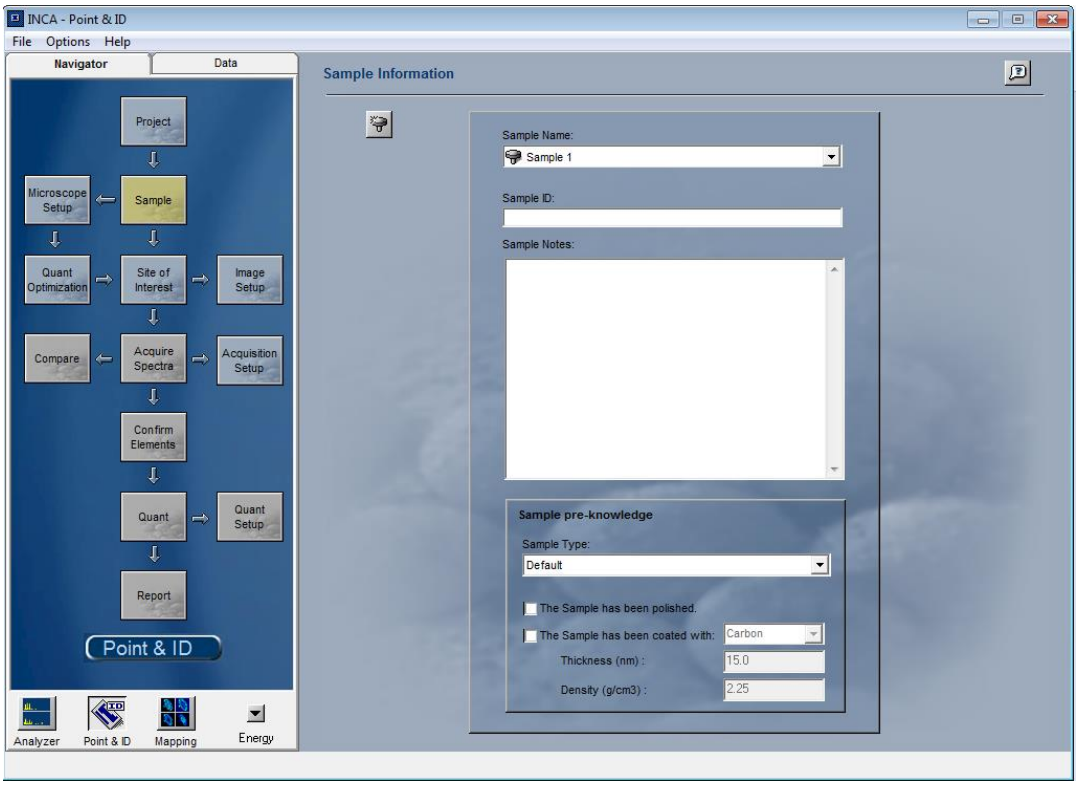
ii. Login to the computer through '**Operator**'

iii. double clique on **INCA** s/w program for EDS analysis

iv. Clique on **full acquisition mode** &select any one of the functions present at the bottom side of **Navigator** for depending on particular sample and its EDS analysis i.e. **Analyzer** for point analysis, **Point and ID** for SEM imaging and EDS analysis of selected points and areas on the image of the sample or **Mapping** for elemental maps and elemental line scan.

v. Follow the interactive/operational **flow chart** provided to do the EDS analysis

4. **Point and ID** function is commonly used points and areas on the image of the sample follow the **flow chart** provided in the program window



1. Clique on project.

2. Enter the project name.

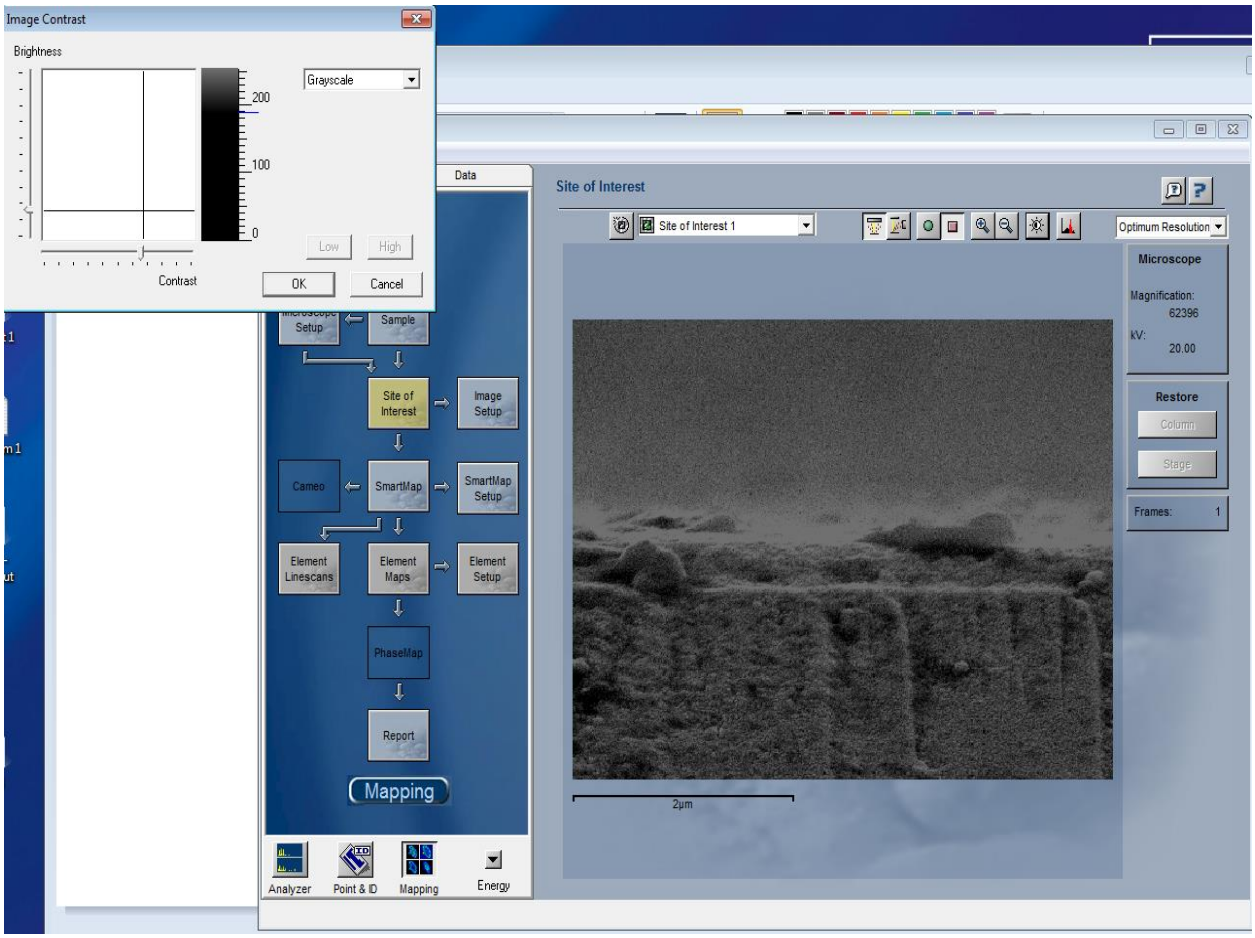
3. Clique on sample.

4. Enter the sample name.

5. Go to the site of interest.

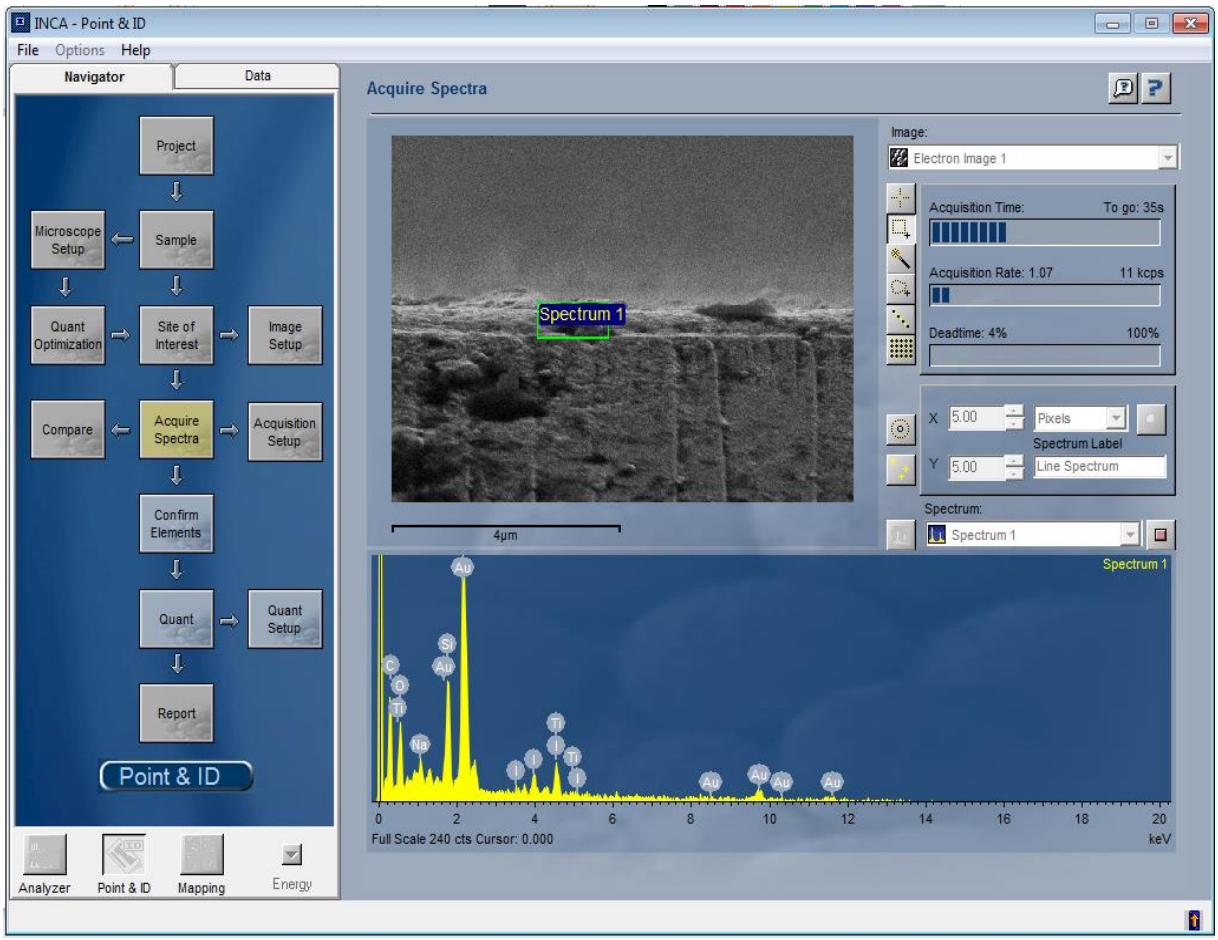
6. Clique on the **green** start button given in tool bar of the program window. (acquisition of image starts, if not then check for "**Remcon32"**communication software whether it is properly running or not)

7. Adjust the **brightness/contrast** of the SEM image using the function given in the tool bar.



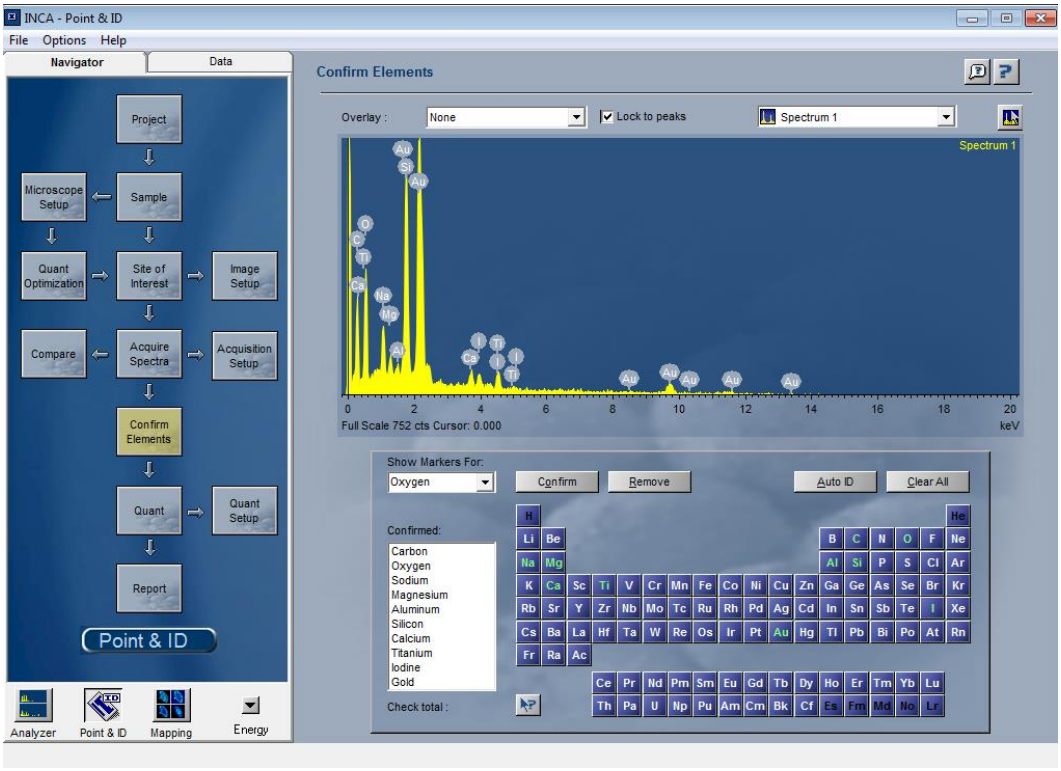
8. Clique on **acquire spectra.**

9. Clique on the image/tool signs given for point/rectangular **area selection** and select the area on the image .(many points/areas can be selected at the same time but system will do one after other is completed)



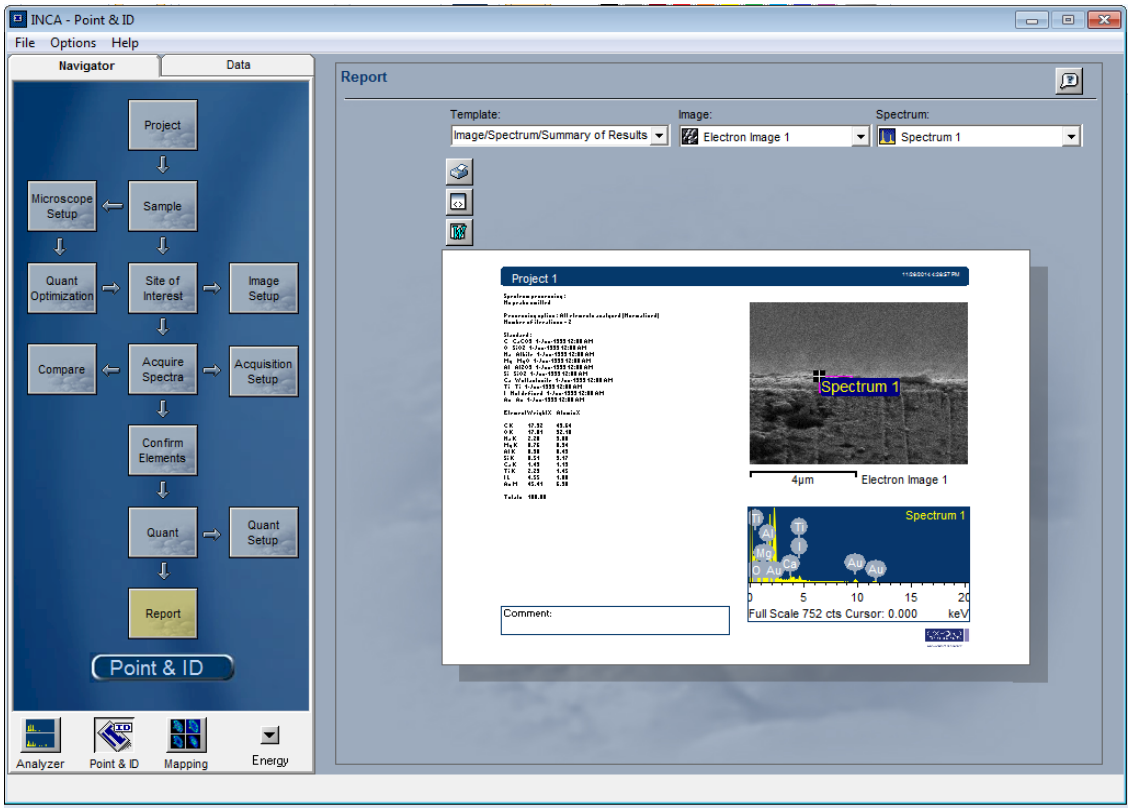
10. Wait till the complete acquisition of spectra.

11. **Confirm elements**(spectra shows automatically identified elements but unnecessary elements can be deselected and additional elements of interest can be selected)



12. Clique on **quant** to quantify the elemental composition in atomic % and weight %.

13. Clique on **report** and select the required format for the report.



14. Do confirm elements and quantification for each spectrum of each site of interest of each sample and save the results/report in word format directly.

15. Finally save the project file in the specified folder for any future spectral analysis required.

16. Close the INCA software.

17. Go the SEM in normal mode and return to original settings made fo EDS analysis.

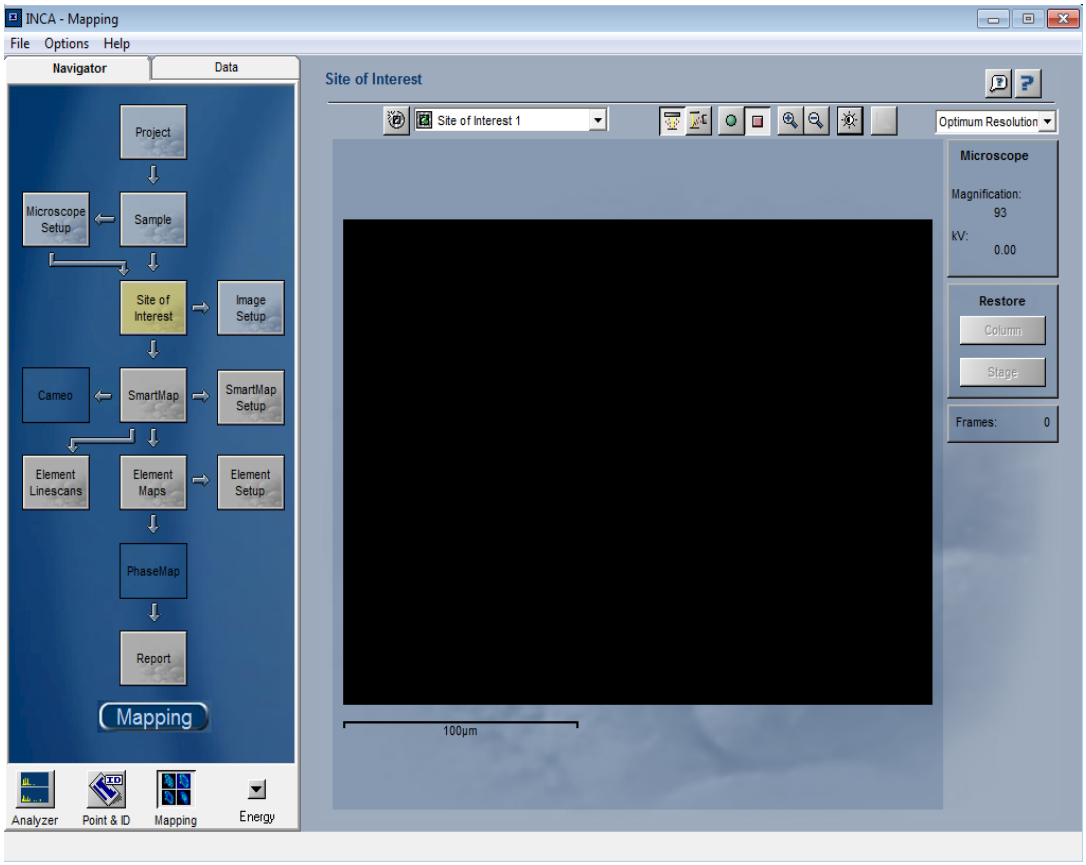
18. For EDS at some other part of the same sample: Let the process of spectral acquisition complete for the already given points in EDS analysis or if necessary, stop the process of acquisition by cliquing on **Red** button given in tool bar of the program window.

19. Go to SEM monitor under **image/normal mode .**Move to the interested part/feature of the same sample using **ctrl+tab centering feature** for smaller movements and joystick/stage navigation map for larger movements in TV mode.

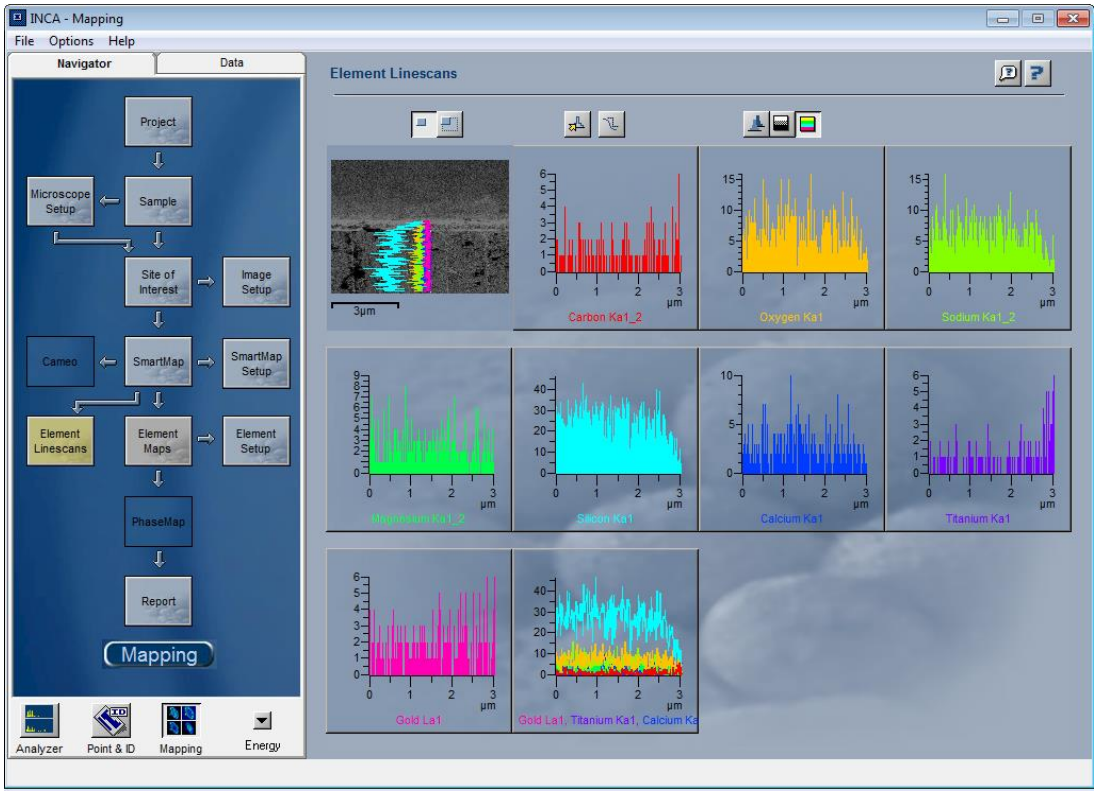
20. Focus the image at required magnification and do all other image quality improvement practices in **pixel avg mode.**

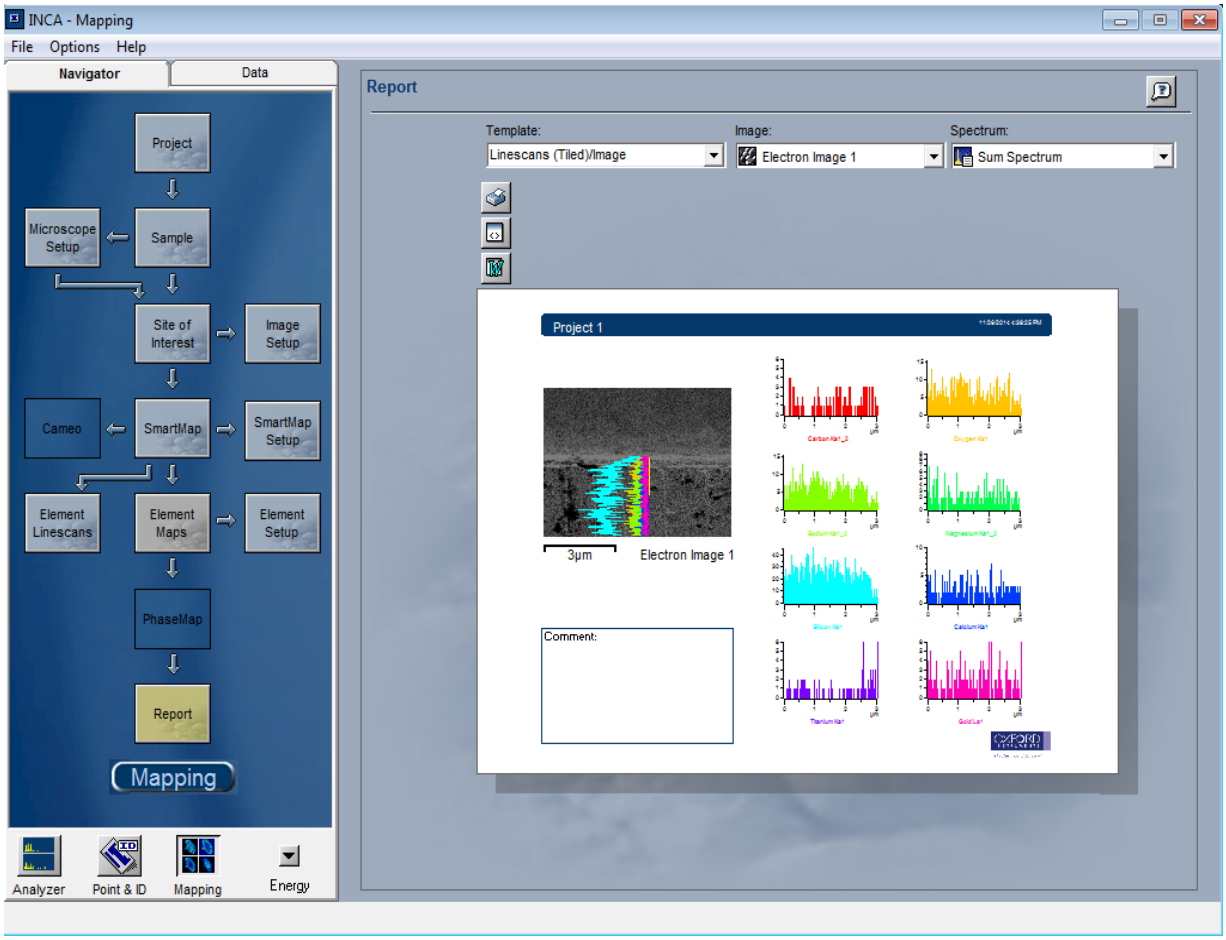
21. Clique on new site of interest under EDS monitor and follow the same procedure for EDS analysis as discussed earlier.

5. **In Mapping,** the procedure is similar to the point and ID but here you get the qualitative map about elemental distribution under selected area or line instead quantitative elemental composition



In smart linescan mapping you can select line of particular region to get qualitative map of colour line profiles about elemental distribution which is shown in snapshot




INCA - Mapping
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Element Maps
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Point & ID
Mapping
Energy
Report
Template:
Linescans (Tiled)/Image
Image:
Electron Image 1
Spectrum:
Sum Spectrum
Project 1
3µm
Electron Image 1
Comment: