2020/3/2 Update

Scanning Electron Microscope (SEM) Basic Manual

Laboratory of XPS analysis

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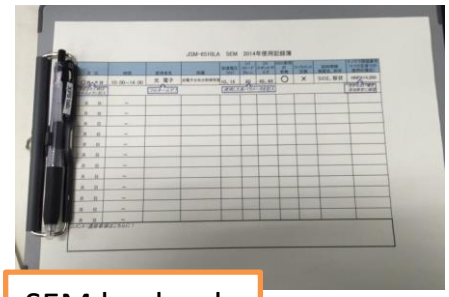
Caution

Please keep the following rules.

- Take off your shoes, and change into slippers.
- No food or drink. Clean up the garbage.
- Contact with staff immediately when you encounter machine trouble.
- Don’t use the machines roughly.
- Don’t take out equipments of this laboratory.
- Keep valuables yourself. Lock a door of this laboratory when you leave here.
- Keep the limit of the stage transfer. The stage will hit the detector if you move the stage beyond the limit.
- Return to original setting after using if you change parameters of software and hardware.
- Don’t connect your USB memory to SEM PC. Use the USB of this laboratory, and take out the data through the PC for analysis.
- Don’t touch with your hand anything which enter SEM chamber. Clean the equipment if you make it dirty.
- Reserve SEM before using yourself, and use the machine on time. Change the reservation beforehand if you can‘t finish on time.
- Respond the all troubles by your laboratory if you use at night, early morning, holiday. Students must let your supervisor know. Emergency contacts are mentioned beside the entrance.
- If you use first time, make contact with the staff before using and reserve the training.
- Don’t enter fragile sample, too big sample, gas exhaust sample to the SEM chamber without permission.

Before using



SEM log book

Write your name and start time on the **log book**, and write finish time and parameters after using. Use as scheduled. Change of reservation on the day is invalid.

The device PC shuts down every time. Press the power button on the device PC and displays. Launch the SEM software by double-clicking the SEM icon on the Windows desktop. When using EDS, turn on EDS power at least 10 minutes before Launching EDS software

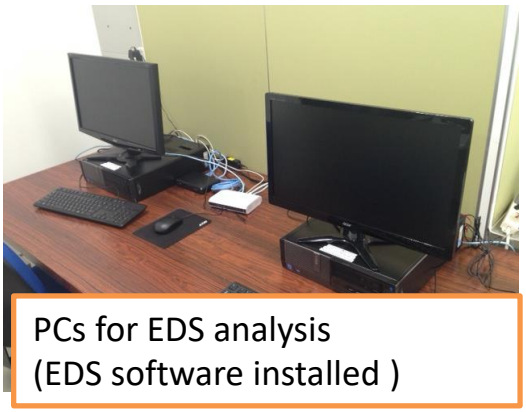
SEM-PC

SEM icon

EDS power

USB memory for data copy

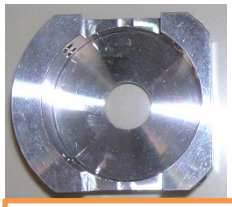
Please manage your data by yourself. We do not guarantee your data stored in SEM PC or analysis PCs. To retrieve the data, use the USB for data copy, copy it to PCs for EDS analysis, and save it on your own data media. It is forbidden to plug your own USB into SEM PC.



PCs for EDS analysis
(EDS software installed)

"**Analysis Station**" which is EDS software has been installed in PC for analysis.
Please feel free to use PC for analysis.

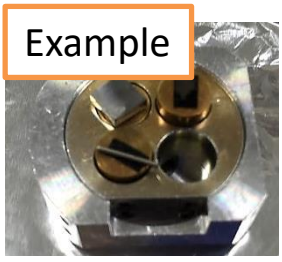
Sample preparation



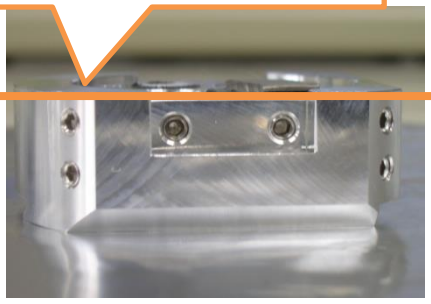

SEM holder（Large / Small）

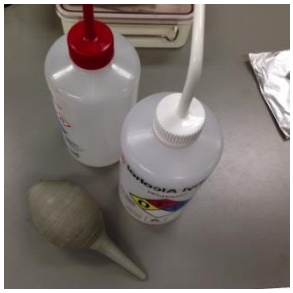
Stages

Example



Same height as holder



Bonding agents

Set sample on the stage

SEM holder and stages are in **SEM box.** Carbon tapes and carbon pastes are prepared.

Don’t touch with bare hand anything which is put into SEM chamber.

SEM holder

Large (32mmΦ × 10mmh)
Small (10mmΦ × 10mmh)

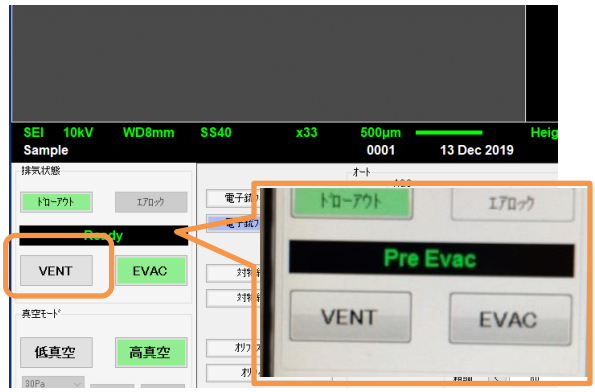
Stages

4 kind of stage are prepared.
Set the stage to the SEM holder by hexagon head bolts.

- ✓ Don‘t put the large samples on the stage beyond the height of holder.
- ✓ Set the sample at same height of SEM holder.
- ✓ Set the sample strongly to prevent moving the sample in SEM chamber.
- ✓ Don’t insert samples which make SEM chamber dirty.
- ✓ Enter the sample in SEM chamber after blowing

Please ask the staff if you have some questions.

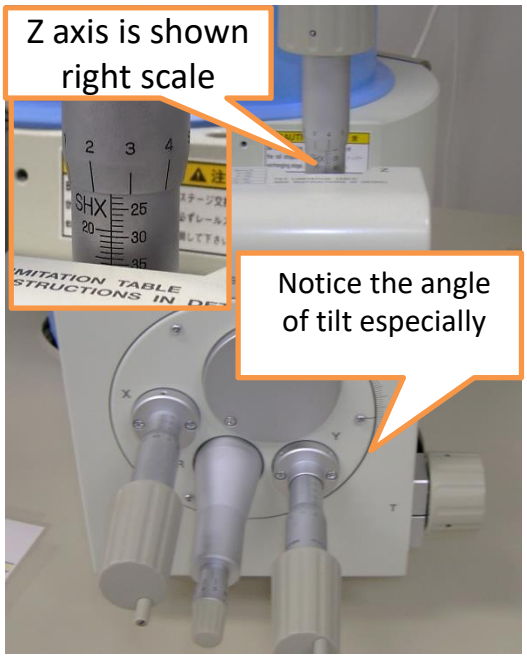
Enter sample in SEM chamber #1



Open to air pressure

Click a “VENT” on SEM software, and then click a “OK”.

＊The Black display section notice chamber conditions.



Confirm the stage position before open the chamber door.

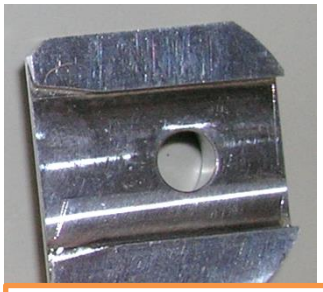
- ✓ Z axis : more than 25 mm
- ✓ X・Y axis : around 20 mm
- ✓ Rotation・Tilt : 0°

The following are prohibited

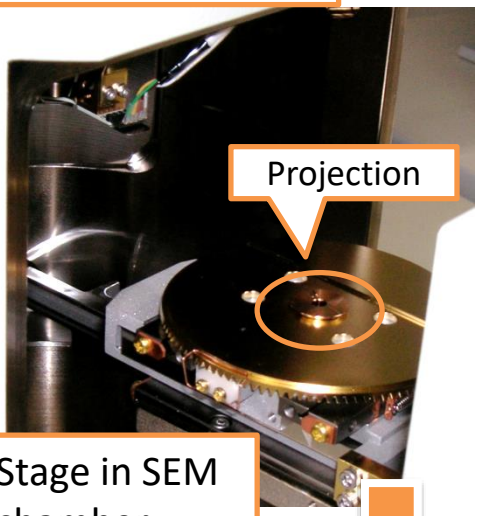
- ✓ Z axis : less than 10 mm
- ✓ Tilt : more than 20° or -5° (When WD=10mm)

After confirming , Lay your hands on knobs, and then open the door

Enter sample in SEM chamber #2



Back side of holder



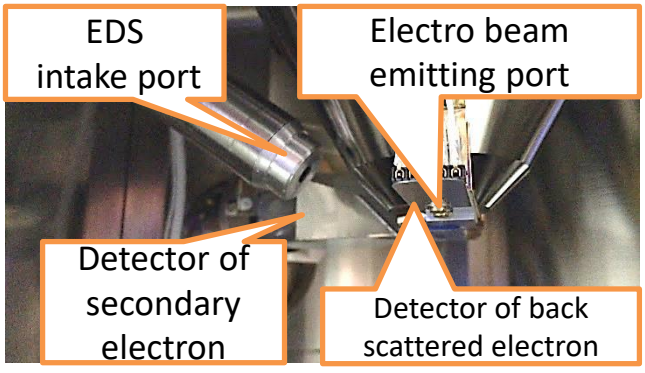
Stage in SEM chamber

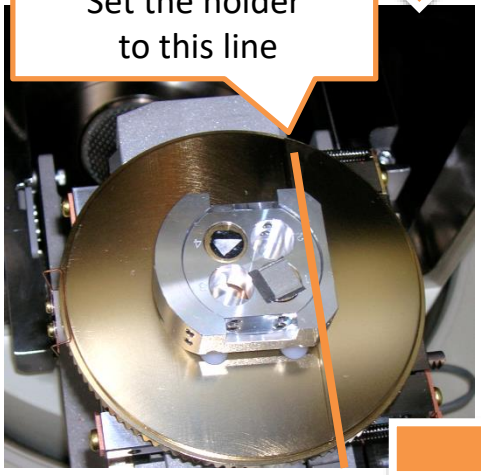
<u>Set a holder on a stage in SEM chamber</u>

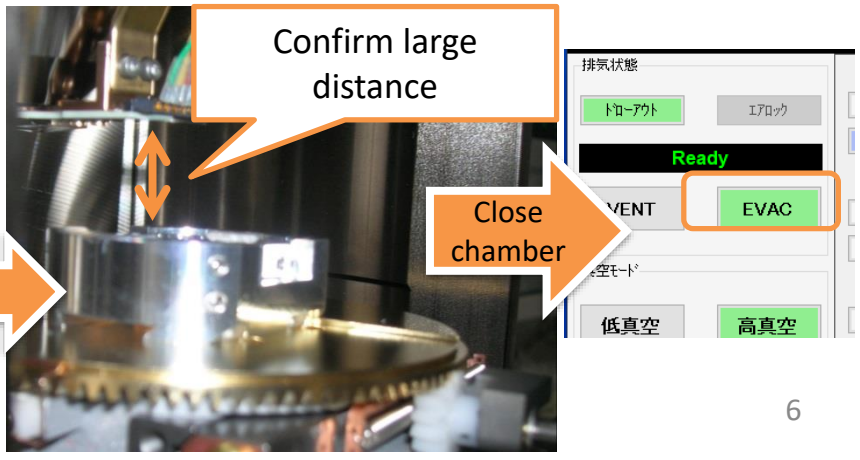
Slide the holder to the projection on the stage from left side, and the projection insert to chase on back side of holder. Confirm the holder don't hit any detector, and then close the door.

Click a **"EVAC"** on the SEM software, and then click "OK". Evacuation start.

Hold the door by your hands after click "OK" for several seconds.



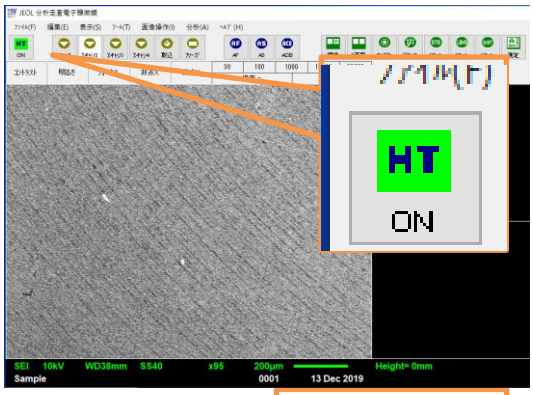




Observation #1

You can observe sample, after changed the condition of the chamber to "**Ready**".

Click a "**HT**" on SEM software, and then SEM image will appear.



Set conditions of electron gun

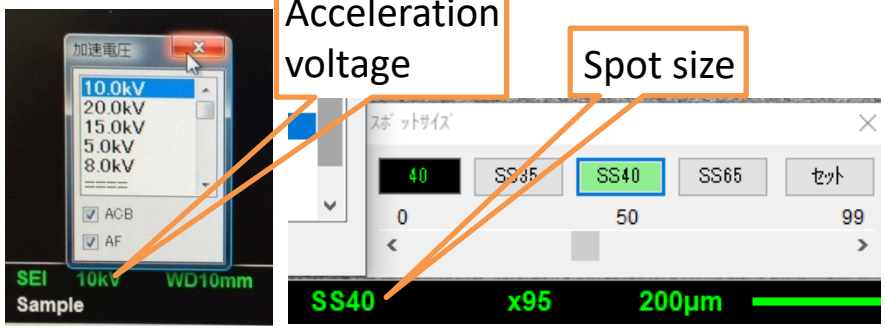
Acceleration voltage : 0.5kV - 30.0kV

Spot size (SS) : 0 - 99

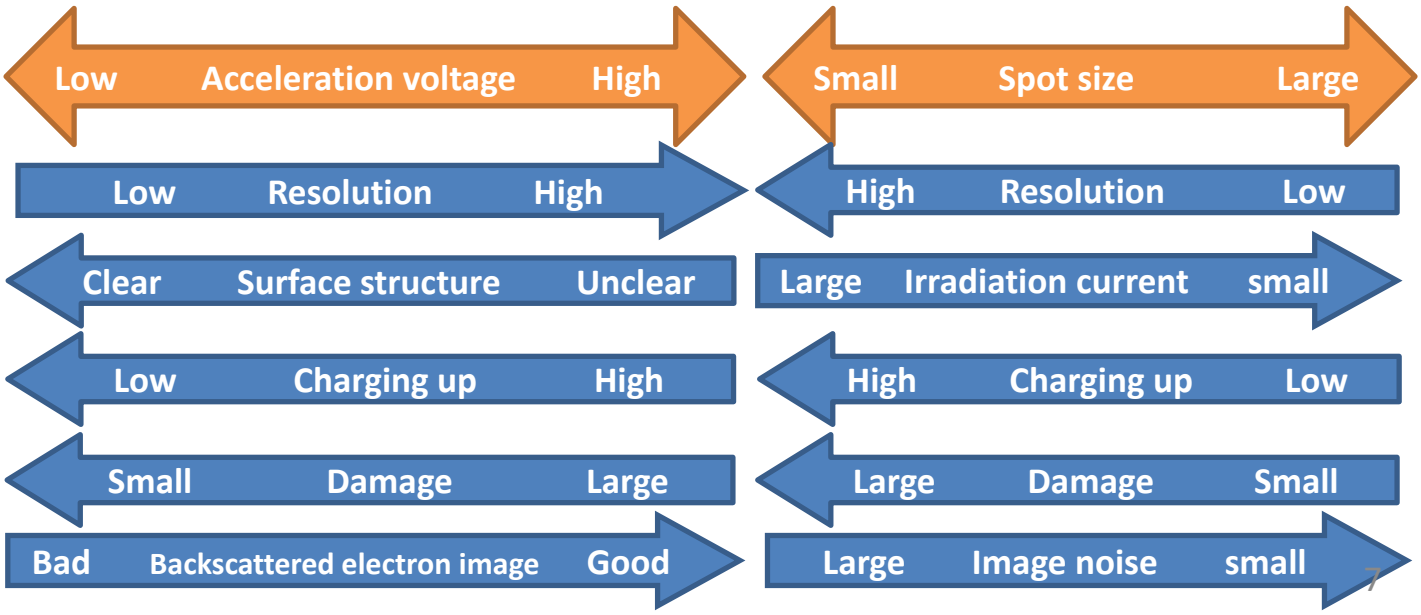
General conditions

SEM: Acceleration voltage 10 kV, SS40

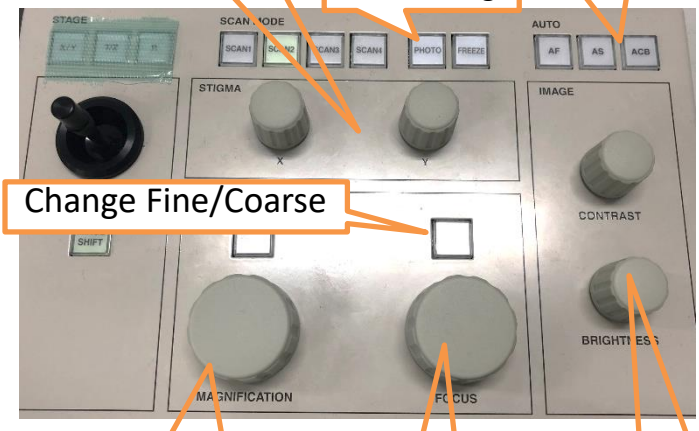
EDS: Acceleration voltage 20 kV, SS65



The effect of conditions of the electron gun



Observation #2



Move the stage (X/Y axis) to target position at low magnification.

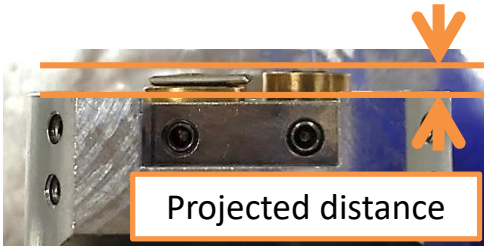
In the case of no image on SEM software, put the ”ACB’ or click a “scan 2”.

Set the working distance (WD) at 10 mm, and then adjust Z axis to match the WD.

SEM observation : WD = 10 nm and more
EDS analysis : WD = 10 nm

If you set large WD, the focus easily be adjusted even rough samples. However, the resolution will decrease.

Working Distance



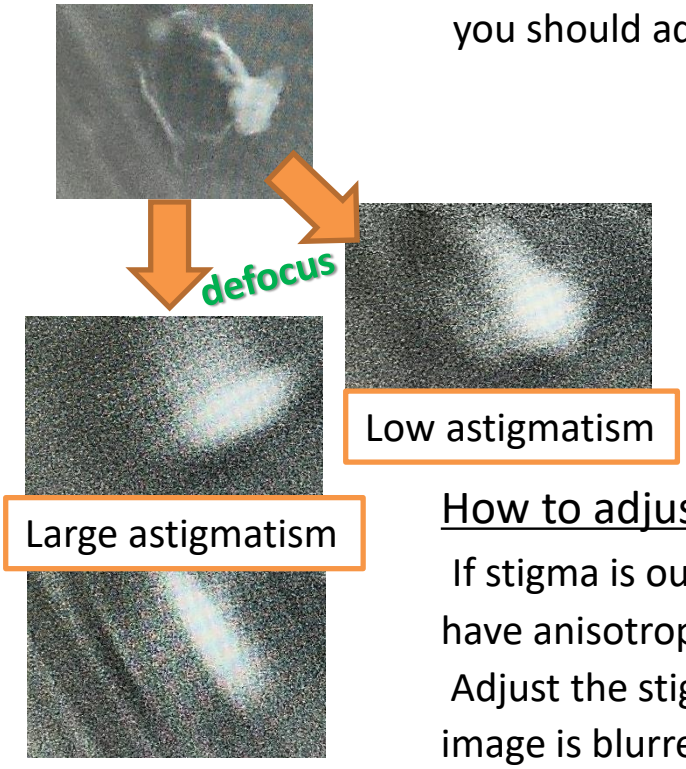
The image will be focused at Z axis = 10 mm if the height of sample and the holder are the same.
In the case of the sample project over the holder, the image will be focused at
Z = 10 mm ＋ “Projected distance”

Recommended use of scan mode
Scan 1 : Set stigma X/Y
Scan 2 : Search target position
Scan 3 : Adjust brightness
Scan 4 : Take a picture

Adjust the focus by focus knob at suitable scan mode, and observe the images.

If WD deviate from 10 mm after adjusted focus, adjustment of Z axis is not collect.
Adjust the Z axis at WD 10 mm again.

Alignment



In the case of observation at high magnification, you should adjust stigma X/Y and objective aperture.

- Adjustment of stigma is need >x5000 observation.
- Adjustment of objective aperture is need >x10000 observation
- Adjust objective aperture after change the electron beam conditions.
- Adjust alignment at scan 1 and at more than twice magnification taking picture.

How to adjust the stigma X/Y

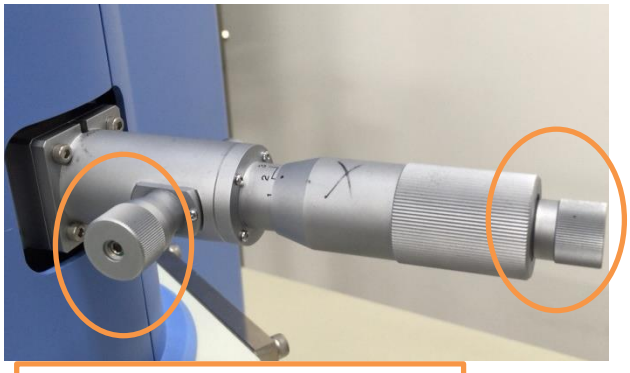
If stigma is out of alignment, the defocused image have anisotropy (Large astigmatism).

Adjust the stigma X/Y knob so that the defocused image is blurred a circle.

Click a “Wobbler”, and then the image will be defocused periodically, and you can easily adjust the stigma.

How to adjust the objective aperture X/Y

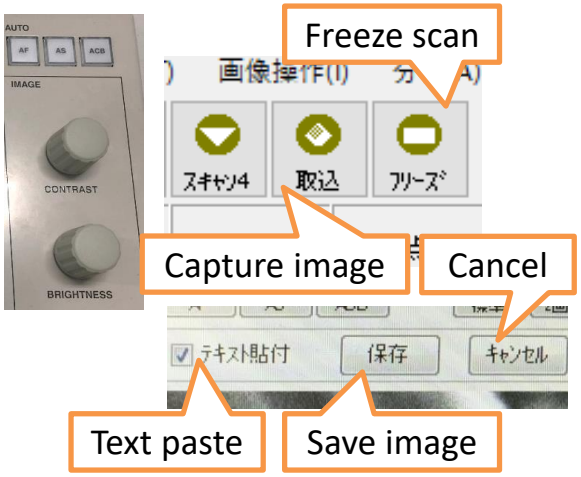
Click the “Wobbler”, and then confirm whether observing position shift or not.



If the position shifted, you need to adjust the objective aperture X/Y to prevent the moving.

After adjust the aperture, adjust the focus and the stigma again.

Save images



Save a image after adjustment of contrast and brightness.

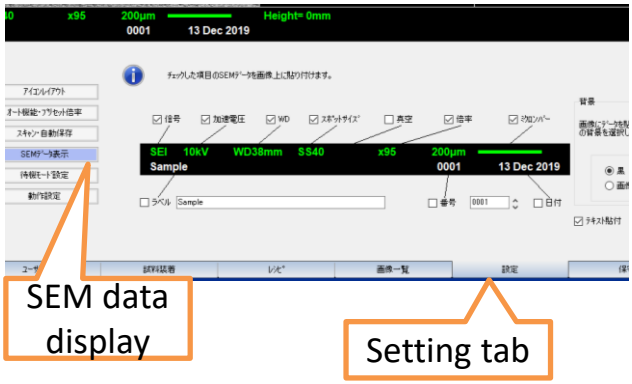
Adjust contrast and brightness yourself not only ACB.

To save image, click a **“acquisition”** or put a **“Photo”**, and then you can get the image.

Check ☑ **”text paste”** in the case of put the information under the image.

If you want to change the content of the information added to a photo, select "SEM data display" in the "Settings" tab under the software and change conditions.

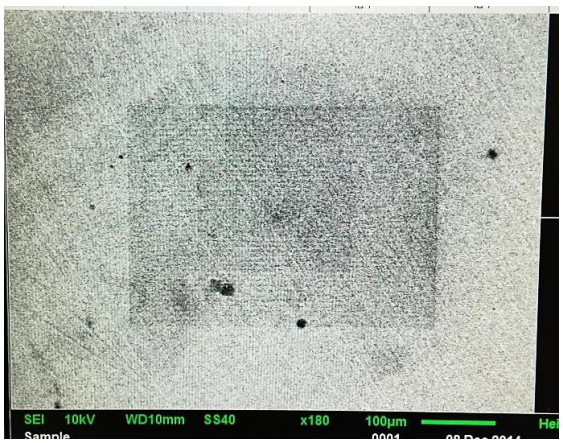
If there is a charging phenomenon, the image will be less distorted if you take a picture with scan 2 or scan 3. To shoot with Scan 2 or Scan 3, select the scan mode and click “Freeze”



If contamination exist on the sample, the beam irradiation area will change to dark like a left image.To prevent the changing image, keep the sample clean.

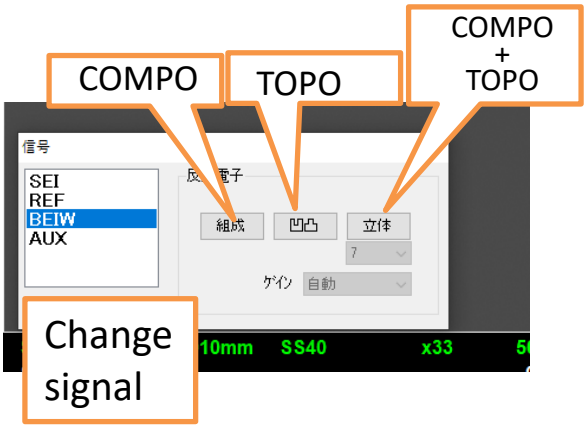
To use following methods, the affect of contamination will decrease.

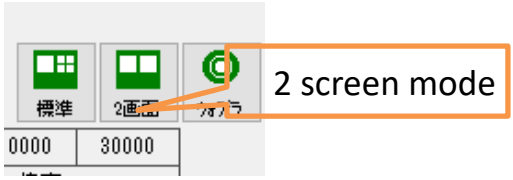
- Decrease the electron beam power.
- To adjust the focus and stigma at different place from the target position for saving image.



You can change the scan speed with "Scan / Auto Save" in the "Settings" tab under the software. Adjust the settings if there is a charging phenomenon, if you want to take a lot of photos quickly, if add signals and increase the resolution, etc.

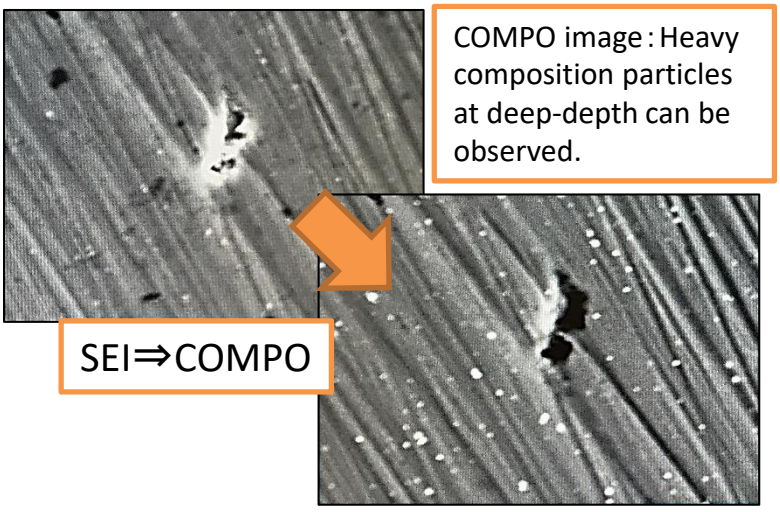
Observation of backscattered electron image (BEI)

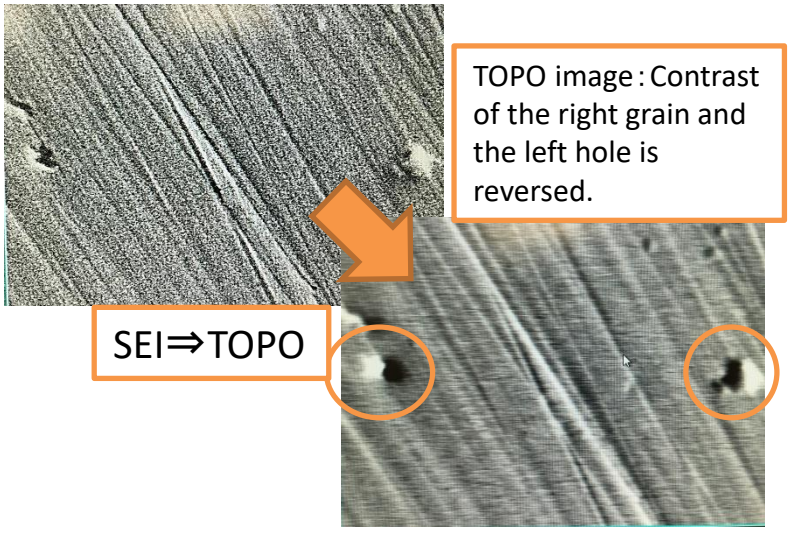




This SEM can observe composition image (COMPO), topographic image (TOPO), and stereoscopic image (COMPO+TOPO) by backscattered electron detector besides secondary electron image (SEI).

To observe BEI, change the signal and then select suitable mode, and change to high acceleration voltage and large current.





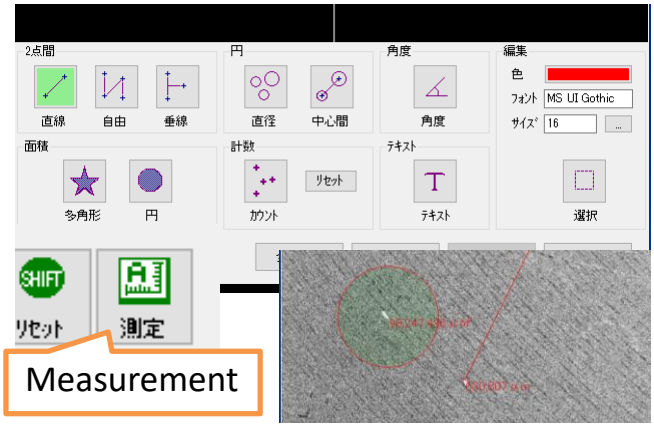
- Secondary electron image (SEI)

Contrast of SEI image is reflected that information of sample shape.
SEI image is strongly affected by surface electrification of sample.
The information is gotten from less than several nm depth.

- Backscattered electron image (BEI)

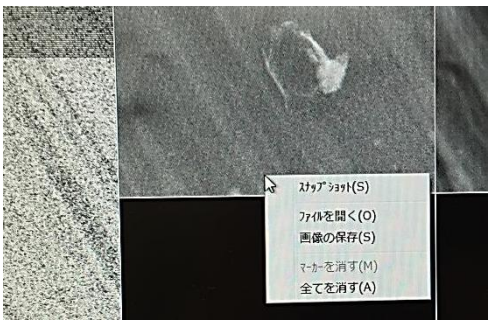
Contrast of COMPO image of BEI is reflected that surface composition of specimen. A heavy composition is bright and a light composition is dark.
Contrast of TOPO image of BEI is reflected that surface topographical information. Shadow appears like there is a light source on the right side of the screen.
The information is gotten from less than several μm depth. Also contrast based on the crystal orientation appears.

Other functions of SEM

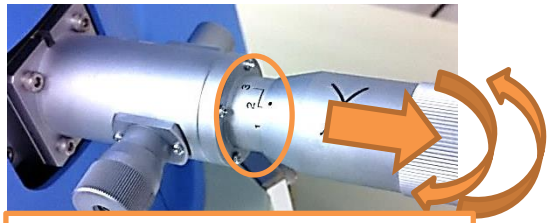


Measurement

To click a “Measurement”, you can measure the length of target .



It is possible to save images on right 4 windows temporarily.



Turn a knob after pulled out

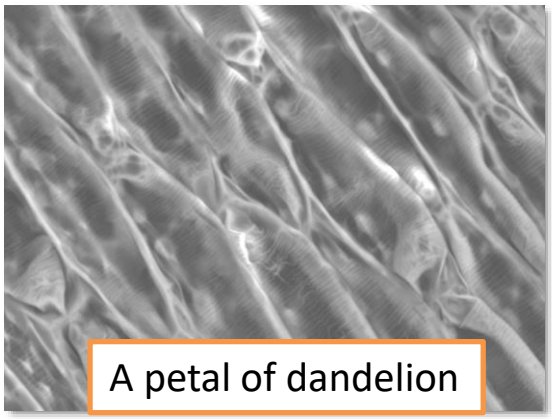
Diameter of objective aperture can be changed. Small number of the knob means small diameter of objective aperture, and you can get small spot size.

If you change the diameter, please turn knob back No. 2 position after using.

Icons

- Blank
  Don’t irradiate electron beam to samples.
- STIG reset
  Initialize stigma X/Y
- LENS reset
  Degauss hysteresis of objective lens. Please click before using.
- SHIFT reset
  Initialize of observing position.

Low vacuum mode



This SEM can observe and analyze samples in low vacuum conditions (30 Pa - 270 Pa)
Biological sample, water including sample, and sample has no electrical conductivity can be observed without pretreatment.

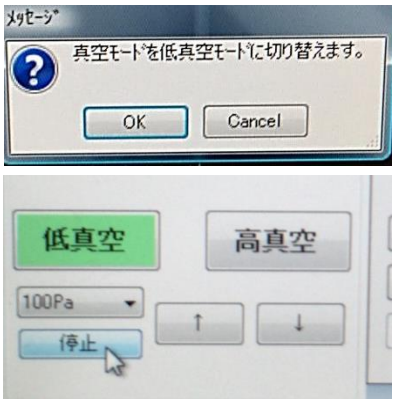
Please ask the staff if you use low vacuum mode!

It is possible to observe only backscatterd electron images (BEI) in low vacuum condition.

Select to "Low vacuum mode" before the sample into vacuum chamber, and then click the "VENT". Enter the sample in the chamber and click the "EVAC".

Don't use "High vacuum mode" when observing the sample need low vacuum condition! SEM will be broken by emitted gases!

"**Vacuum degree**" of the chamber can be adjusted from 30 Pa to 270 Pa. Select the vacuum degree, and then click "**Start**".
Please wait several minutes after the click.



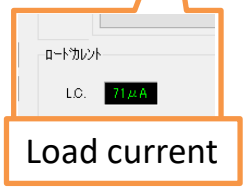
You can observe SEM after turned on "**Ready**".

After finish SEM use, change "**High vacuum mode**" and click a "EVAC".

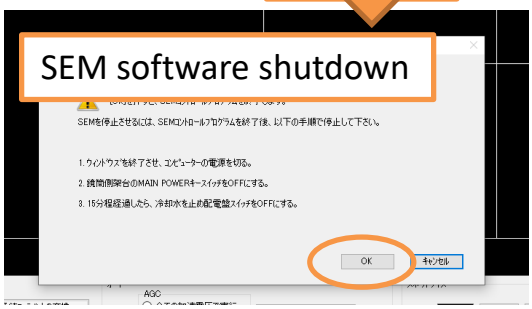
How to end SEM observation

power of EDS

In the case of using EDS
Save the project folder, and close **“Analysis Station”.**
Turn off the power of EDS.



Load current



Only exchange sample

- Turn off “HT”
- Return the stage position.
  - ✓ Z axis : more than 25mm
  - ✓ X/Y axis : around 20 mm
  - ✓ Rotation / Tilt : 0°
- Click the “VENT” to return the chamber to air pressure.
- Open the door, and take out sample holder from the stage in the chamber.
- Close the door, and exchange the sample
- New sample set on the stage. Close the door, and click the “EVAC”

- Return the changed parameters.
  (Acceleration voltage 10 kV, SS 40, WD 10 mm, Objective aperture No.2)
- Select "Maintenance" on the lower tab of the software and check the load current value
- Turn off **“HT”.**
- Return the stage position.
  - ✓ Z axis : more than 25 mm
  - ✓ X/Y axis : around 20 mm
  - ✓ Rotation / Tilt : 0°
- Click the “**VENT**” to return the chamber to air pressure.
- Open the door, and take out sample holder from the stage in the chamber.
- Close the door, and click the **“EVAC”** to evacuate the chamber. (If you used low vacuum mode, return to high pressure mode and click the “EVAC”.)
- Reject the sample from SEM holder, and clean the holder, and then the holder into SEM box.
- Data collection(do not use your USB)
- After the vacuum state of the chamber changes to “Ready”, SEM software shutdown. And PC shutdown. Displays power off.
- Write on the SEM log book.