Instructions for operating JEOL JSM-7500FA analytical field emission scanning electron microscope at Nanomicroscopy Center

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Juuso Korhonen (juuso.t.korhonen@tkk.fi)

Latest version of this document can always be downloaded from:
http://nmc.tkk.fi/en/instruments/sem/jsm-7500fa/SEM-instructions.pdf

Official information

New user training

Inexperienced users have a couple of options, listed below in the order of preference.

1. Ask for training from the most experienced SEM user of your research group.
2. Attend to the courses **Tfy-125.4313** and **Tfy-125.4314** Microscopy of nanomaterials (5+5 cr). They are lectured each spring by Prof. Janne Ruokolainen.
3. Ask one of the administrators to arrange a training session.
    a. Small groups of 2-3 people are preferred for the trainings. Allow some time to gather enough people for the group.
    b. Training is done using a practice sample and personal samples are usually not imaged.

Experienced users can contact one of the administrators for a short introduction to the equipment.

Every new user has to be approved by one of the administrators before they are allowed to use the SEM on their own. The administrator keeps a short (15-30 min) session where the essential skills of the user are checked.

User application

User application has to be filled in order to gain reservation access to any of the NMC equipment. The form can be found from http://nmc.tkk.fi/en/documents/nmc_user_application_form.pdf and it is returned to one of the administrators for approval.

Pricing

Billing is done using the current NMC price list. Contact Prof. Janne Ruokolainen for the most current list. Please note that individual training given by the administrators will also be charged.

Precautions – read carefully

- Always **check the liquid nitrogen level** and fill if necessary.
  - **First user** of the day **always** fills the tank.
- **Keep** all parts **clean** and clean them if necessary with ethanol.
  - **Wear gloves** when handling holders.
- Fill the **log book** on the computer.
  - Mark any **strange behavior** or **problems** to the **log book**.
- If something is **missing** from the SEM or from the sample preparation room (e.g. gloves, ethanol, holders, carbon tape), please inform one of the administrators (send email or call).
- **Use only features you are trained to use**. For example, do not use EDS or RBEI if you don't know how to operate them safely.
- Use of **USB sticks** is strictly **prohibited** due to security issues and hardware incompatibility.
- **Stay calm** and use your **common sense**.
- **Contact administrators** if you are in doubt. Contact information is found on the last page of these instructions.

Quick startup procedure

1. **Turn on both monitors** and check that SEM software and usage log (Excel) are running. Start them if necessary. Log in as Guest (no password).
2. **Check the liquid nitrogen level** and fill if necessary. The **first user** of the day **always** fills the tank.
3. Fill the usage log:
    a. **Date, start time** (and end time).
    b. **Your name** (and the name of your host if you do not have reservation permissions).
    c. **Vacuum levels** before starting.
    d. **Amount of filled liquid nitrogen** (write "0" if you only checked the level).
    e. Write notes and comments to the last field is necessary.
    f. Save the file (Ctrl-S).
4. Prepare your sample.
5. Insert sample into microscope:
    a. Press **Exchange position**.
    b. Press and hold **VENT** for ca. 1 sec. Open securing latch. Wait.
    c. **Open chamber** and **insert holder** along the direction of the arrows.
    d. **Close chamber**.
    e. Press and hold **EVAC** for ca. 1 sec. Wait until blinking stops.
    f. Operate the rod to move the sample to the stage. If you're not absolutely certain how to do this, read the detailed instructions!
    g. **Take out** the rod.
6. Wait until vacuum level reaches **less than $5 \cdot 10^{-4}$ Pa**.
7. Set **Emission current** to **10 µA**.
8. Select **Acceleration voltage**.
9. Press **Observation ON**.

Shutdown procedure

1. Press **Observation OFF** to turn off acceleration voltage.
2. Press **Exchange position**.
3. **Take out holder** using the rod.
4. Press and hold **VENT** for ca. 1 sec. Open securing latch. Wait.
5. **Open chamber** and **take out** the **holder.**
6. **Close chamber**.
7. Press and hold **EVAC** for ca. 1 sec.
8. Mark ending time and observations to **Usage log** and **save file** (Ctrl-S).
9. Set SEM Monitor software to normal settings:
    a. **Exchange position** pressed (green).
    b. Mode: SEM
    c. **Magnification:** minimum for both SEM and LM
    d. Probe current: 8
10. **Turn off** special features you have used: Image rotation, dynamic focus, etc.
11. If you made any changes in the **Operation Settings** menu, change them to normal values (scan speeds, image function, etc.).
12. **Clean the holders** with ethanol if necessary.
13. **Clean tables**. If you want to store your samples, mark them with your name and put them onto a shelf. Things left on the table are thrown into the trash.
14. **Transfer your images** from the small computer on the back table. You can find your files at the network drive called *Harley*.
    a. Use USB stick, SSH, email, or burn a CD.
    b. The files cannot be transferred directly from the SEM computer due to security reasons.
15. **Turn off** monitors. Do **not** log out from the software or close the Excel log book.

Changing sample

1. Press **Observation OFF** to turn off acceleration voltage.
2. Press **Exchange Position** to move the stage to correct position.
3. **Take sample out** by operating the rod.
4. Press and hold **VENT** for ca. 1 sec. to flush chamber and **open latch**. Wait.
5. **Open chamber** and **take out sample** (pull along the arrows, not up).
6. **Change sample** and insert holder along the arrows.
7. **Close chamber** and **secure** with latch.
8. Press and hold **EVAC** for ca. 1 sec. Wait until blinking stops.
9. **Insert sample** by operating the rod. Take out rod.
10. **Wait** until chamber vacuum reaches **$5 \cdot 10^{-4}$ Pa** before turning on acceleration voltage.

Special features

This is only a quick reference. Special training is required to use RBEI or EDS, because of safety issues.

Infrared camera

You can see inside the chamber using the infrared camera.

1. Switch camera on from the button on the table.
2. From SEM software select Navigator -> Infrared camera
3. Turn camera off when using RBEI or EDS.

Probe current meter

Probe current meter can be used to check the current going to the sample. It is most important in EDS analysis.

1. Insert the detector by checking PCD from the bottom right corner of SEM software.
2. Take out detector after you have read the current from the SEM software.

Retractable backscattering detector (RBEI)

Backscattering detector is used to distinguish between elements on the sample.

1. Set working distance to 8 mm or more.
    a. Inserting RBEI with less than 8 mm between the sample and the objective lens will result in serious damage.
2. Turn off infrared camera.
3. Insert detector by checking RBEI from the bottom right corner of the SEM software.
4. Select COMPO or TOPO for image mode (same menu as SEM and LM) and user a slow scanning speed for observation.

X-ray analysis (EDS)

This guide is not adequate for proper operation of EDS, but is only a quick reference for trained users.

1. Set working distance to exactly 8 mm.
    a. Focus with Z height using the ring of the scroll wheel instead of FOCUS.
2. Insert RBEI.
3. Turn on bias voltage by clicking the lightning indicator.
    a. Wait until count rate stabilizes.
4. Select Analysis from the right side of SEM software.
5. Click DT (dead time) and select T4 from the list.
6. Adjust probe current so DT becomes green (around 20-30 %) and count rate is ca. 2000-3000 cps.
7. Take spectra, line scan, or mapping using the appropriate buttons.
8. When asked about saving to a network drive, select OK.
9. Save the analysis before exiting analysis mode in order to be able to return to the analysis later.

   a. Exporting only saves the image and you cannot return to make more analysis on the data.
10. When you are finished with analysis, turn off the bias voltage and take out RBEI.

Saving EDS spectra

If you want to be able to plot your EDS spectrum, select Export and then select MSA file. It will save the spectrum in a compatible file for use in Origin, Excel, or some other plotting program.

Detailed instructions

Operating the rod (sample exchange mechanism)

This procedure describes how to use the sample exchange mechanism in order to either remove or insert a sample holder into/from the microscope. Read this section completely through before proceeding and make sure that you understand every step.

Precondition: The exchange chamber is in vacuum and the door separating it from the microscope is open. Confirm that **EVAC light is lit** and **not blinking**. Depending on whether you are inserting or taking out a sample, the holder might be in the exchange compartment (HLDR light is off) or inside the microscope (HLDR light is on).

See the video on the computer desktop for a demonstration. **UPDATE: The figures are from an old version of rod.**

1. **Push the bar inside** the microscope by following the procedure:
    a. **Lower the rod** to horizontal level, while lightly holding it back.
    b. **Let the rod be pulled in slowly.**
    c. **Push the bar** gently **all the way inside** until it stops (d).
        - There is a little resistance at the final couple of centimeters.
        - The sample should be now either released from the bar or attached to it (depending on whether you are inserting or removing the holder).
        - If you have not pushed the sample all the way inside and start to pull backwards there is a danger that the sample holder will fall to the bottom the sample compartment. If this happens, the whole sample compartment has to be opened. Contact SEM administrators in this case.
2. **Pull the bar out** from the microscope using the following procedure:
    a. **Pull the bar** out **as far as it comes** (e).
        - The two arrows on the holder should align with the pipe end.
        - If you have not pulled far enough, the rod might be damaged during the lift.
    b. **Lift the rod** upwards to vertical.
        - Now you should either have the sample inside the microscope or in the exchange compartment and the exchange compartment is in vacuum.

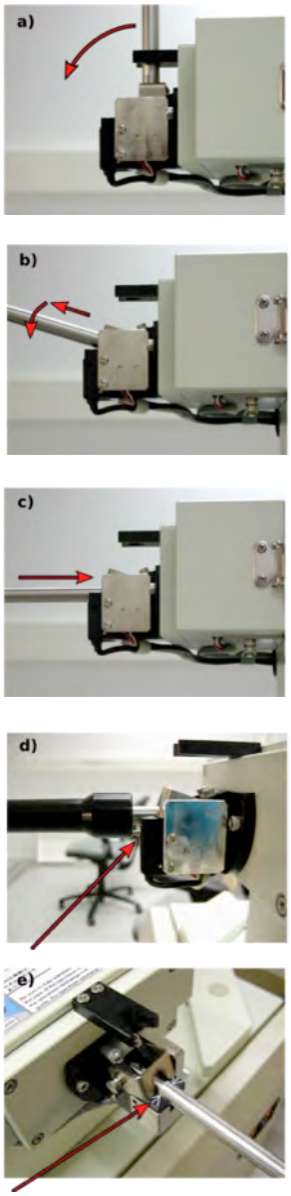


Figure. Sample exchange rod operation.

Opening the sample exchange compartment

The following procedure describes how to bring the exchange compartment to atmospheric pressure.

Precondition: There is no sample inside the microscope or it has been brought to the exchange compartment, and the exchange compartment is in vacuum. First check that **HLDR light is off** on the sample exchange compartment (i.e. there is no sample inside the sample compartment).

1. Pressurize the exchange compartment:
    a. **Press and hold** (for about 1 second) the **VENT** button on the exchange compartment.
        i. The button starts to blink and you hear some sounds.
        ii. In a few seconds, the door between the exchange compartment and the sample compartment closes. You can observe this by ear and by looking at the bottom right corner of the SEM Monitor.
2. **Open the latch** as soon as you hear the click.
3. **Open** the exchange compartment **door** (it should open almost by itself).
    a. You do not need to wait until the pumping has stopped.
    b. The compartment will continue purging for a fixed amount of time. You do not have to wait until it stops and you can evacuate it as soon as you like.
4. Now you have the sample compartment open and ready for loading/unloading the sample holder.

Inserting a sample

Precondition: There is **no sample** inside the specimen chamber and exchange compartment is **in vacuum**. First check that **HLDR light is off** on the exchange compartment (i.e. there is no sample inside).

1. Move the **stage** to **exchange position**:
    a. Click **Exchange Position** on the SEM Monitor.
        i. If button is not visible, click "Specimen" from the rightmost edge of SEM Monitor.
        ii. Make sure that **EXCH POSN is lit** on the exchange compartment, before proceeding.
2. Bring the **exchange compartment to atmospheric pressure** by following procedure in section “Opening the sample exchange compartment”. Quick notes:
    a. Press and hold **VENT** for ca. 1 sec.
    b. **Open latch**. Wait.
    c. **Open** chamber **door**.
3. **Put on gloves** if you do not have them already on.
    a. Parts that are in contact with the vacuum should be kept absolutely clean. If you have touched some part, clean the part with ethanol (not acetone).
4. **Insert holder** to the specimen chuck:
    a. **Slide** the specimen holder into the specimen chuck **along the arrow** direction on the specimen holder.
5. **Check** that the **O-ring** seal on the door is OK and wipe it with a clean glove if needed to get rid of any dust.
    a. If the ring is really dirty, wipe it with ethanol or isopropanol (do not use acetone or methanol).
6. **Close** the chamber **door** and **secure** it with the **latch**.
7. **Evacuate** the compartment by **pressing and holding EVAC** (for about 1 second). The light will start blinking.
    a. **Wait** until the **light stops blinking** and the door separating the exchange compartment is closed. You can observe this from the bottom right part of the SEM Monitor.
8. **Insert** the **sample holder** inside the microscope
    a. Refer to section “Operating the rod” if in doubt.

9. A popup window should appear on the SEM Monitor. Now select the appropriate holder and set the offset value.
    a. If popup does not appear, take out the holder and insert it again.
10. **Wait** until the **vacuum level** reaches $9.6 \cdot 10^{-5}$ Pa (if that is not possible, wait at least until $5 \cdot 10^{-4}$ Pa).

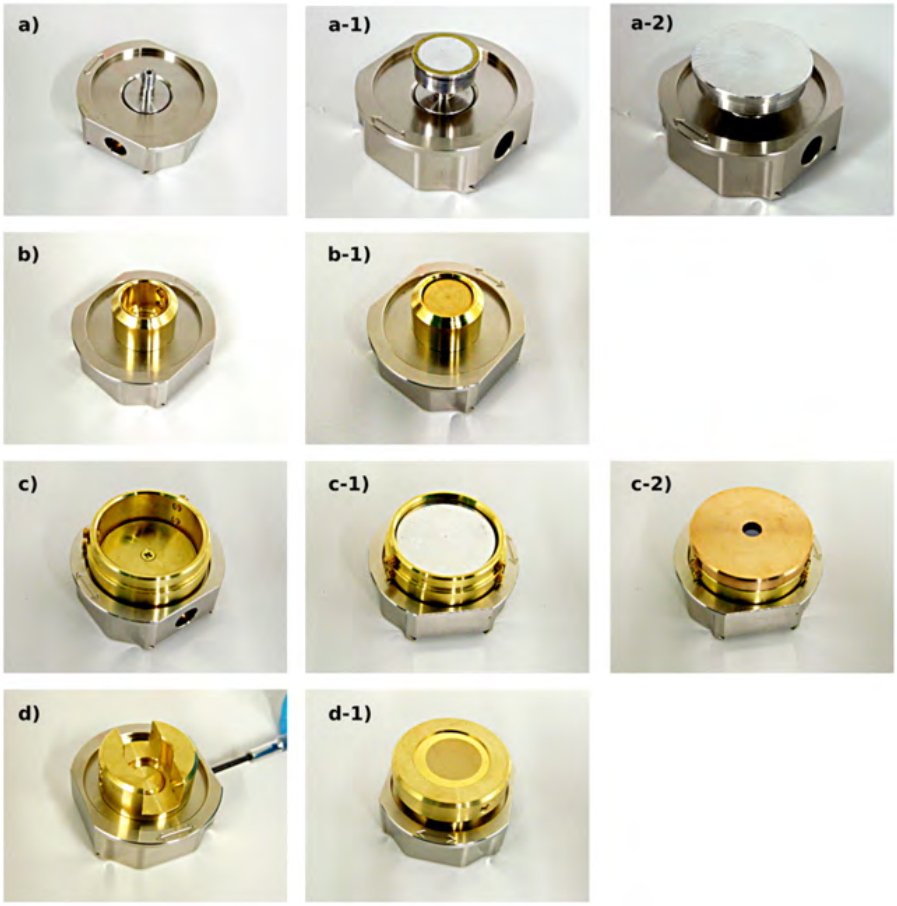
Taking out sample

Precondition: There is a sample inside the microscope and exchange compartment is in vacuum. First check that **HLDR light is on** on the sample exchange compartment (i.e. there is a sample inside) and **EVAC light is on** and not blinking.

1. Click **Observation OFF** to turn off acceleration voltage.
2. Click **Exchange Position** to move the sample holder to the exchange position.
    a. Make sure that **EXCH POSN is lit** on the exchange compartment before proceeding.
3. **Bring the sample** to the **exchange compartment** by **operating the rod**.
    a. Refer to section "Operating the rod" if you are not absolutely certain how to do this.
4. **Pressurize** the **exchange compartment**:
    a. **Press** and hold **VENT** for ca. 1 sec until it starts to blink.
    b. **Open** securing **latch**. Wait.
    c. **Open** chamber **door**.
5. Now you have the sample compartment open and you are ready take out your sample. If you are done with the imaging, just close the exchange chamber and evacuate it otherwise continue with inserting a new sample. Do not leave the chamber open for a long period of time, but evacuate it if needed.

Sample holders

Sample holders consist of a base part and an adapter part (show on the figure right). There are three different adapters for different specimen stubs shown in the figure below (a, b, c, d).



The most basic holders are the 12.5 mm (b-1) and 25 mm (c-1) aluminum *"JEOL" stubs*. They should be used whenever possible. Stubs should always be available at the sample preparation room, but you can also order your own ones e.g. from *EMS* (order numbers 75730, and 75700). The use of regular holders is included in the operation price of the microscope.

Also *"mini-stubs"* are available for use with a provided adapter. They are preferred for small samples. They can be ordered from *Ted Pella* (order numbers 16180, and 16181).

For special occasions, a *Hitachi adapter* (a) can be used. Special care must be taken when using these holders, because they lack some safety features.

Ask administrators, if you have special requests for holders. There are also different kinds of *cross-section holders* available. Ask the administrators for more information.

Attaching adapter to base part

a) Make sure that parts are not dirty, clean if necessary.
b) Place adapter on the base part.
c) Tighten screw on the base part lightly.

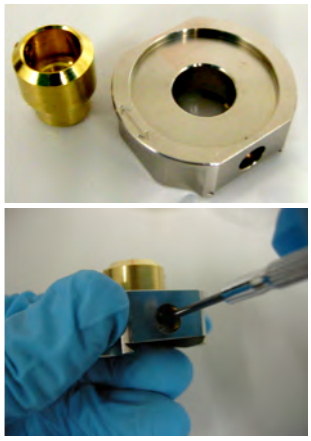


Figure. Attaching adapter to base part.

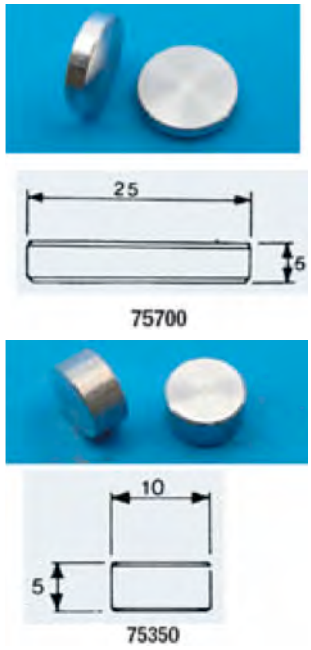
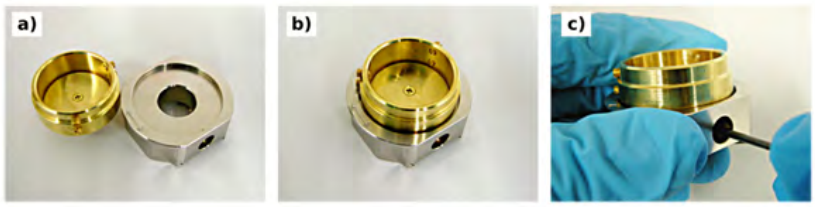


Figure. 12.5 mm and 25 mm "JEOL" stubs.

Figure. "Mini-stubs" and 12.5 mm adapter.

Figure. Cross-section holders.

Sample height



Align the top of the sample with the groove inside the JEOL adapter part. Use the screw on bottom to raise of lower the sample. When using another holder, make sure that height from table top level is exactly 25 mm.

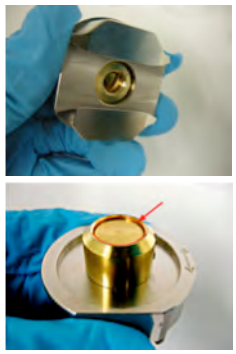


Figure. Align top of stub with the groove on the sample holder.

Coating

For non-conductive samples a coating is usually needed for observation in SEM. This can be easily performed by using sputter coating of gold, platinum, or gold-palladium. There is a sputter coater at NMC, which can be used for this purpose. Resolution limiting factor is the grain size, which is usually 5-20 nm depending on the conditions of sputtering.

Also carbon coating can be used to make samples conductive. It is an appealing method, when doing X-ray analysis. It creates a very uniform layer without noticeable grains.

Basic microscope operation

Basic concepts

Working distance and Z value

Working distance (WD) value sets the effective focal length of the objective lens.

Z height value sets the distance of the (supposed) surface level of the sample from the objective lens.

These two values are equal, when top of sample is aligned with the top of the holder (ie. 25 mm high from table level, see figure). **WD > Z**, if your sample is lower than the correct level and vice versa. If **WD < Z** you need to set the Sample Offset value accordingly.

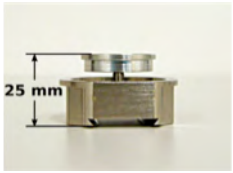


Figure. Sample height should be exactly 25 mm measured from table top level.

Sample offset

The height of the top level of the sample measured from table top level should be exactly 25 mm. The sample can be set also 0-4 mm higher than the nominal level, but then the Sample Offset value has to be set after inserting sample. It is located at the bottom of the sample holder select window, which pops up automatically after holder insert.

Acceleration voltage, emission current, probe current…

The first thing to think about when starting imaging is the selection of **acceleration voltage**. The choice depends on the type of the sample. See table below for some examples.

Sample	Observation condition	Notes
Gold particles on conductive surface	5-30 kV, probe current at ca. 10, working distance 1.5-8 mm	
Coated porous polymer	1-5 kV, probe current 6-10, working distance 4.5-8 mm	
Uncoated polymer	0.5-1 kV, probe current < 8, gentle beam mode, working distance ca. 8 mm	
Coated biological sample	1-5 kV, probe current ca. 10, working distance 4.5-25 mm depending on feature size	
Uncoated paper	1-2 kV, gentle beam (GB-L) mode, working distance 4.5 mm, probe current 6-10.	
Coated paper	5 kV, working distance 4.5-25 mm, probe current ca. 10	
X-ray analysis of conductive sample	15-30 kV, working distance exactly 8 mm, high probe current	Retractable BEI detector inserted
X-ray analysis of poorly conductive sample	5 kV, working distance exactly 8 mm, probe current as high as possible	Retractable BEI detector inserted

The **emission current** is the current drawn from the emitter. Set it always to 10 µA.

Probe current is the current directed at the sample. Higher values give better signal to noise ratio, but cause more charging artefacts in poorly conducting samples. Value of 8-10 is usually a good choice.

Aligning

Usually the microscope is aligned well enough for micrometer scale operation. In this case, only focusing is necessary. For higher magnification work, the electron beam needs to be aligned and astigmatism of the objective lens has to be corrected.

Focus

The first level of aligning is always focusing. Focusing is done using the **FOCUS** knob on the operation console. Clockwise rotation is under focus (weaker lens) and counterclockwise is over focus (stronger lens).

If possible select some feature, which you can use in the magnification range from ca. 1000 to 20000.

Start from a low magnification and when you get good enough image move on to higher magnification for focusing. It the alignments are really off, you might not get a clear image at all.

Beam align

Beam align is always done at probe current 8. Select the correct probe current value from the software.

Press ALIGN on operation console. The image starts to move on the screen. Use the X and Y knobs to minimize the movement. Press ALIGN OFF (STIG) button when image has stopped. Repeat for magnifications up to ca. 20000. Focus the image whenever necessary.

Astigmatism correction

To correct the objective lens astigmatism press the STIG button on the operation console (it is usually already selected at this point). Move on to a **spherical feature**, which you are able to observe at magnification 10000 or more.

Move the FOCUS knob so that you go from underfocus to overfocus and back several times. When you have astigmatism, the image gets elongated in diagonal directions when moving around the focal point. Select the focal point where no elongation occurs.

Adjust the X and Y knobs so that you get the clearest image possible. Focus whenever necessary.

Other corrections

There are also other alignments, such as source align, condenser lens astigmator, low magnification center, and stigmator center corrections. These values should not usually be changed and their use is not described here.

Problems and troubleshooting

Answers to common problems

I want to use USB stick to transfer my files!

You can transfer your files to a USB stick from the small computer at the back wall. You'll find your files under the network drive *Harley*.

Help! There is no image.

Follow the checklist to find the cause:

1. Are your Z and WD values the same? If not press WD to set correct distance.
2. What detector are you using? If WD<8 mm you usually do not get image with LEI detector; and if WD>8 mm SEI gives only static noise. LM mode should work fine in this case.
3. What is your probe current value? If it is low, try increasing it.
4. If none of the above is true, try resetting alignment. Go to Alignment panel and click Reset All.
    a. In a really bad case the source alignment has gone bad. Contact an administrator to align it.

If there is no image when starting operation

First, press ACB (auto contrast & brightness). If you even see some static noise, you only need to find the correct focal point. See previous section.

In case you have completely black screen when you start imaging, follow the list until you have image.

1. Restart of SEM software:
    a. File->Exit to go to login screen.
    b. Close login screen from Exit button.
    c. Wait one minute.
    d. Start SEM_Monitor software.
    e. Log in as Guest.
2. Restart computer:
    a. Close SEM software.
    b. Save Excel log book and exit.
    c. Restart Windows.
    d. Start SEM software and Excel log book.
3. Restart operation console:
    a. Read instructions below.

If vacuum breaks during sample exchange

Vacuum usually breaks if the lever is pushed or twisted during the sample insertion. The computer will raise a maintenance window showing error messages. The microscope will automatically shut down
some parts and the vacuum pumps have to be restarted. Bring the microscope **back to its normal condition** before proceeding. For example, **lift the rod** back to its **upright position**.

1. **Locate** the two **VAC SW** buttons below the table. There are also MAIN SW buttons, but do not touch them.

2. **Shut down vacuum pumps** by pressing **VAC OFF** (0=OFF, 1=ON) button. The pumps should now stop, if they were not shut down already.
3. **Wait** a moment and **restart pumps** by pushing **VAC ON** button.
    a. There is a 20 min timer for starting the pumps so you will have to wait at least 20 minutes before proceeding.
4. After all of the error messages have disappeared from the display, you can continue operating.

Turning off computer

1. Log out from the SEM software (File->Exit).
2. Save the Excel log book (File->Save or Ctrl-S).
3. Close the Excel log book.
4. Select Shutdown from Start menu.

Powering on computer

1. Start computer from the power switch.
2. Log in as SEMUser (password: SEMUser).
3. Start Excel log book by double clicking “SEM Usage Log” on the right monitor desktop.
4. Start SEM software by double clicking SEM_Monitor.
5. Log in as Guest.

Restarting operation console

1. Turn off computer.
2. Press OP SW OFF from below the table.
3. Wait ca. 10 seconds.
4. Press OP SW ON.
5. Turn of computer.
6. Wait a couple of minutes before starting SEM_Monitor software.

Contact information

Microscope administrators

Juuso Korhonen
Molecular Materials
juuso.t.korhonen@tkk.fi
+358-50-344 1892

Reservation system, user training, maintenance.

Antti Kaskela
Nanomaterials Group
antti.kaskela@tkk.fi
+358-50-301 2102

User training, maintenance.

Nanomicroscopy Center

Professor **Janne Ruokolainen**
Molecular Materials
janne.ruokolainen@tkk.fi
+358-9-470 22167

Reservation system administrators

Antti Nykänen
Molecular Materials
antti.nykanen@tkk.fi
+358-9-470 26067

Bugs and issues in ERS.

JEOL

Pekka Koskinen
Engineer TEM/SEM/EP
+358 50 528 7664
+358 9 812 90 350

JEOL service contact in Finland.

Jalo Janhunen
Chief Engineer
EP/FEGSEM
+46 7 398 25 064
+46 8 564 87 605

Andrew Yarwood
Application Specialist
TEM & SEM
JEOL (U.K.) LTD.
+44-1707 377117 (tel.)
+44-1707 373254 (fax)
andy@jeoluk.com

Training on SEM.