ThermoFisher Scientific – LUMIS EBSD – Instructions (v2 revised 24.6.2025)

Disclaimer: *These instructions are meant for users who have received initial guidance from supporting team member(s) (Santeri Kurkinen).*

Prior going through these instructions, the sample preparation by grinding and polishing is assumed to be done in advance.

Cleaning the sample surface:

1. Rinse the sample with ethanol and dry the sample with pressured air.
2. Open the CO_2 bottle and use a snow-jet to clean the metal surface.
   a. Keep the sample under the air flow during the snow-jet cleaning process
   b. Avoid freezing the sample since it could result in deposition of impurities from the air to the sample surface
      i. Long duration of snow-jetting generates static electricity build-up. Make sure that you can conduct the static charge away from the sample. Static charge could cause small scale arching.
3. Make sure that at the end of the cleaning process the sample is dry and at the room temperature. Leftover ethanol vapors could contaminate the inner parts of the SEM.

Attaching the sample to the holder:

1. EBSD needs to be measured at 70° angle. There is a specific sample holder for that purpose (2 different sizes currently available depending on your sample size).
   a. There is a possibility that the sample holder might be in slightly incorrect angle relative to the base of the holder (i.e. they are not parallel)
2. Although the sample is attached to the holder by tightening the screw, attach a small piece of carbon tape to the sample to ensure contact with the sample holder (conductivity)
   a. If you are using samples which are in clear resin, please ensure the conductivity of the sample with copper tape.
3. Check the height of the sample. Sample's height is measured either from the holder or the sample itself whichever is the highest position.
   a. When inserting the sample inside the SEM, it's better to have too much space than too little. For EBSD, the highest point is often on the side. Therefore, when inserting the sample inside, the highest point does not go directly under the safeguard.
4. In practice, choose the 51mm sample holder for EBSD samples.
   a. There is a possibility that the screw of the sample holder might be outside the radius of 51mm sample holder but in general, this does not cause any problems.

ThermoFisher Scientific – LUMIS EBSD – Instructions (v2 revised 24.6.2025)

i. Make sure that the screw is pointing on the side (left or right), not up (screw is not the highest point of the sample)
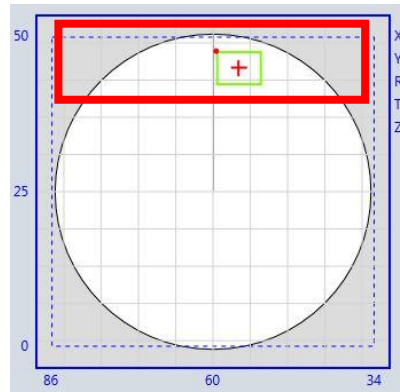

Set-up for the measurement (SEM):

1. Turn on the beam normally.

2. Parameters that you could use in the beginning are V_{acc} : 20 kV and spot intensity: 90

   a. Aperture can be changed to 2.

      i. *Depending on the material, you might need to use alternative conditions (too high x-ray formation from the material → Detector becomes saturated, and software begins to fail)*

3. Since the sample surface for EBSD is tilted, turn on the tilt compensation (Upper right corner of SEM software "Etc." –> "Tilt compensation"

   a. Turn on the "*Dynamic focus*" and make sure it's 70 degrees.

      i. Tilt-Mag. Compensation is not required.
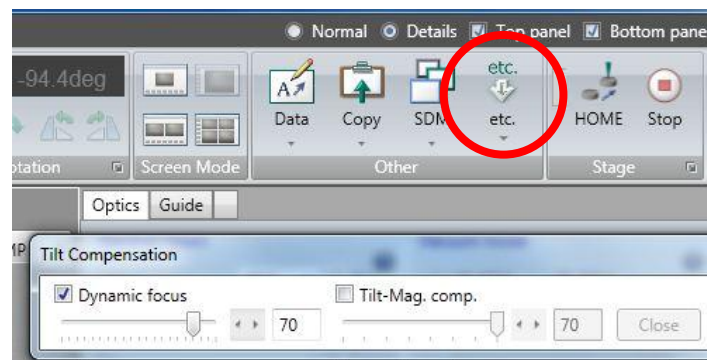
4. Do all necessary adjustments to the image that it looks good (Alignment(s) etc.)

5. Locate the area of interest from your sample surface.

   a. Use a BSE-comp detector for this purpose

   b. Adjust the magnification to suitable level (what you prefer)

   c. Try to have the target area in focus, good as possible.

      i. *If measuring large areas, the edges can be slightly out of focus, this could affect the measurement little bit.*

   d. WD (working distance) should be approx. 14mm (± 3mm).

24.6.2025 Santeri Kurkinen

**ThermoFisher Scientific – LUMIS EBSD – Instructions (v2 revised 24.6.2025)**

For EBSD, the sample surface can be found from this region (default)

6. Whenever you have successfully found the place which you want to measure you can leave the SEM as it is.

Set-up for the measurement (Pathfinder software; EBSD)

Windows's clock needs to be in US format (AM/PM), otherwise the software is not capable of communicating with the detector.

1. Open the Pathfinder software, create a new project under your name in EBSD main folder.
   a. EBSD raw data files are large. To reduce the risk of losing the data due to software crashes, create a new folder/project for every measurement.
2. From Pathfinder, select the EBSD.
   a. To adjust the settings for the measurement, select "*Setup*"
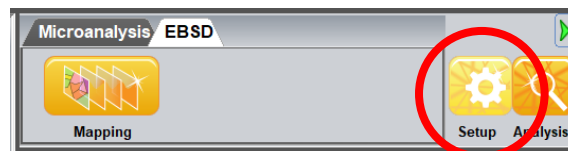
3. First, insert the camera next to the sample by pressing "*In*" from camera controls.


   a. Pressing "Stop" might cause camera to lose its position during the insertion. It should not be used by default if not needed.
   b. When the camera is in place, the slide position should show "168"

24.6.2025 Santeri Kurkinen
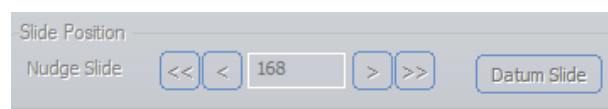
**ThermoFisher Scientific – LUMIS EBSD – Instructions (v2 revised 24.6.2025)**

Occasionally, the software might ask you to datum the slide (it needs to verify and calibrate its position). This can be done by simply pressing "Datum Slide" button.

4. Second, the lens position needs to be selected (pos. 2 is the default position). Changing lens position does not move the camera itself.
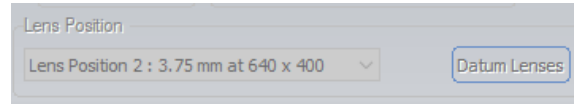

Same thing than with the slide, lenses might need datuming. It can be done similarly to the slide datum.

5. When the camera is inside, avoid moving the sample. The camera is in proximity to the sample and moving the sample could cause damage to the screen.

6. Check that the stored rate is ok. Too high values lead to sluggish behavior of the software and ultimately the measurement will time-out and crash with high certainty.

HV: 20.0 kV | Mag: 251 x | Stored Rate: 313 kcps | Dead Time: 49 %
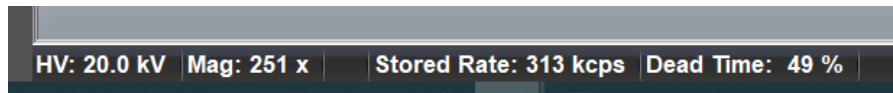
Rates around 300k or less (if the pattern is good) are okay. Dead time of the detector should stay at 50% or less. Too high rates are indicated by Dead time being > 50% and it becomes yellow.

7. Obtain the image of your sample that will be mapped.
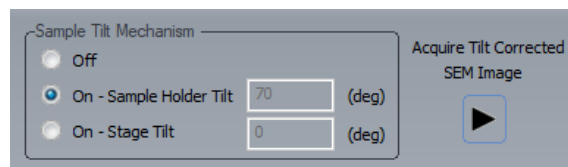
   a. Since the sample is tilted, select the "*On – Sample Holder Tilt*"
   b. Press "*Acquire Tilt Corrected SEM Image*"
        i. If you don't acquire image, check the following:
            - Chamber camera is turned off (IR lights interfere with BSE detector)
            - Brightness/contrast is at suitable level

8. You should now be able to see pattern of your sample on the "Uncorrected" view.

24.6.2025 Santeri Kurkinen

ThermoFisher Scientific – LUMIS EBSD – Instructions (v2 revised 24.6.2025)

Uncorrected
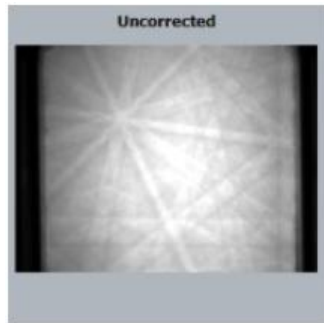
a. If not, adjust the exposure time and gain so that you see clear, recognizable pattern.

b. If still not, then adjust the beam properties from SEM (Higher voltage, higher spot intensity or larger aperture).

c. Histogram also should have peaks throughout its whole scale.

Histogram
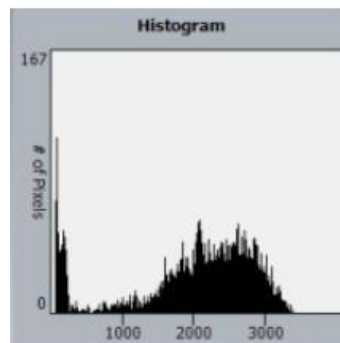
i. Red areas on the uncorrected image are signs of too high exposure → Reduce the exposure time and/or gain.

9. When the pattern is recognizable, collect the background information.

a. As a default: Division and local background are good starting points (other options for dynamic removal can be tried out to find the best option)

i. Make sure to collect a good number of images for background. Then the average value for background is okay (e.g. "*Number of images to average: 100*")
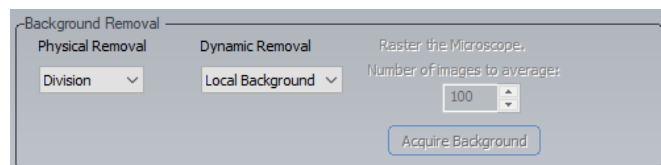
10. Go through the sample area and make sure that the pattern is recognizable and software shows the orientation of the crystal in most areas.

a. If yes, you can proceed to mapping

24.6.2025 Santeri Kurkinen

   i. Sometimes it might be challenging to get good patterns throughout the whole sample. If the software can index it, then it's fine.

b. If not, fine adjust the beam (focus, astigmatism etc.) from SEM but don't move the sample.

   i. If still not, try fine adjusting the pattern center for your desired crystal structure from the "*Band detect and indexing*".

c. Note: Make sure that you have selected the correct crystal structure (FCC, BCC, HCP etc.) This can be changed from "*Add/Remove Crystals*". Also, during the post-processing, this can be changed.
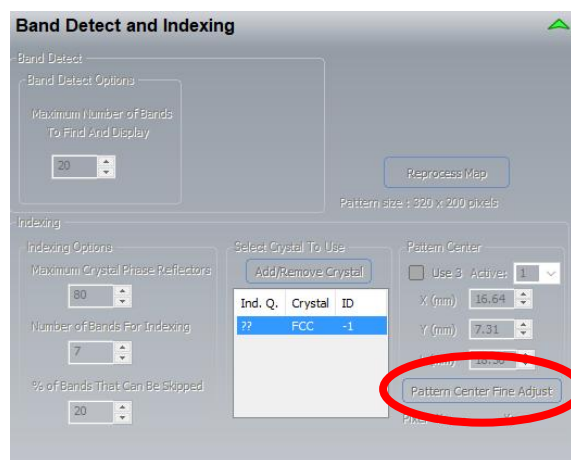
If the indexing is still bad, the bigger calibration of pattern center might be required. In these occasions, please contact Santeri.

Mapping:

1. Adjust the settings for the mapping

2. Make sure that the "*Save pattern on disk*" is checked

   a. Without the pattern data, the post-processing data is impossible, and the information is obtained as it is.

3. Select a suitable resolution for your needs. Measurement time for one map increases when the resolution goes up.

   a. Measurement time is affected almost solely by exposure time. Make it short as possible during the earlier steps. Already cutting the exposure time by 10ms can result in significant change in the total duration of the mapping.

4. When these settings are ok, start data acquisition by pressing "*Start map*"

   a. Measurement can be left after it has started and maps begin to form.

24.6.2025 Santeri Kurkinen

     i. Windows can be also locked during the measurement. Software does not crash anymore during the measurement like it used to do.

<u>Finishing the measurement:</u>

1. After the measurement, save the data from the "File" → "Save"
   a. This moves the acquired data away from the temp folder.
2. Move the camera back to it's home position by pressing "Out" from setup page.
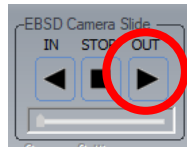
DO NOT TOUCH SEM BEFORE THE CAMERA IS BACK TO ITS HOME POSITION

3. When the camera is back to it's home position (slide position 0), you can turn off the beam and remove the sample from SEM as usual.

<u>Post-processing the data:</u>

Data can be treated to certain extent with the pathfinder software after the measurement. I won't include instructions for this part here, since so many things can be done. Separate guide for this will be done later.

24.6.2025 Santeri Kurkinen