

User Instructions For The Tescan Mira3 FESEM 10/7/2015

Loading A Sample

Always use gloves when handling samples and SEM sample studs.

Make sure the stage is in the home position after loading your samples.

After loading your sample, close the chamber door and hold it while clicking the pump icon. Keep holding the door until you see that the chamber and column are actually pumping down. Look for the red bar increasing to the right and the pressure dropping. Once you see that the chamber is pumping you can release your hand from the door. As a rule of thumb, for every minute you leave the chamber door open there will be a minute of pump down time, especially during periods of high humidity. Wait until the column and chamber pressures are $< 2 \times 10^{-4}$ torr before opening the gun valve (Beam On). The indicator bars have to be green to open the gun valve.

If the stage calibrate button is flashing, click it and let it finish calibrating before loading your sample.

Tilt angle = ± 70 degrees

Setting The Working Distance

When decreasing the working distance (raising the stage - WD & Z field), the sample **must be in focus** to prevent the stage from crashing into the objective lens. Use Resolution mode and a magnification of at least 1000 times. There is a touch alarm that will sound and should stop the stage, but if your sample is not electrically conductive it will not stop before crashing into the pole piece. Also, if your sample charges excessively the focus will not be correct and the stage will not stop before crashing into the pole piece. As a precaution with non-conductive samples, keep the cursor on the stop button so you can stop it from hitting the objective lens pole piece if it doesn't stop. Warning: In Beam Deceleration Mode (see below) the touch alarm is disabled.

To safely set the working distance, go to Resolution Mode and to a magnification of at least 1000 times; focus the area of interest as best you can and then change the working distance in WD & Z. After this adjust the objective aperture by using the manual column centering wobble function, focus again, and then stigmat and focus. The best working distance for the SE detector is 10 – 8 mm. (You can get an image using the SE detector at smaller working distances by adjusting Auto Brightness and Contrast, but the image quality will not be as good because of the geometry.) The best working distance for the InBeam SE detector is 5mm – 2mm, depending on surface planarity. The smallest working distance for the chamber backscatter detector is 6mm. You can use the InBeam backscatter detector for smaller working distances.

If you load more than one sample that have different heights, lower the stage before rotating to the next sample if it is higher than the one you are looking at. This will avoid the sample hitting the objective lens.

Increasing Depth of Focus

If your sample has varying height and you want the top and bottom to be in focus at the same time, use Depth Mode, lower magnification and a larger working distance. Depth Mode selects a small aperture to get a smaller convergence angle.

Using the InBeam Detector - Focusing and Aligning (same procedure for SE Detector).

- 1) Focus (WD knob) your sample in Resolution mode. Set the working distance (WD & Z field) to 9mm. If you feel comfortable you can go straight to 3mm.
- 2) Once at a working distance of 3mm, switch to the InBeam Detector
- 3) Adjust contrast and brightness.
- 4) **Objective Aperture Centering**. If the image moves when focusing, the objective aperture is not aligned (Objective Wobble). Go to Manual Objective Centering (second to bottom icon and vertical column next to acquisition window). A screen will open for Objective Centering. Click Next and a box will open on the

image. The image inside the box will be moving horizontally, vertically or at some angle (wobbling). Adjust the vertical movement with the y-component knob (right knob) on the control panel. Adjust the horizontal movement with the x-component knob (left knob). Note: the middle knob is the step size control (coarseness). The object here is to get the image pulsing in and out of the screen, that is, there is no horizontal or vertical movement. If either of the Objective Centering x and y values (top right of menu) is 100% and you cannot adjust the wobble, you must do a gun alignment. Go to the bottom right side and select Auto Gun Alignment from the drop-down menu. This will run automatically and take several minutes to complete. Once this is finished start the Objective Centering wobble again and from the top right click reset before adjusting the wobble again. After the aperture is centered the stigmators will have to be adjusted -> next step

- 5) **Stigmatate.** First adjust the focus by going through the focal point on both sides of focus. You will notice that on either side of the focal point the image will be stretched out, but the stretch will be 90 degrees to each other. Focus so that it is in between these two extremes even though it might be blurry. Adjust the y-direction by holding down the F11 key and rolling the track ball vertically. Adjust the x-direction by holding down the F12 key and rolling the track ball horizontally. Adjust these x- and y-stigmators until the image is sharp. If there is not stretching of the image as you go through both sides of focus then you have successfully eliminated the astigmatism. Finally focus for the best image.

You have to do the objective alignment and astigmatism correction whenever you change voltage, working distance or beam intensity.

Spot Size

Minimize the spot size to improve the resolution if necessary.

Beam Current

For low magnification and EDS work use a beam current setting of 10. For high-resolution work use a lower beam current setting.

Beam Deceleration Mode For Voltages from 0.2KV to 1KV

When using BDM to work at low voltages, make sure your sample is grounded to the sample stud. Electrically floating samples will charge and can subsequently arc to the objective lens locking up the SEM. You will have to turn off the beam, turn off BDM, go to home position and then turn the beam back on. Also, your image quality will not be the best it can be. The minimum working distance in BDM is 3mm.

To align BDM for low voltage work, set the voltage to 5KV plus the BDM voltage at which you intend to work. Do the normal alignments at this voltage in the area you want to investigate. Go to BDM mode and change the high voltage to the desired voltage low voltage. Fine-tune the alignment. It is done this way for Basic users because the stage is at fixed bias of -5KV.

Here is an example. If you want to work at 0.2KV in BDM, set the high voltage to 5.2KV, align, go to BDM and then set the voltage to 0.2KV.

Make sure the Bruker EDS detector is retracted before using BDM to prevent a discharge that could ruin the EDS detector.

Auto Gun Alignment

The auto gun alignment needs to be done when changing voltages outside of one of the four voltage ranges: 0.2 KV – 3KV, 3KV – 7KV, 7KV – 15KV and 15KV – 30KV; or when the objective centering has reached end of range (see Objective Aperture Centering above). Go to the bottom right side and select Auto Gun Alignment from the drop-down menu. This will run automatically and take several minutes to complete. Once this is finished start Objective Centering.

Ending Your Session

When your are finished using the Mira, lower the magnification, set the stage to home position, select the SE detector, go to Wide Field Mode, go to the lowest magnification and finally click on Auto Brightness-Contrast.

Bruker Energy Dispersive Spectrometer

To launch the EDS application, click on the Esprit icon on the desktop of the monitor on the right-hand side. The optimal working distance for EDS is 15mm. There is a small icon on the upper right side of the Esprit application that you click to automatically insert the detector. Please retract it when you are finished. When you click Acquire to take a spectrum the LED light on the SEM chamber should shut off. If it doesn't, right click on the camera video screen to turn it off.

There is a remote computer in the lab where you can analyze your EDS data at a later date without disturbing a user. Login into EDS-User. Open the Esprit software.

A beam voltage 15KV or 20KV is good for most EDS analyses. You want an overvoltage of approximately 3 times the highest energy line you want to use of the element with the highest atomic number. For example, the $\text{CuK}_{\alpha 1}$ x-ray energy = 8.04 KeV, so set your beam voltage to 25KeV. Ideally, you want a dead time around 35%, but get the maximum you can, but do not go above 50%. If you need more counts increase your beam intensity.

EBSD

To use EBSD mount your sample using the pre-tilted holders. These are at a 70° degree tilt. Choose -70° tilt correction from the drop-down menu. You can use Resolution, Depth or Field Modes. Depth Mode works best. For low magnification work use Field Mode. Always align Resolution Mode before aligning the other modes. You will find aligning Field Mode in the tilted sample tedious and difficult but is necessary to get undistorted EBSD images at low magnifications (<2000x).

Typical voltages are 15 - 30 KV and absorbed currents 3-8 nA.

Use at least 400p of scan resolution and 400 x 400 EBSP resolution to avoid too many zero solutions.

Insert the EBSD detector to DD=25 mm, then proceed slowly, going a mm at a time. You should be able to get to 17 mm for most samples unless your sample is a cm tall or more, than the DD distance will be larger. Watch the detector to sample distance in the small monitor on the desktop. Be careful not to hit the EBSD screen to avoid scratching it. Usually the foreshatter or backscatter detectors will hit first and compress the screen head. If this happens, you are too close -back off a mm.

If you have a sample that is not in the database ask me and I will show you how to enter it. You will need the space group, lattice parameters and atom positions. You can get these from published XRD data.

THE FIRST TIME YOU USE THE MIRA AFTER THE INITIAL TRAINING, USE IT DURING REGULAR WORKING HOURS, WHEN I AM HERE. DO NOT ALLOW OTHERS WHO HAVE NOT BEEN TRAINED ON THE MIRA TO USE YOUR MIRA OR CORAL ACCOUNTS. ONLY THE CCMR FACILITY MANAGERS ARE ALLOWED TO TRAIN USERS. IF YOU HAVE AN ISSUE THAT YOU CANNOT FIX CALL OR EMAIL ME (djw326) OR ANY OF THE OTHER FACILITY MANAGERS.