



This guide obtains just brief information. In case you need detailed instructions, read chapter "Getting Started" in TESCAN MIRA3 OEM manual. It is recommended to read the **Safety Instruction and Basic Troubleshooting** manual prior to your work.

TESCAN MIRA3 SEM

Content

Getting SEM Image	1
Mira TC Workspace	4
Guide for Low vacuum "UniVac" mode	5
Scanning transmission electron microscopy (STEM)	7
EDS (EDX) – Energy Dispersive X-Ray Spectroscopy	9

Getting SEM Image

- 1. Turn on the SEM monitors. Left screen is for SEM, right screen is for EDS. Both monitors and computers are interconnected via Mouse without Borders software.
- 2. Type your username and password in **MiraTC Log in** screen. Wait until self-test is done. Contact the guarantor immediately if an error occurs.
- 3. Check that all the detectors are in the parking position and vent the microscope by using the VENT button on the Vacuum panel. Wait until the pressure is at the atmospheric level and sound of N2 infill stops. Chamber door should be opened by N2 pressure. The gun pressure should be always in green status. If not, contact the guarantor immediately!

Vacuum	- ?	
Gun Pressure:	1.9e-007 Pa	
Column Pressure:	2.3e+003 Pa	
Chamber Pressure:	1.3e+003 Pa	
Vacuum off.		
STANDBY VENT	PUMP	

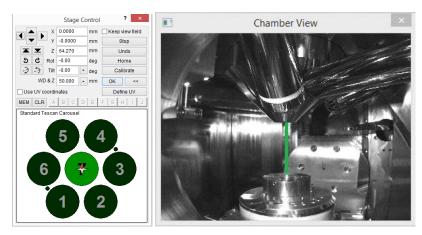
4. Select the position for uploading your sample by clicking on the appropriate number button on the carousel in the **Stage Control** panel.

Wear gloves while manipulating inside the SEM chamber and with samples! Do not touch the carousel or load the samples until carousel fully stops its motion. Carefully open the chamber door and load your sample.

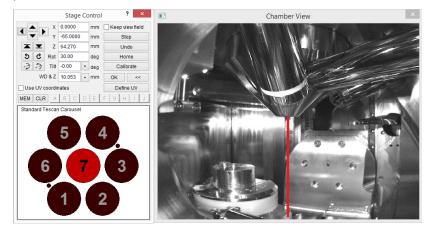
Note that carousel has two statuses indicated in different colours:

The positions are green: When choosing and clicking on the position, carousel will load the position right in the electron beam axis while keeping the actual Z-axis distance.





The positions are red: When choosing and clicking on the position, carousel will unload. This is active only at low pressure.



- 5. Carefully close the chamber door and press **PUMP** button on the **Vacuum** panel. Wait until pressure indicator bar turns from red to green.
- 6. Click on the appropriate number button on the carousel in the **Stage Control** panel where you have placed your sample to move it under the SEM objective.
- 7. Click on the **BEAM ON** button on the **Electron Beam** panel to turn the electron beam on. **NOTE:** The emission is always running but the gun is physically separated from the chamber and the column when these are vented to maintain the high vacuum of the gun.

The button is active only if the Column and Chamber pressure bars are green.

						Vacuum	
					Gun Pressure:		1.8e-007 Pa
	Electro	n Beam		?	Column Press	ure:	2.4e+003 Pa
Emission:	203 µA	HV:	1.00	kV	Chamber Pres	sure:	1.3e+003 Pa
BEAM	ON	1.00 (0.2.	3.0) kV	~	Vacuum off.		
Adjustment >>>			STANDBY	VENT	PUMP		

8. Magnify and focus properly at your specimen to lock the working distance (WD&Z). Use Degauss function to be sure you stay in focus. If not, repeat the procedure. Magnify at least to 10kx and repeat the focusing procedure.



- 9. Go to WD 9 mm using **WD&Z** function in the **Stage Control** panel. It is recommended to move in two or three steps when the change in WD is large. Place the cursor at **Stop** button and watch the **Chamber View** (IR camera) when the stage is moving. Hit the **Stop** button immediately when it seems there might be a collision!
- 10. Choose HV you will use: select **HV** in the **Info Panel** and type desired value of the accelerating voltage (e.g. 20 kV) to **Pad** panel.
- 11. Check that you are in **RESOLUTION** scan mode and you are using **SE detector** (Uncheck the **Continual Wide Field** function).
- 12. In the case you have chosen voltage different than 2, 5, 10 or 20 kV: Check there is no charging or Faraday cup in your image or move to ADJ sample or clean SEM stub, focus properly and set the minimum magnification. In the Electron beam panel click on Adjustment >>> and choose Auto gun centering function. It will take approx. 3 minutes to finish the procedure.
- 13. Click on **Spot Size:** in the **Info Panel** and type 0 value to the **Pad** panel. Minimum spot size and optimum beam intensity will be set automatically for current HV and WD.
- 14. Placing the cursor over the **SEM Scanning window** and clicking the mouse wheel move area of interest into the centre of the image.
- 15. Set the desired view field and focus the image. In the case that the image is moving during focusing, it is necessary to check the centering of the objective. Go for Adjustment >>> and Manual Column Centering or press the icon. (Hold down the F12 key to change only X movement at the Trackball, and the F11 key to change only Y movement). The image should not move in X or Y axis, it should be stationary and only "flashing".
- 16. Correct astigmatism using the **Stigmators**. In case that the image is moving when stigmating,, click on **SEM** in the main menu and choose **Stigmators A/B centering**. Follow the procedure.
- 17. Select a suitable scanning speed (at least 4, but 5-7 is recommended). Clicking on the **Acquire** button saves the image.

When finished:

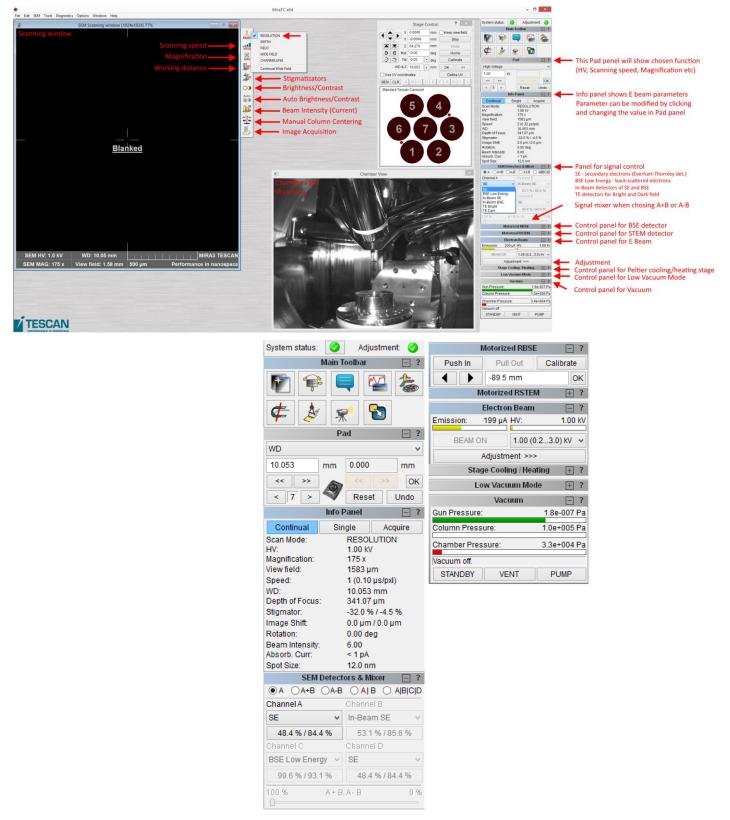
18. Once finished, set the HV to 5 kV, retract all detectors to parking position, withdraw samples and evacuate chamber. Put the SEM to standby mode (unless the next user is already present) and log off. Leave MiraTC - Log in screen on. Turn off the PC monitors but leave the PCs on.

Transfer your data via internet. Flash drives are not allowed. BUT users can use for example Google to send and share data. Every BUT student/employee has access to Google drive with large storage capacity via BUT login and BUT password. You can access the drive from anywhere and download your data. Non-BUT users without google drive please use alternative internet services such as OneDrive, Dropbox, email, FTP, etc.

Google		Gmail Obrázky 🏭 Př
Sign in Continue to Google Drive BUT login@vutbr.cz	Google	Učet doogle Vyhledáviní Map Vortube Play _ 2gele
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Mira TC Workspace





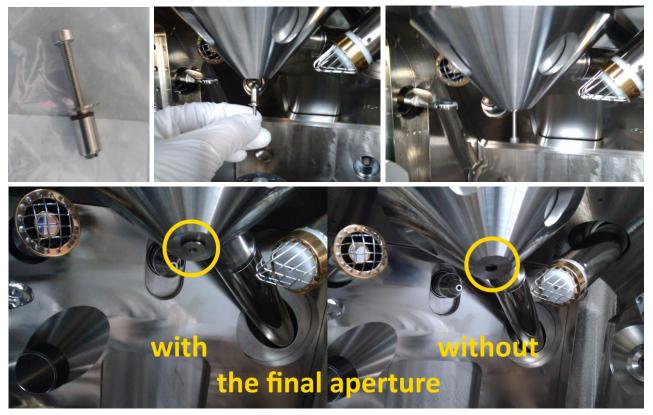


Guide for Low vacuum "UniVac" mode

Low vacuum compromises the imaging quality but helps carrying away charge from the sample and, thus, allows to measure non-conductive samples without a metal/carbon coating.

- 1. Vent the chamber.
- 2. Install the final aperture into the objective via following instructions:

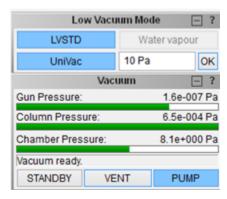
Find the final aperture in the accessory box. Wear gloves! Do not touch anything inside the chamber with bare hands! Avoid contamination! Carefully push the aperture inside the objective with the screw heading downwards, then remove the screw. You might need to bring the stage down to make enough manipulation space below the objective.



- 3. Place your samples on the stage and close the chamber.
- Activate the UniVac In the Low vacuum mode panel. Insert the final pressure (1-150 Pa) and confirm it by pressing OK button. Press Pump buton to evacuate the chamber and wait until the final pressure is reached. Then press BEAM ON button in the Electron Beam panel.



5. Activate the LVSTD detector in the Low vacuum mode panel and wait until it initializes.



- 6. Set minimum magnification and beam intensity (BI) 10 or higher.
- 7. Use the **Auto Signal** function to set suitable brightness and contrast.
- 8. Select the **OBJ Centering** function from the combo box on the **Pad** panel and turn the Trackball to set the brightest area into the centre of the **SEM Scanning window**.
- 9. Proceed as normally to collect images. Only the RESOLUTION and DEPTH modes can be used and LVSTD and BSE detectors are available.
- 10. To change the pressure, insert a new value and confirm it by pressing **OK** button. Wait until the new pressure is reached and stabilized.

When finished:

- 11. Deactivate the UniVac mode by pressing the UniVac button in the Low Vacuum mode panel.
- 12. Press **BEAM ON** button, then vent the chamber and remove the final aperture. **Do not continue directly to high** vacuum mode without removing the final aperture!

Place in the screw and gently pull the final aperture out of the objective. Wear gloves!

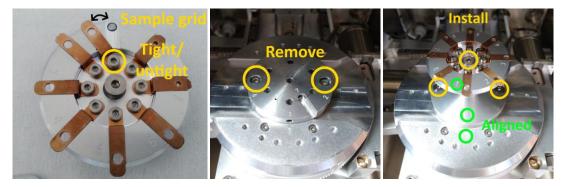
13. Remove your samples or proceed to high-vacuum mode. Then continue as usual.



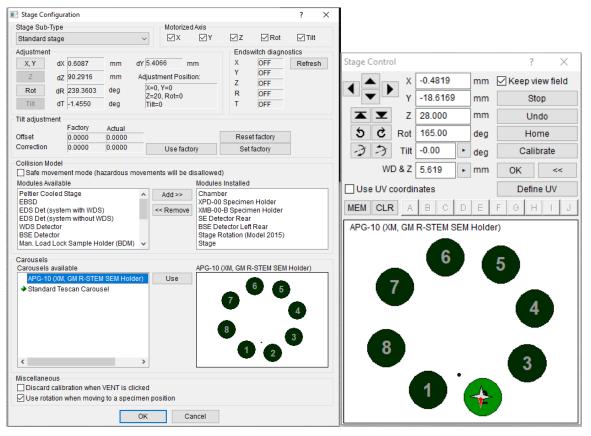
Scanning transmission electron microscopy (STEM)

- 1. Remove the standard stage carousel.
- 2. Load the grids into the APG 10 STEM carousel and install it on the SEM stage.

The carousel is stored in the large toolbox in the cupboard. All parts of the stage must be aligned with the number 1 mark pointing the same direction.



3. Activate the APG 10 STEM carousel in the *Stage Config* window located in the *SEM* menu. Select the new carousel and press *Use*. Then press *OK*. The carousel change is indicated visually in the *Stage control* window.



4. Pump the vacuum and turn on the electron beam as usual.





5. Select the sample position using the *Stage Control* window.

WARNING: The stage will move up/down. Be ready on the Stop button to avoid any crash inside the chamber.

The WD in the final position is about 5 mm. *Hazardous movement warning* may appear depending on the actual WD.

6. Check that the actual stage position is inside the stage opening for the TEM grids using the SE detector. If not, you may try to calibrate the stage (see below). Return to the point 5 after the calibration. If the calibration does not help, <u>DO NOT PROCEED</u> with inserting the STEM detector and contact the guarantor!

Calibrating the stage: Make sure that all the detectors are in their parking positions. Then press Calibrate button in the Stage Control window.

7. **ONLY PROCEED IF THE CHECK OF THE STAGE POSITION WAS SUCCESSFUL!** Push in the STEM detector into working position by clicking the appropriate button in the *Motorized RSTEM panel*.

WARNING: Watch the chamber view camera and <u>be ready to push the Stop button</u> to avoid any crash inside the chamber.

- 8. Switch of the chamber IR LED illumination and select the TE Bright or TE Dark detector.
- 9. Proceed with the beam alignment and observation as usual.
- 10. When changing the sample position, **retract the STEM detector first**. Switch the sample position and repeat the steps 6–9.

When finished:

- 11. Pull of the STEM detector to the parking position, select SE detector and switch on the chamber IR LED illumination.
- 12. Switch off the beam, vent the chamber, remove the APG 10 STEM carousel and install back the standard stage feature.
- 13. Activate the *Standard Tescan Carousel* in the *Stage Config* window located in the *SEM* menu. Select the new carousel and press *Use*. Then press OK. The carousel change is indicated visually in the *Stage control* window.
- 14. Proceed as usual. Continue the work or pump the chamber and switch the microscope to the Standby mode.



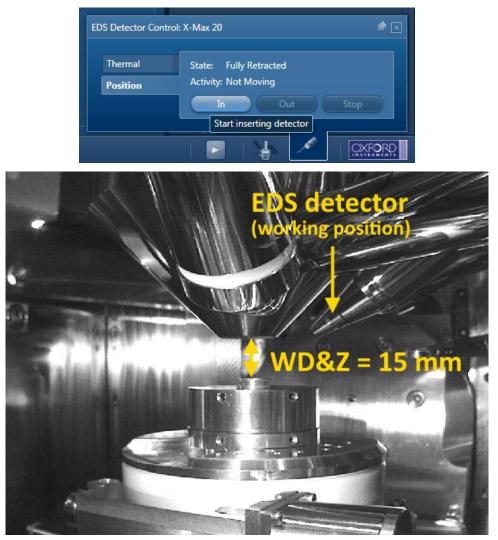


EDS (EDX) – Energy Dispersive X-Ray Spectroscopy

1. Prepare your sample as normally and focus. Recommended WD&Z is 15 mm (detector aligned with the stage).



- 2. Click on the icon Azec on the right screen to start the AZtec software.
- 3. Start a new project or continue with your previous work. Setup the project details.
- 4. Check that the detector is cooled. If not, cool it down.
- 5. Push the detector into working position. Use the icon in the bottom right corner of the AZtec software.



6. Turn off the chamber **IR camera**. Right click on the camera window -> IR camera.

EDS detector is not compatible with the IR camera due to oversaturation. The camera must be turned off all the time you are collecting EDS data. For safety reasons, avoid the stage movement in Z-direction when the camera is off.



7. Select suitable accelerating voltage (HV) and electron beam current via beam intensity (BI).

Higher voltage (max. 30 kV) decreases spatial resolution and surface sensitivity. Higher beam intensity (max. 20.99) increases the intensity of the EDS signal but may decrease the quality of the SEM image. Consider taking SEM image at lower HV/BI than will be used for the EDS analysis. Use ratemeter in the AZtec software (in the mid-right panel by default) to help you select suitable BI (keep in the green part of the Dead Time bar). The recommended HV is at least double the energy (in keV) of the electron transition you want to observe. See the periodic table on the mousepad for transition energies of individual elements (focus mainly on K α lines).

WARNING: High HV/BI may damage your sample.

NOTE: The following figure is illustrative, the optimal HV/BI depends on the sample and the detector's setting.



HV = 5 kV, BI = 8 (low signal); HV = 25 kV, BI = 15 (optimal); HV = 30 kV, BI = 20 (high)

8. Use the tabs in the top to navigate you through the settings and the measurement.



9. **OPTIONAL:** Add a new site/specimen to your project. Provide details of your specimen to be stored with your data. Specimens, sites and results are listed in the **Data View** panel in the top right corner.



10. Select the type of spectrum you want to acquire.



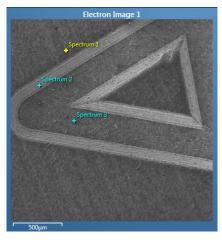
Data View						
Current Site	Data Tree					
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- (Site 1					
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	Electron Image 1	â				
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11. Collect the SEM image in the Scan image tab.

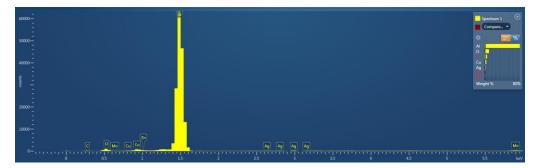
Use **Settings** in AZtec software to select SE/BSE detector, your current choice in MiraTC software will not be reflected. Use the MiraTC software to control other parameters (position, HV, BI, view field, focus, manual centering, etc.)

12. Define the position(s) and acquisition parameters in the Acquire Spectra tab.



13. Press **Start** button to collect the EDS spectra. The example of collected spectrum is displayed below.

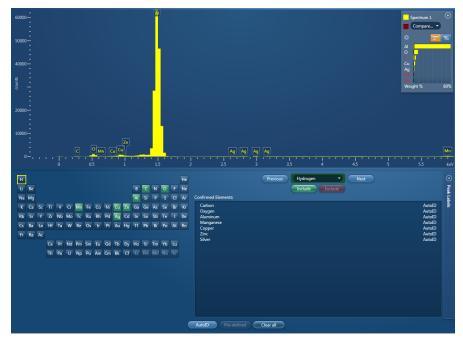
NOTE: The peak at 0 position represents noise. Adjust HV and/or BI to improve signal-to-noise ratio.



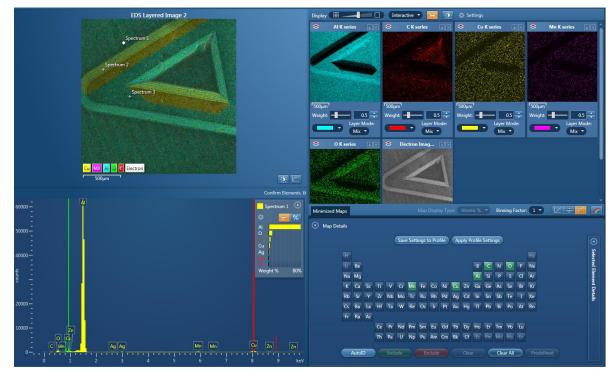




14. Via periodic table in the **Confirm Elements** tab, you can manually add (**Include**) or remove (**Exclude**) elements to be analysed from the collected spectrum.



15. If you are measuring EDS maps, the **Confirm Element** tab is replaced by the **Construct Maps** tab. You can overlay maps for individual elements into a composite picture, include/exclude elements, etc. By default, maps are acquired perpetually to improve the results until stopped. manually



16. Use the Calculate Composition (Analyze Phases for EDS maps) tab to evaluate your results.





17. Export the results.

Most of the content (i.e., SEM images, spectra, overview tables) could be exported from the local menu (right click on the object that should be exported).

You may also use the "Report results" feature: Select the template in the **Report Results** tab submenu -> **Report Template** to export your results. Use an existing one or use **Template Generator** to make your own. Press "**Report results**".



18. Continue with new site/specimen.

When finished

- 19. Pull out the EDS detector to parking position (icon in bottom right corner).
- 20. Close the AZtec software.
- 21. Turn on the IR chamber camera.
- 22. If you will not continue with SEM imaging or other work, decrease accelerating voltage (HV to 5 kV or less) and Beam Intensity (BI to approx. 10) and put the microscope into standby mode (follow the usual procedure).