(S)TEM Techniques

Inserting the holder

1. Align the small pin extending from the metal part of the holder with the angled line next to closed on the faceplate.
2. Insert the holder such that the O-ring seals the airlock.
3. Connect the cable and select the toolholder in the interface. Wait for the timer to run out such that the vacuum is high enough.
4. Hold the holder in such a way that you can resist the pull of the vacuum and rotate the holder counter-clockwise until the pin extending from the holder is aligned with the hole in the faceplate.

Acquiring an image using the phosphorous screen

1. Check the vacuum in the octagon and make sure the vacuum is lower than 20 Log, if so you can open the column valves.
2. Make sure the phosphorous screen is inserted into the optical column.
3. Load the alignment files for the FEG and the operating mode of the microscope.
4. Open the column valves and check for a bundle.
5. Press the eucentric focus button to reset all displacements.
6. Using the sample z-height buttons try to minimise the contrast of the picture.
7. Further minimise the contrast of the picture by magnifying and then using the focus knob.

High Resolution TEM image using digital Camera

1. Make sure the screen current is roughly less than 10 nA.
2. Retract the phosphorous screen using the R1 button.
3. In the Velox camera software press the play button to start acquiring.
4. Before changing the beam intensity or magnification always reinsert the phosphorous screen
5. Open the fast Fourier transform window on the top right.
6. By pressing the stigmator button and using the multifunction knobs correct the stigmator such that there are concentric circles visible in the FFT. Using the focus buttons make the circles larger. The flat inner section of the innermost circle should fill most of the FFT image.
7. The most detail can be achieved if there are bright spots or rings on the edges of the FFT.
8. Acquire a HRTEM picture using the camera button in the top toolbar.

Diffraction Image

1. Spread the bundle as to illuminate the whole sample on a small magnification
2. Insert the largest aperture and centre.
3. Insert smaller apertures until only the selected area is illuminated.
4. Insert the beam stop through the button at the top of the screen.
5. Position the centre beam behind the beam stop such that the rings show on the phosphorous screen.
6. Use the focus knob to tune the focus such that the outermost rings are sharp.
7. Adjust the Velox camera settings to minimal exposure time and take a short live image.
8. Make sure that the individual bright counts are not too high.
9. Adjust exposure accordingly.

STEM imaging

1. Load alignment file for high tension STEM. (STEM 300 kV)
2. In the Velox software activate the HAADF detector by clicking it in the column overview.
3. Make sure the Titan PC is in control of the electron beam deflection by checking that the box under the monitor is set to ”INT SCAN”.
4. Refocus on the sample using the same method as in the HRTEM section.
5. Pause the beam and set it to illuminate amorphous material.
6. Activate the condenser stigmator and correct the Ronchigram to show a flat circle in the beam.

EDX

1. Re-check if the focus of is correct.
2. Select a region for the EDX inspection.
3. Select a region for the drift correction, make sure it has well-defined horizontal and vertical features.
4. Use the analysis toolset to analyse a region of interest.
5. Using the periodic table on the right of the screen you can select which elements you want to show.

EMPAD

1. Enter STEM mode by loading a register or creating your own (following alignment steps).
2. Create a reference circle by using the HAADF detector as a guide and note its centres
3. Create two lines a vertical and a horizontal one and create a crosshair through the centre of the circle.
4. Adjust the Ronchigram to lie in the centre of the crosshair-like shape.
5. Connect to the EMPAD PC by opening ”nomachine” and selecting ”EMAPD-PC (New), yes it is misspelled”. EMPAD PC is the second PC from the top in the rack.
6. Open the EMPAD GUI and open or create a project folder.
7. Make sure that the HAADF is retracted before inserting the EMPAD since the EMPAD doesn't know about the HAADF sensor.
8. Switch beam control to ”EDX SCAN”
9. Align the Ronchigram to the centre of the EMPAD detector by using the ROI and Cross options in the video screen. You can mark this location for convinience in the TEM user interface.
10. While ”Acquire” is selected in the Scan Settings window you can take a image/dataset by pressing ”Take”.
11. Shutdown the EMPAD and retract the sensor from the column.
12. **Optimising the conditions of the electron beam**
    a) Will follow later

Shutting down

1. Close the column valves.
2. Center the stage using (Search) $\rightarrow$ (Stage) $\xrightarrow{\text{fly-out}}$ Reset Holder.
3. Take out the holder, remove the sample and reinsert it.
4. After octagon vacuum is sufficient, and the holder is fully inserted, turn of the turbo pump.

Diagnostics

Correcting dark and gain (Removing horizontal bars)

1. On the top left of the screen open the camera settings page.
2. Then select the CETA camera and open the fly-out.

3. In the fly-out menu open the ”dark and gain” settings tab.
4. Press the singular button and load a correction file or the most recent one.

No beam control in STEM mode

No image using the HAADF or othe dark field detectors $\rightarrow$ check that beam control is on the Titan PC by having ”INT SCAN” pressed.
No image using the EMPAD detector $\rightarrow$ check that the beam control is given to the EMPAD PC by having the ”EDX SCAN” button pressed.