OPERATION MANUAL FOR JEOL JEM2800

Liquid Nitrogen filling

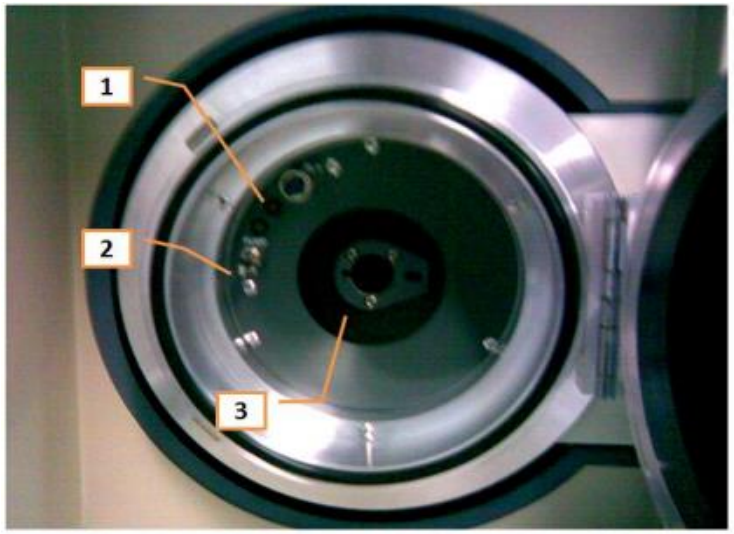
- The liquid nitrogen run out from the dewar in about 10-12 hours, so if there was a user before the filling is not necessary.
- Make sure that ACD Bake is off. Remove (if it still in the ACD) the ACD heater and secure it in the cabinet behind you.
- Fill microscope dewar with 1L of liquid nitrogen (LN2) using the Jeol funnel. When there is no more LN2 vapor coming out from the dewar top it up with approximately 300-500 mL of LN2

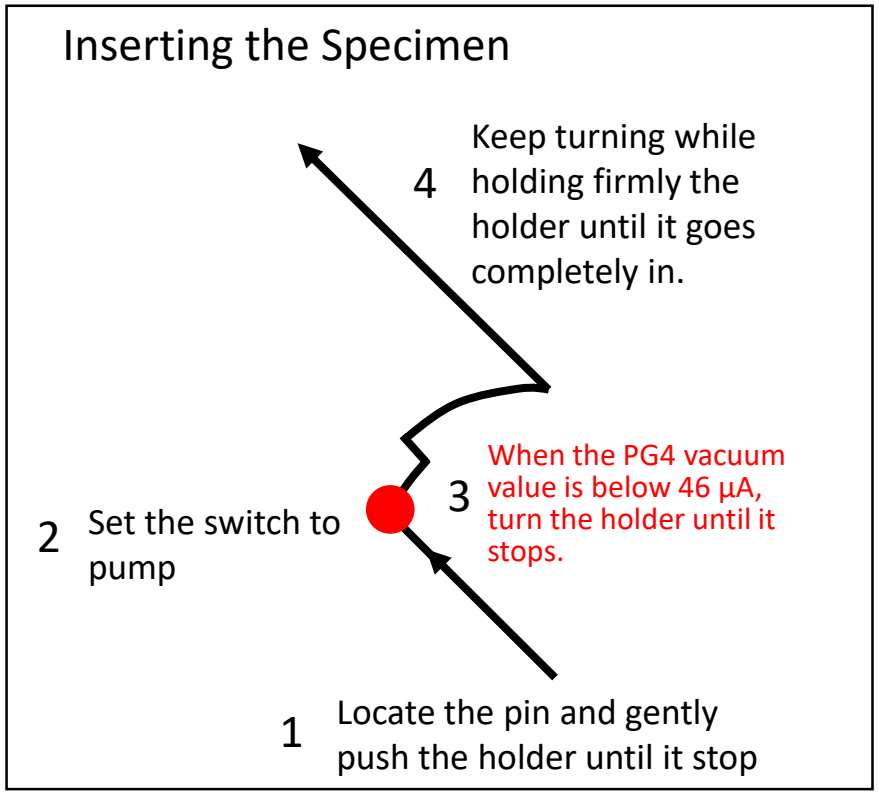
Mounting of the sample on the holder.

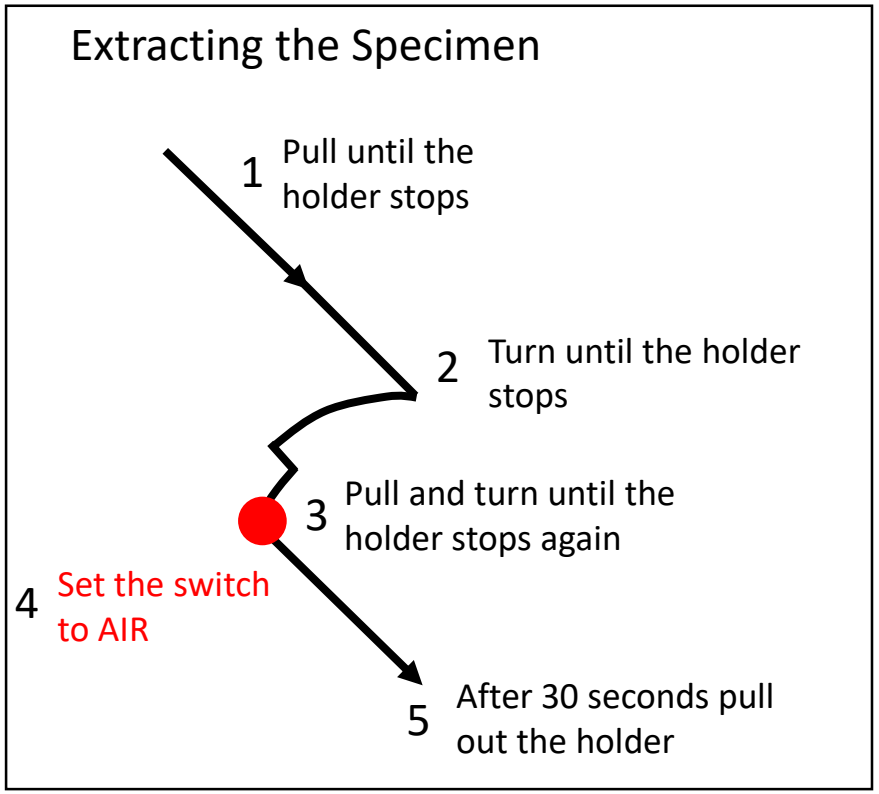
- Mounting of the TEM grid depends on how you prepare the sample.
- There are three sample holders available:
  - ✓Single tilt and single grid holder
  - ✓Single tilt and three grids
  - ✓Double tilt
- Ask EMF staff for a demonstration on how to mount your sample on the specific holder required for your project.
- If you have organic materials in your sample is good to consider plasma clean the sample in the plasma cleaner to avoid contamination while imaging. Ask EMF staff for a short demonstration.

Insertion of the specimen holder into the microscope.

- Cleaning of the samples using the plasma cleaner is always advised.
- Before inserting the specimen holder make sure the stage is neutralized.
- Follow the instructions on the goniometer stage (or the picture below here) to insert the specimen holder.

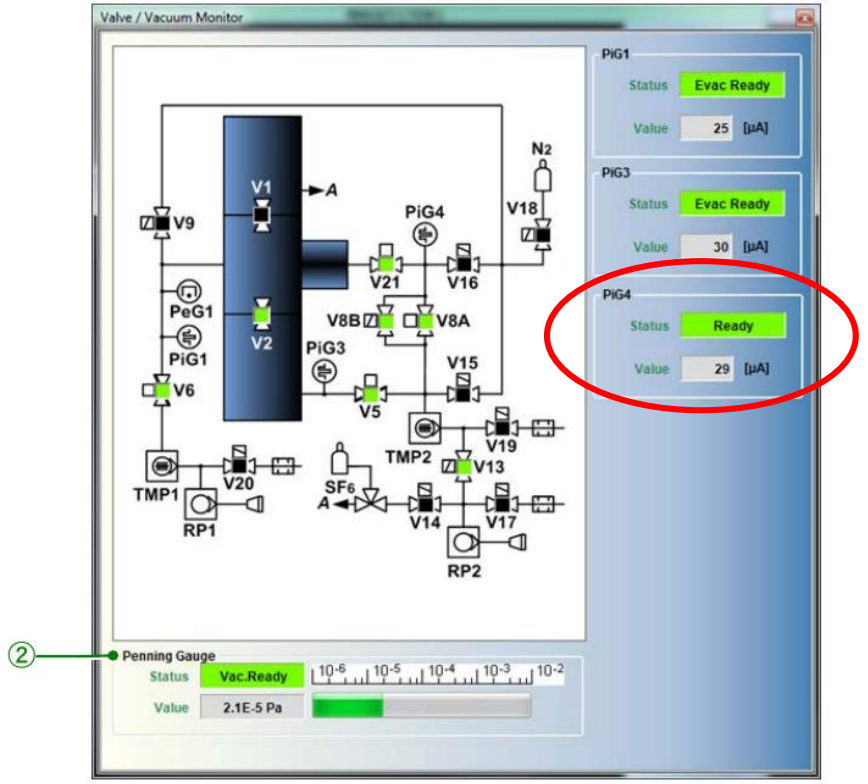






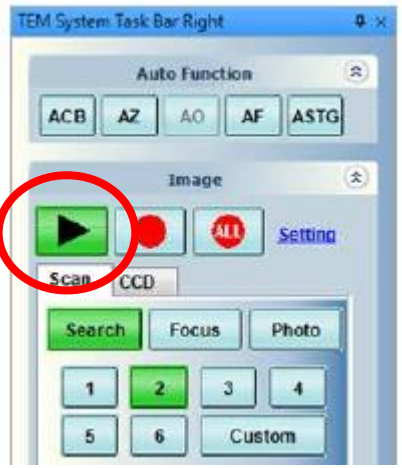
Insertion of the specimen holder into the microscope.

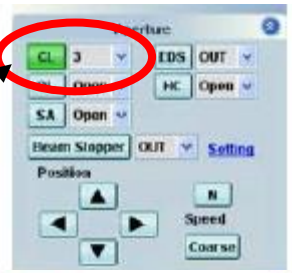
- Locate the pin on the holder. This pin goes into a notch on the goniometer opening (3). Gently push the holder in till you hear a click. Then start the pump by turning the switch on the left side.
- Wait until the PG4 in the vacuum window is below 46 uA; turn the holder clockwise slightly and it will go in a little bit. Then continue with turning the holder further till it goes completely in. **DO NOT** let the holder go in alone otherwise it may cause damage to goniometer.
- Afterwards go to „TEM Center-Stage“ and choose the correct sample holder.

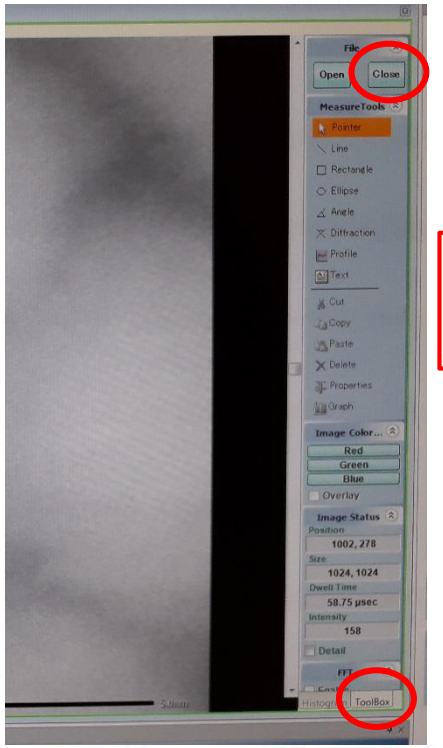


Session start

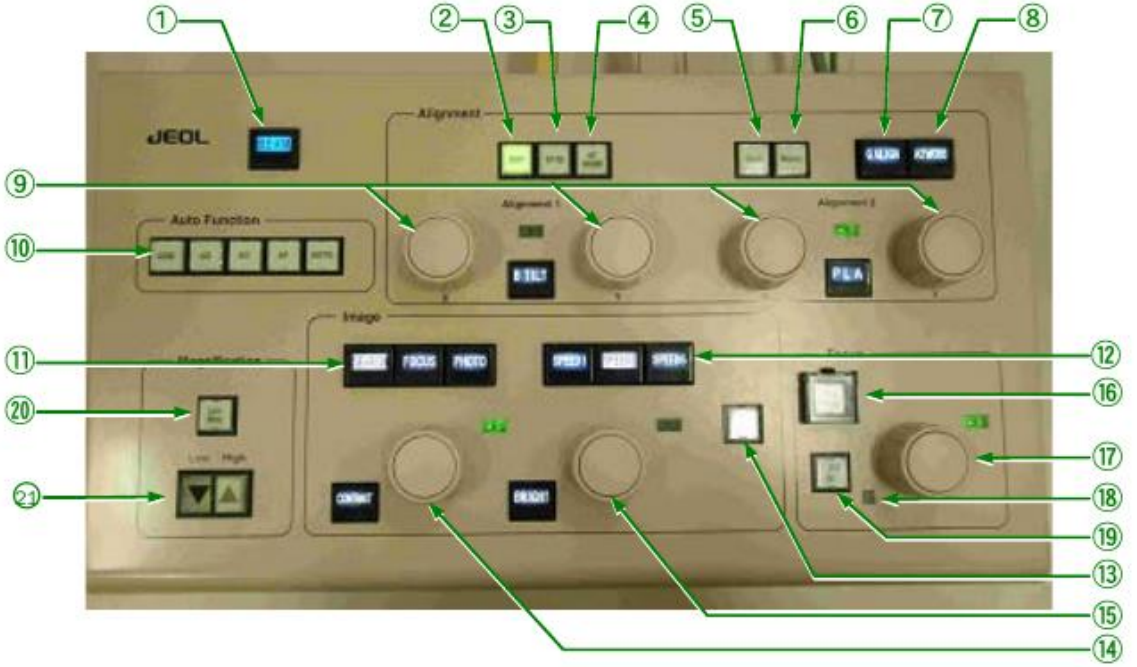
- Start the live cam (play arrow turn green when live)
- If there is an image on the screen the live will not start. Close the image to see the live 1
- Insert the largest CL aperture
- Press STD Focus to reset any defocus (16)
- Press the BEAM (1)







1

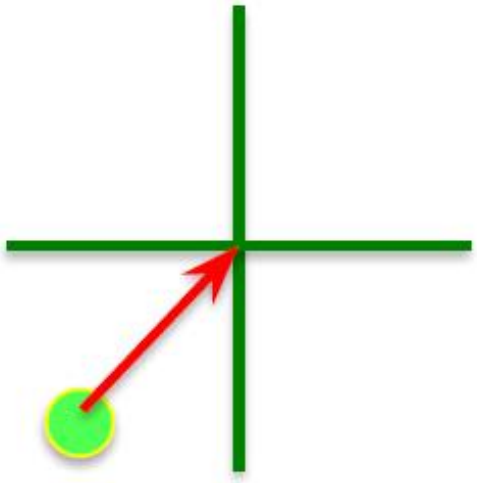


Setting up the electron beam

- Find the beam. Remember that you might be on the grid bar. To check it, reduce the magnification and move the stage.
- ***Condensed beam might cause a damage to the scintilator or a camera – please minimize the time you work with the condensed beam.*** *Please read through the next 3 slides in advance before you proceed with the following actions*:
  - ✓Center the beam
  - ✓Center the CL aperture
  - ✓Correct for CL astigmatism

Condenser system alignment

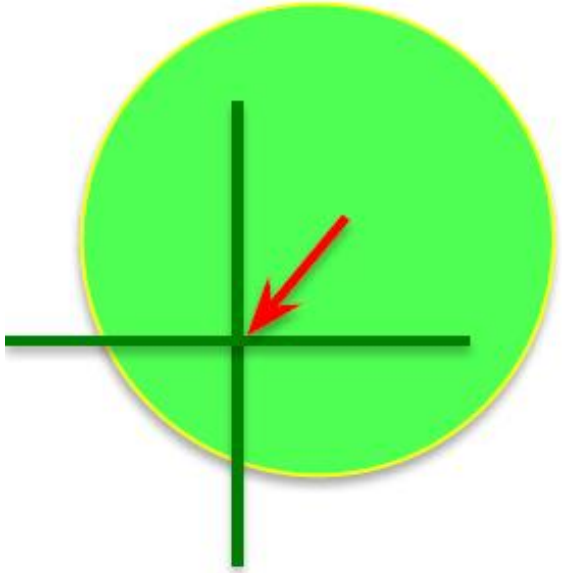
- Set a magnification around 200kx and set short exposure time for the camera (Search 1)
- Focus the beam by turning the Brighthness knob and center the beam using the B SHIFT X, Y (Alignment 1).
- Keep the beam condensed for **short time only** to avoid burning marks.

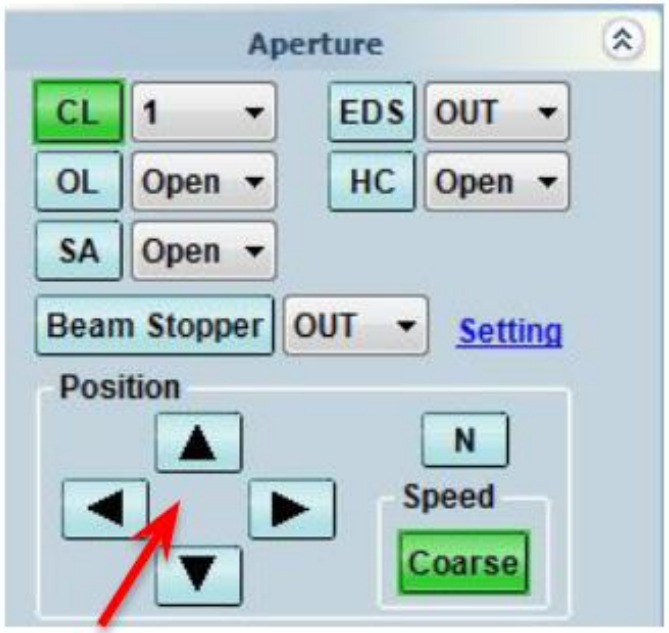


Bring the beam to the center with Bshift x and y

Condenser system alignment

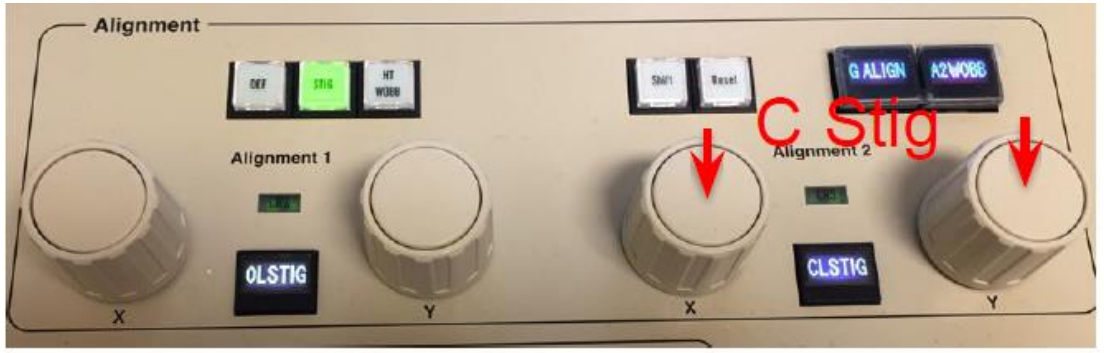
- Turn the brightness knob clockwise to spread the beam and center the aperture using the „Aperture-position adjustment“.
- Concentrate the beam again by turning the brightness knob counter clockwise and center it using B SHIFT X, Y (Alignment 1).
- Repeat these steps till the beam is centered.

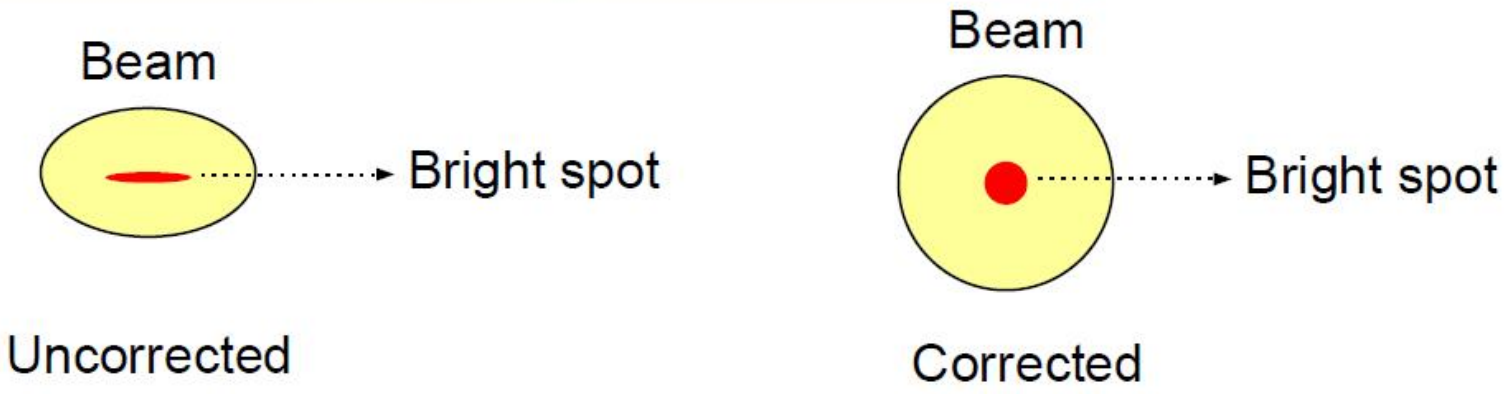




Condenser Astigmatism correction

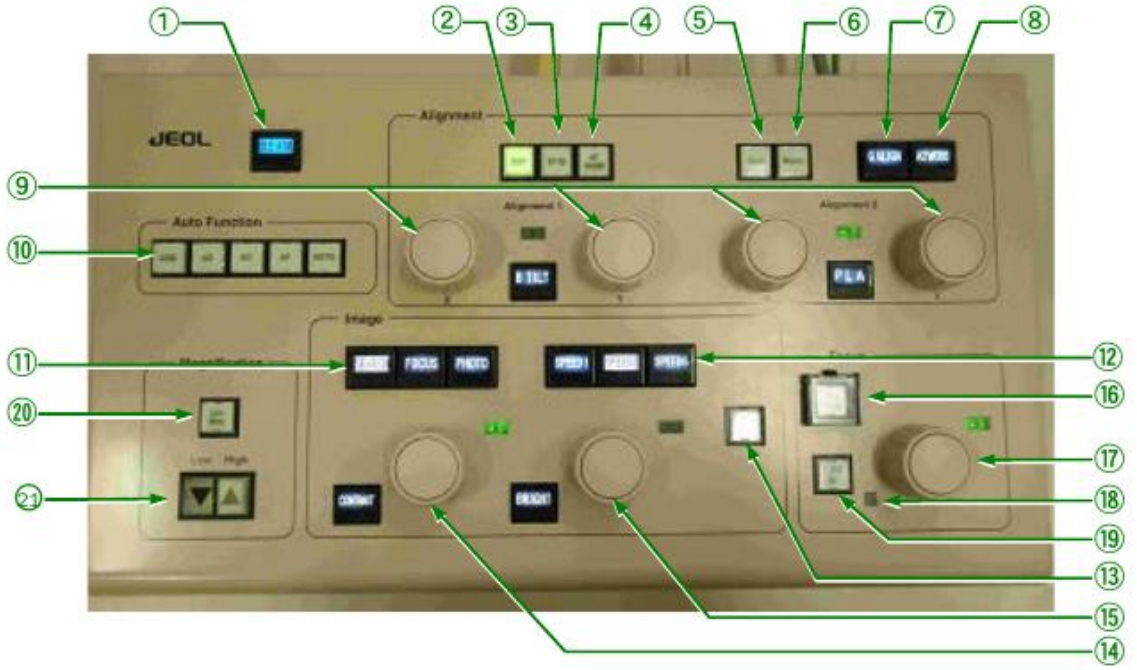
1. Focus the beam using brightness knob. In case of astigmatism the beam would look eliptical.
2. Press Stig button and use CLSTIG X, Y (Alignment 2) to make the beam as round as possible.





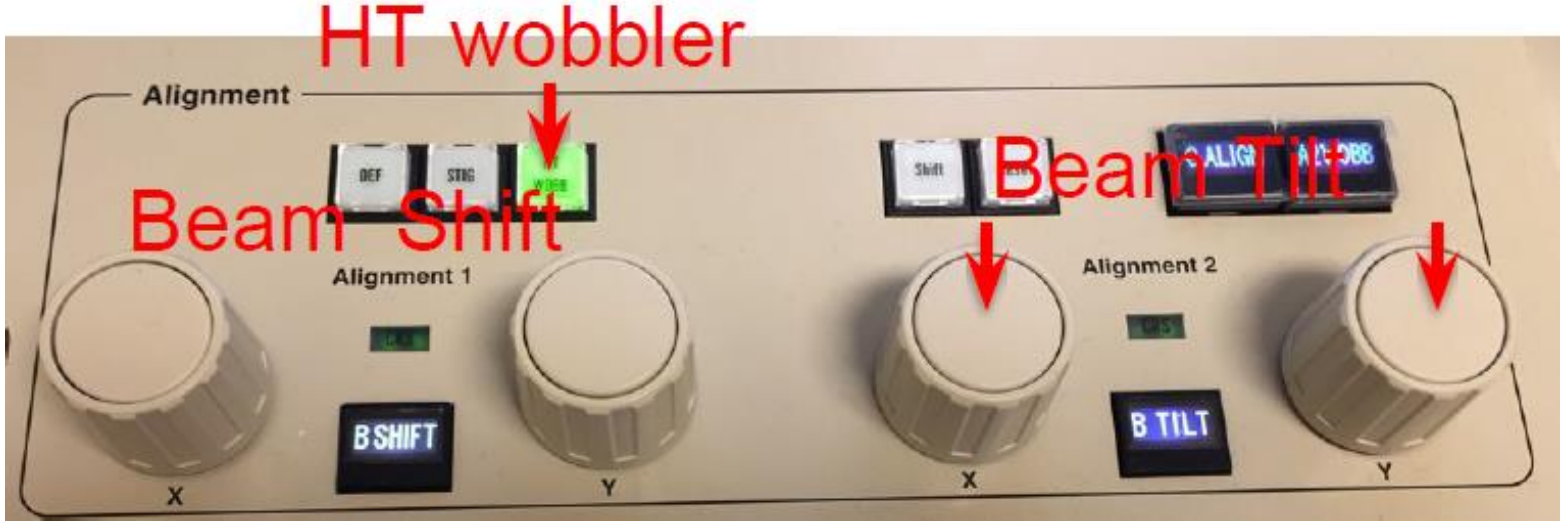
Eucentric Height

- Before starting the alignments. Find your sample and locate the area of interest. Press STD Focus button (16).
- Activate the Image Wobbler (19)
- Press and hold -z or +z on the Stage Control panel to minimize the image shift.
- Start with coarse and when you are close to eucentric height change to medium or Fine.



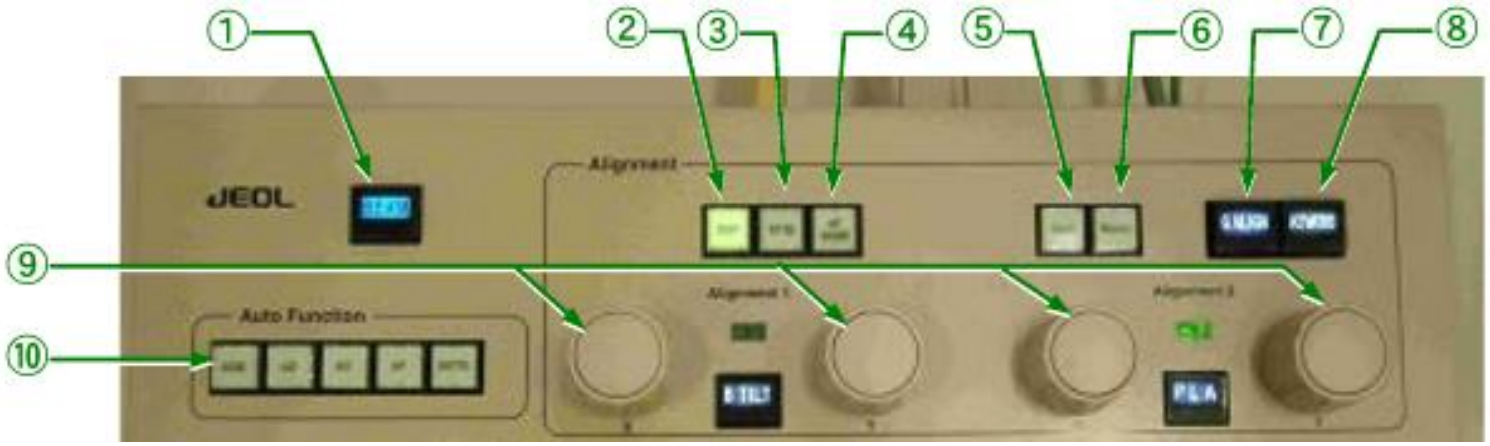
HT centering

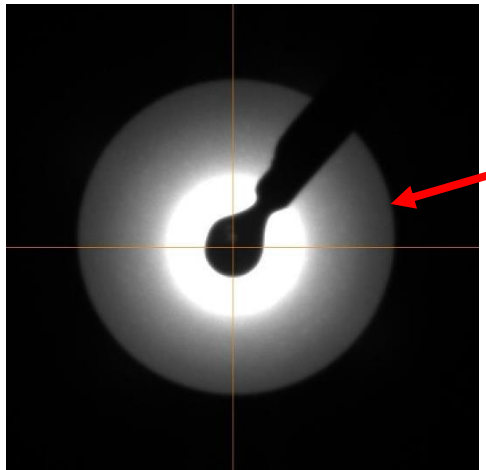
- Go to magnification 1Mx and find a feature.
- Make sure the beam is centered.
- Spread the beam and turn on the HT wobbler.
- Make the wobbling as concentric as possible using B TILT X, Y (Alignment 2).



Objective aperture

- Go to diffraction mode by clicking DIFF in the Image Function Selector (SW).
- Press the SHIFT button (5) on the Control panel.
- Center the direct beam on the beam stopper with PLA X, Y knobs (Alignment 2).
- Insert the OL or HC aperture.
- Adjust DIFFFCS to focus the OL or the HC aperture.
- Center the apeture with the position arrows in the Apertures panel (SW).

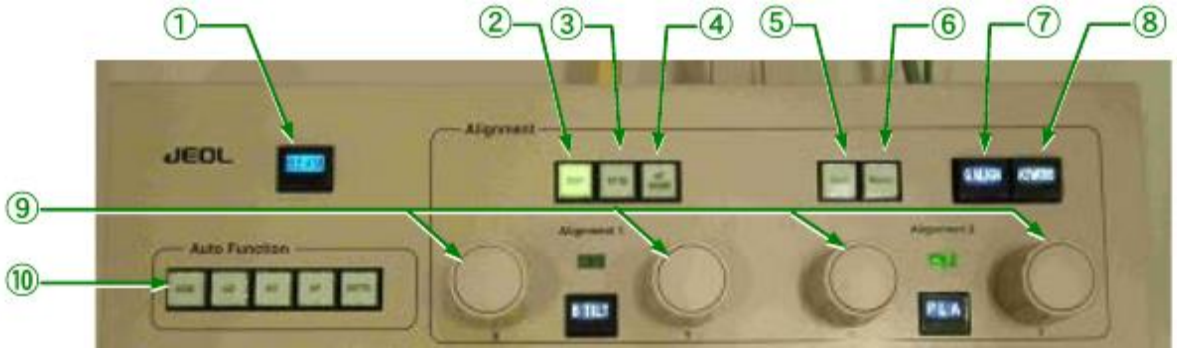


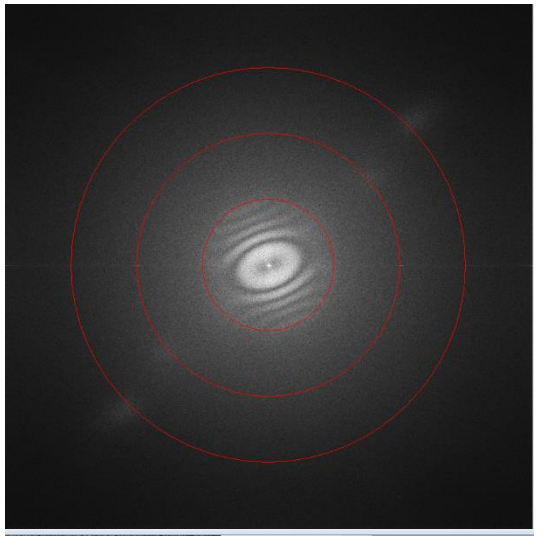


OL aperture centered

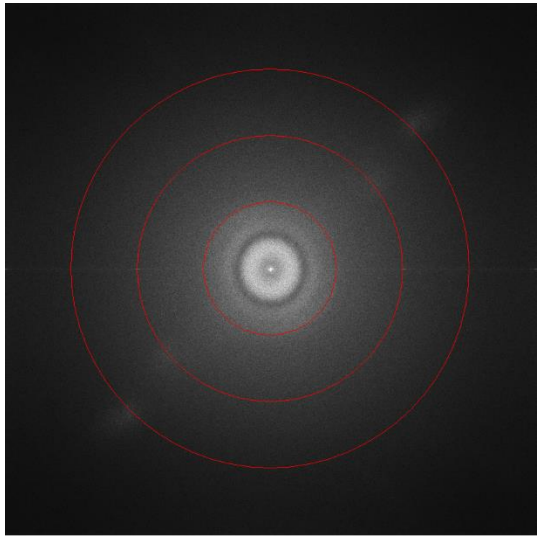
Objective Astigmatism

- Find anything amorphous on your sample.
- Go to a magnification higher than 3Mx.
- Spread the beam and switch to either the bottom mounted (see the next slide) or side mounted camera to correct for the astigmatism.
- The power spectrum (FFT) must be round and change concetrically by going through the focus.
- Press STIG (3) on the Control panel and use OL STIG knobs (Alignment 1) to make the power spectrum round





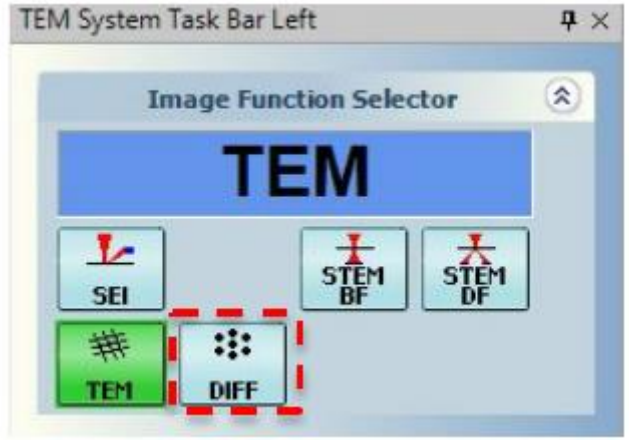
Astigmatic power spectrum of amorphous region



Astigmatism corrected power spectrum of amorphous region

Selected Area Diffraction

- For diffraction experiments a parallel beam condition is required (refer to the table on next page).
- Please make sure that you have custom exposure with short exposure time and then click DIFF.
- When the microscope is in DIFF mode a beam stopper is automatically inserted.
- Shift direct beam to the beam stopper by pressing SHIFT on the control panel and use PLA X, Y knobs (Alignment 1).
- Set the camera lenght (Magnification buttons) desired – depends on the sample.
- Use DIFFFCS to make the diffraction spots as sharp as possible.
- Record the image with the red record round button on the camera section (SW).
- **Limit as much as possible the time in diffraction to avoid burning marks on the diffraction spots.**



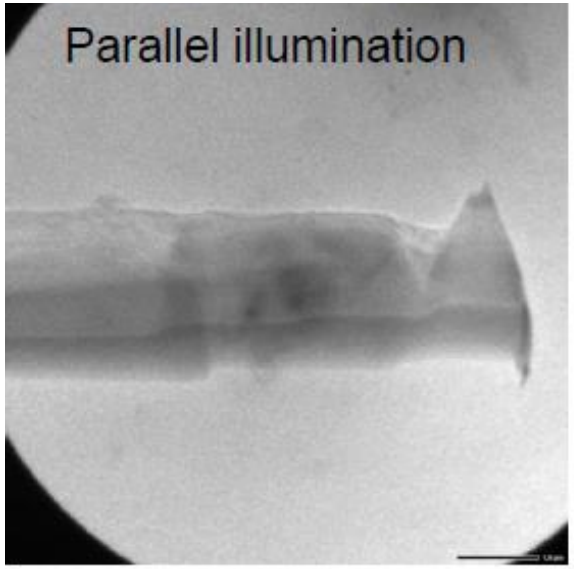
Parallel illumination condition for Jeol JEM2800

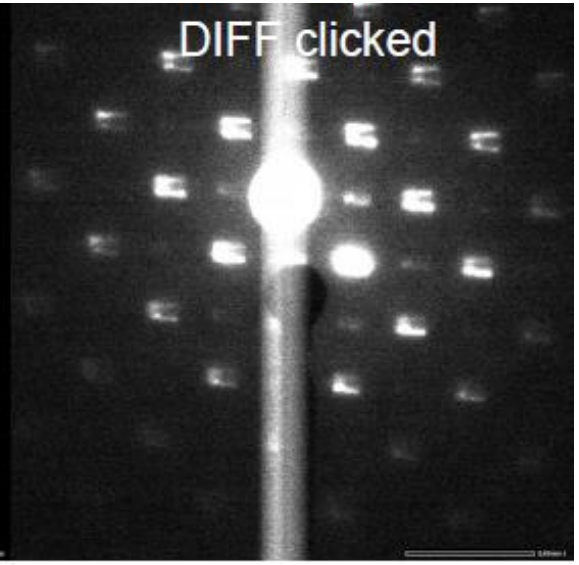
To achieve parallel illumination it is possible to set the CL3 (Brightness) with the value in the table below. To see the current value of CL3:

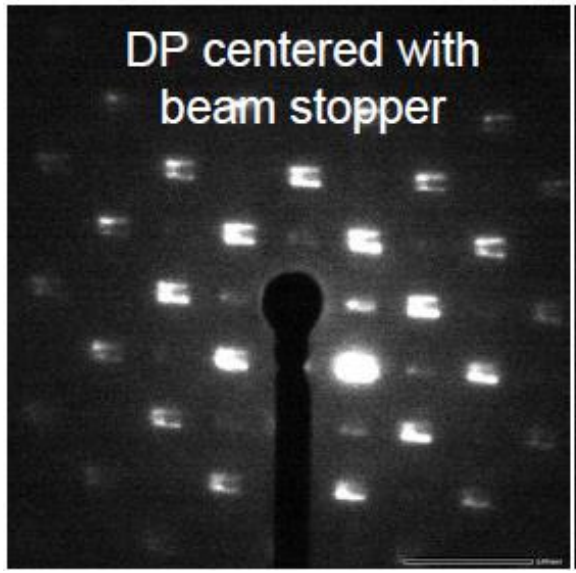
- open the deflector monitor from View Lens -> Deflector Monitor.
- Select A/D [V]
- Select the Lens tab (2)
- Adjust the value of CL3 with brightness know according to table

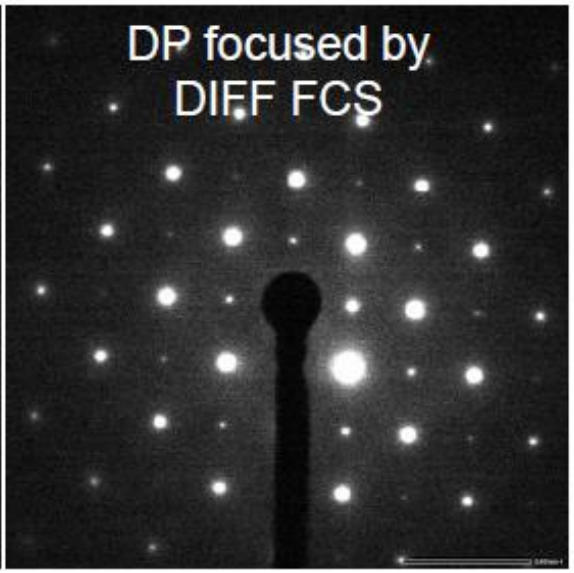
Spot Size	CL3 value in View Lens/Deflector Monitor
1	3.81
2	3.52
3	3.34
4	3.20
5	3.13

Graphical step by step SAED







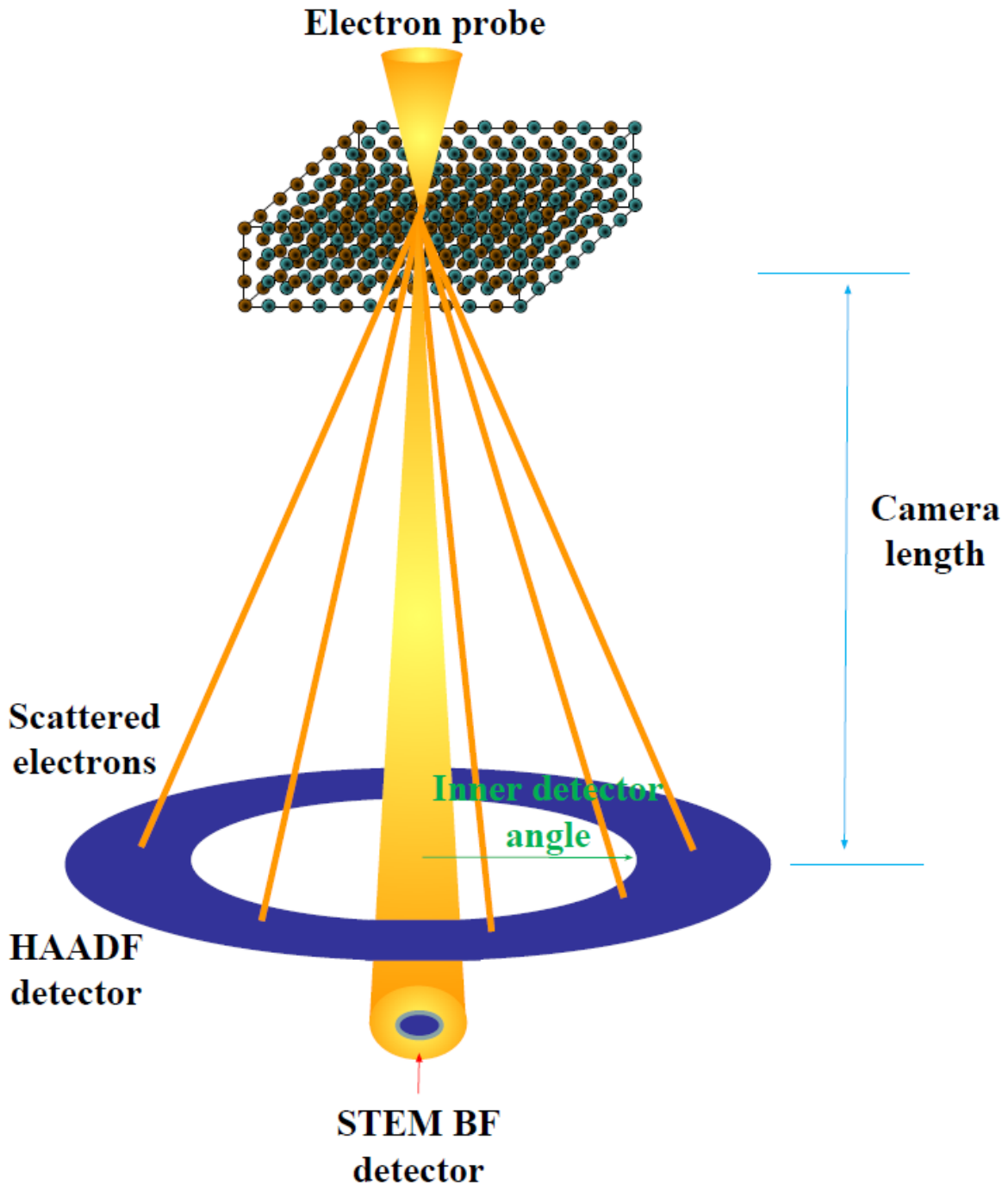


STEM Operation

Scan coils are balanced to scan the focused electron probe over sample without introducing beam tilt.

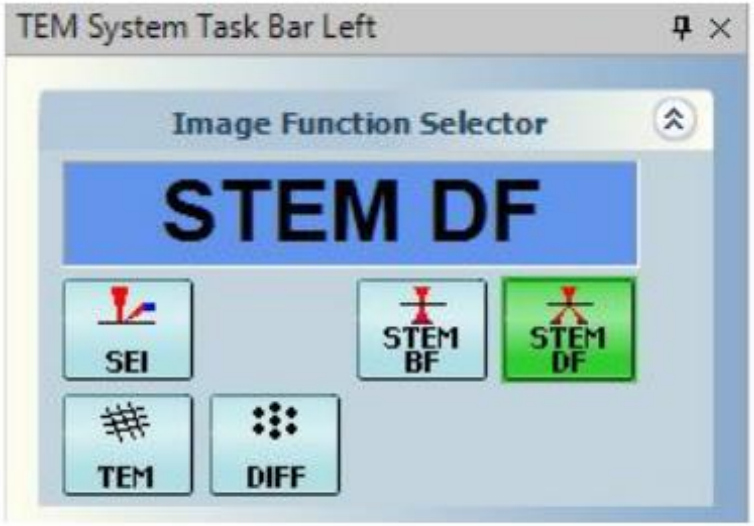
While you are acquiring a STEM image, you are not looking at the probe itself. What you see on the screen is the diffraction pattern (convergent electron diffraction pattern) acquired from each pixel in STEM image.

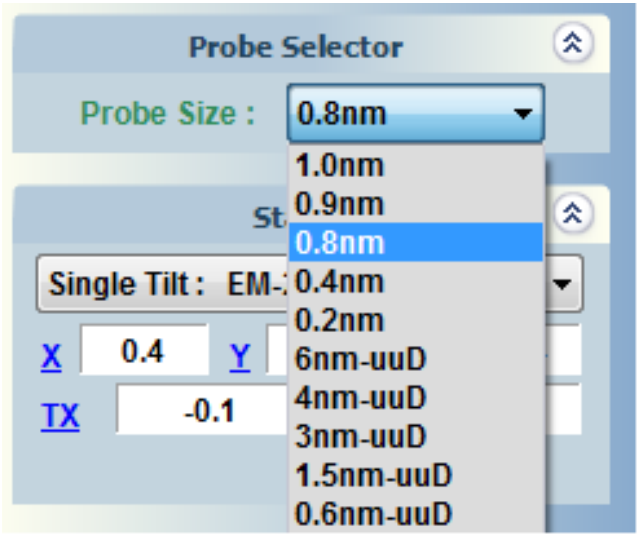
HAADF (High Angle Annular Dark Field) detector collects the electrons scattered to high angle and STEM BF detector collects the direct beam.



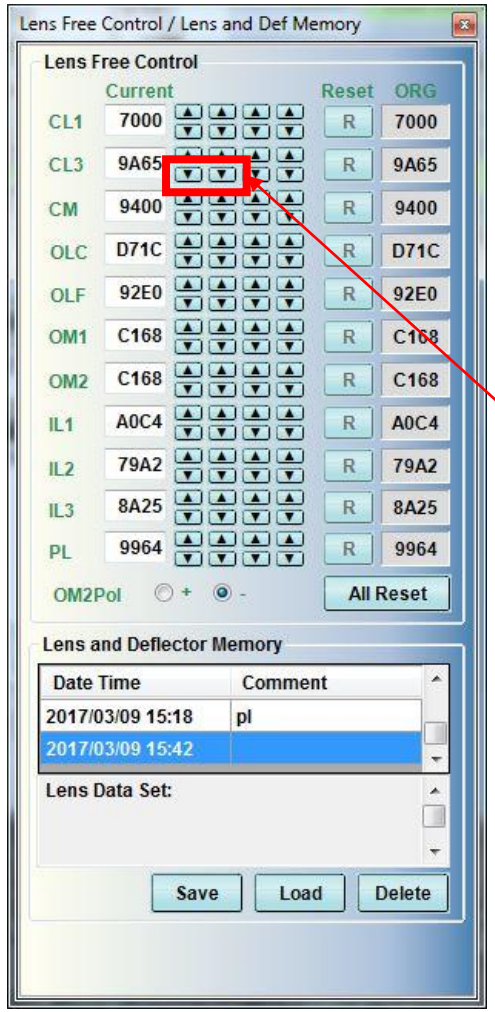
STEM Operation-STEM alignment

- Click on STEM BF or STEM DF button.
- Select probe size (smaller probe gives higher resolution).
- Roughly adjust brigthness and contrast to see something on the STEM detectors.
- Move stage to an amourphous region.



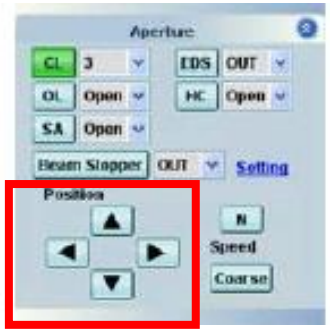


STEM condenser aperture alignment

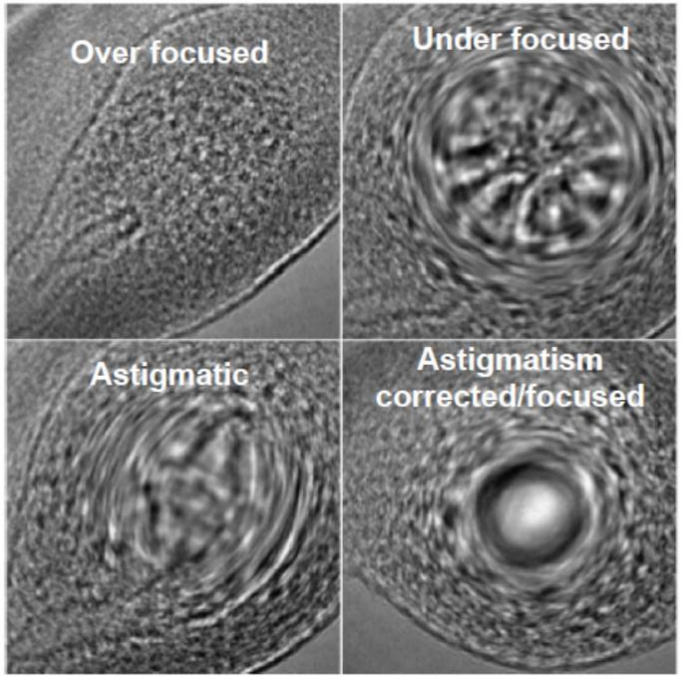


- Open the Lens Free Control shown on the left (Control > Lens / Deflectors > Lens Free Control)
- Click on the Ronchigram button

- Use the down arrow buttons to condense the ronchigram to a spot and center it in the screen with the PLA knobs
- Press the „R“ button on the Lens Free Control panel to reset the the lens value
- Center the aperture in the center of the screen with the aperture buttons.



- Adjust tilt, focus, and astigmatism, referring to the Ronchigram.
  - ✓ Set the camera length to about 4m to 6m. If the Ronchigram pattern is too bright or too dark, change the exposure time of the CCD
  - ✓ Approximately focus (or slightly underfocus) the Ronchigram with the focus knob
  - ✓ Press the DEF button on the operation panel and align the center of the Ronchigram pattern with the center of the aperture using the B TILT X, Y knobs (Alignment 1). If the pattern center deviates from the center of the window, center the pattern using the PLA X, Y knobs (Alignment 2).
  - ✓ Press the STIG button and turn the STIG X, Y knobs (Alignment 1) on the control panel until the Ronchigram pattern becomes round.
  - ✓ Click the Ronchigram button to exit from the Ronchigram mode.



Reference images of the Ronchigram acquired on an amorphous substrate with 150 um condenser aperture.

STEM Image aquisition

- Go to image selection on the right hand side of TEM center and select „Scan“. Make sure that play button is green.
- In order to record images, select on the image you want (e.g. BF or DF) and then click on record button (2). This will save only the selected image.
- In order to record all images (SE, BF, DF) click on (3).
- The images will be recorded in the thumbnails panel.
- Show the thumbnail 1 panel select the image you want to save on the disk and click on save 2 .