

OSIRIS TEM CHEAT SHEET

V.5

FIRST

Check that the high voltage is on: From the “go away” tab, look at the “High Tension” menu. The HIGH TENSION button should be yellow, and the OPERATE button should be yellow.

If the voltage is not on, check the vacuum level and then turn it on. It will take just a few minutes for the voltage to go up to 200 kV.

SECOND

Check the liquid nitrogen level from the SETUP tab. If you are going to use the xray detector, then you will need liquid nitrogen. Otherwise, you do not. If you put nitrogen in the dewar, please fill it all the way up.

LOADING

Line up the pin (the one near the end of the rod) with the line labeled “close”, or even a bit further counter-clockwise, at ~3:30

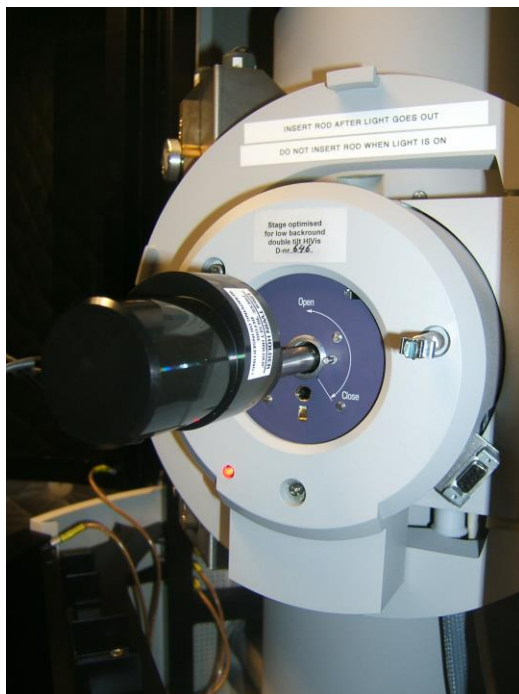
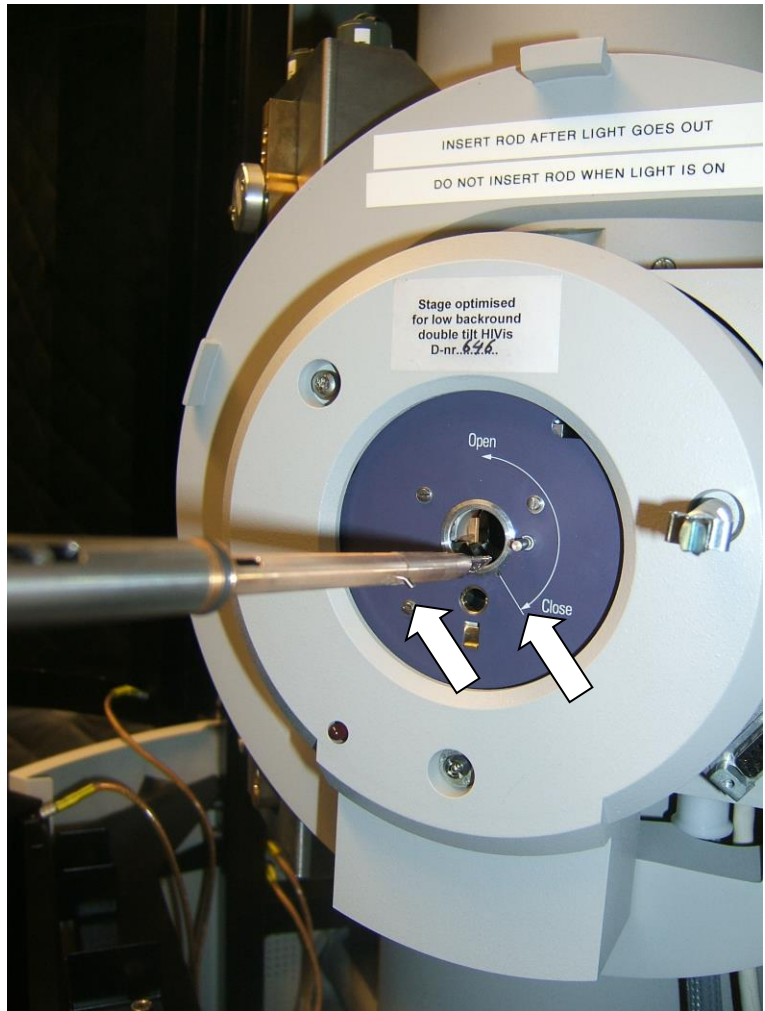
Insert the rod until it stops, turning it a bit **CLOCKWISE** as you go. Do not turn it the other way; that would be bad.

THE BLUE LINE ON THE ROD SHOULD BE AT THE END OF THE HOLE!


Now you can let go. The rod might turn slightly counter-clockwise, so remain calm.


Inserting the rod will start the pump.

IF you are using the double-tilt holder, then plug in the cable, placing the wire up and over the holder.




Go over to the computer console and look at the message box.



Press the  button to clear away any previous messages. **Ignore** messages about the liquid nitrogen level. Instead, check the liquid nitrogen level from the Vacuum Status box.

The message box should then ask you to choose a specimen holder. You are probably using the single-tilt holder, so choose that one, then click on 



If you are using the double-tilt holder then click on  again to confirm that you have plugged in the cable:



If you see the message “pumping sequence aborted” you can ignore it and click  again. That message appears if you insert the rod slowly enough to double-hit the sensor  that starts the pump.

Wait for the light (next to the sample rod) to go out. You will hear a click when the vacuum system switches over to the turbo pump. Soon after, the light will go out. You can think of the light as meaning “not ready”.

Seriously, do not rotate the rod until the light goes out. You could dump the vacuum, which would ruin your day. It is NOT interlocked, so watch out!

Turn the holder counter-clock-wise and let it slip in the rest of the way.

DO NOT PUSH THE ROD. You should be holding it back a little while the vacuum pulls it in. If you think you should push then you are doing it wrong. **GET HELP!**

Wiggle the rod a little to make sure it is seated well, then tap on the end twice.

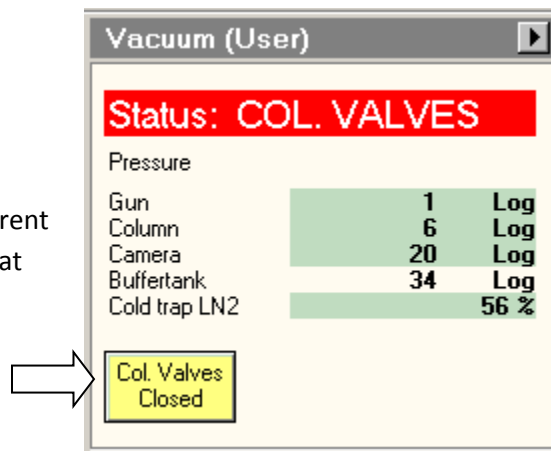


TURNING ON THE BEAM

Workset → Setup → Vacuum → Col. Valves

Click to open the valves.

On any button a yellow background means “this is the current state”. So the button shown here on the right tells you that the column valves are currently closed.

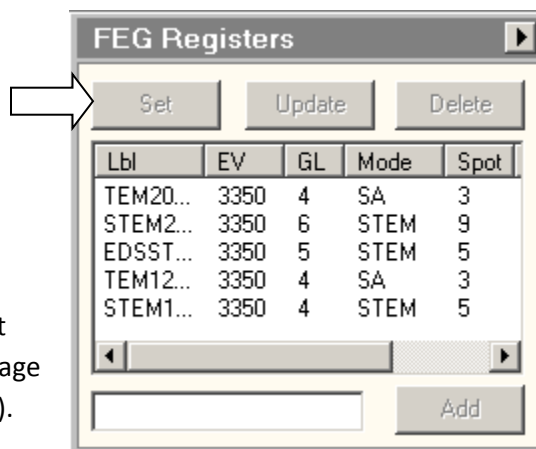


Load standard TEM conditions with

Workset → Setup → FEG Registers → TEM200Kv then click on “Set”.

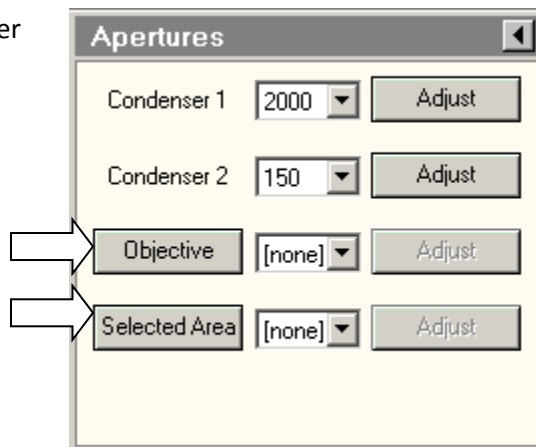
Do NOT click on “update”, “delete”, or “add”.

If you get an error message saying you can't do this without being in the “operate” mode, then this means the high voltage is off (which happens whenever the pressure goes too high). In that case, ask for help to get the high voltage back on.



Pull out the objective and “selected area” apertures, if either of them are currently inserted. You might need these apertures later, but for now, pull them out with


Workset → Tune → Apertures → Objective



Press the “Eucentric Focus”  button on the right knob panel.

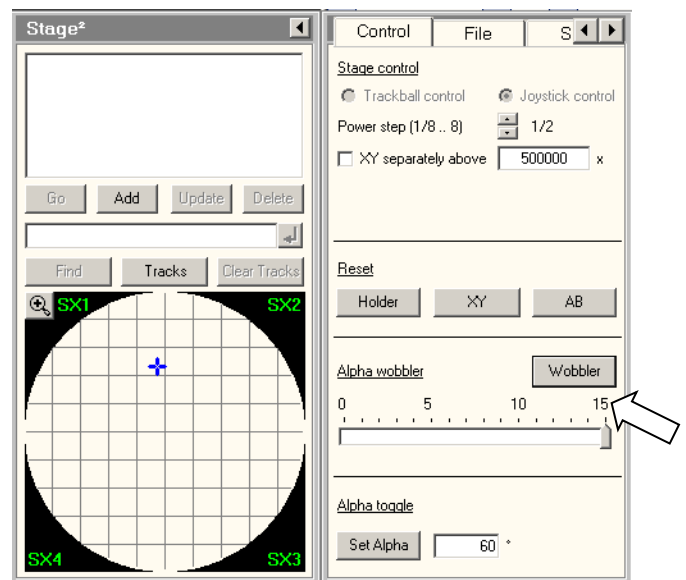
Find your sample! Lower the magnification to M-2300 (or lower, although the LM range can be annoying.) If the image is black, try using the joystick to move away from copper grid lines.

Set eucentric height:

Workset → Search → Stage → Flapout 

Set a wobble amplitude of 5° then click on “wobbler”.

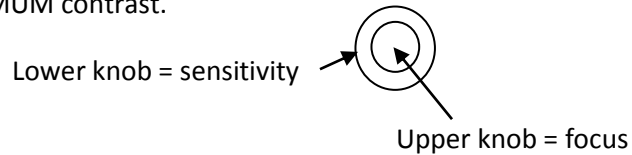
Use the Z axis buttons on the right panel to minimize the image shift. For most samples this will be at a Z value between -40 and -80 um. Note that the contrast also decreases when you reach the eucentric height.



BEAM ALIGNMENTS

Look at some features on your sample, and increase the magnification into the SA range.

Set the focus for MINIMUM contrast.



Check condenser centering:

Use Intensity knob (counter-clockwise) to condense the beam to its minimum size.

Use the trackball to center the spot on the circular target.

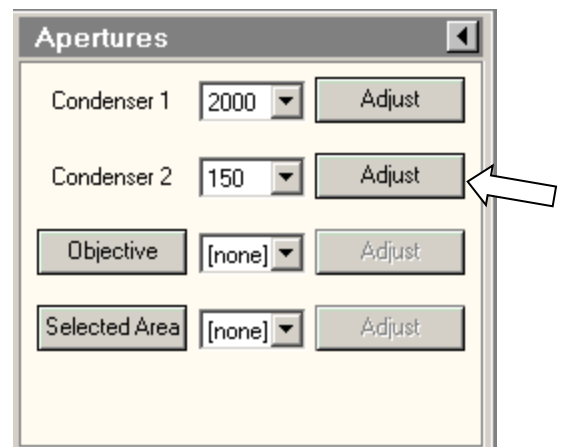
Expand the beam with the intensity knob (clockwise) out to the larger circle.

If the beam does not meet the circle evenly, you should adjust the C2 aperture position

Adjust the C2 condenser position by using

Workset → Tune → Apertures

and then click on
Condenser 2 Adjust



The multifunction knobs will now control the condenser aperture. Move the aperture so that the expanded beam lines up with the outer circle. Then condense the beam to minimum, and iterate if necessary.

Check condenser stigmatism

Look for a circular spot at the intensity crossover (use the Intensity knob).

If the crossover is elliptical or weirdly shaped instead of round, then we need to adjust the condenser stigmatism.

Ask for help if the condenser needs adjustment.

Always leave the Intensity knob set so that the beam spreads when the knob is turned clockwise.

Focus and Objective Stigmatism

The screen is down, right? If not, use R1 to lower the screen.


Set the magnification into the "SA" range, about 39kx.

Set focus  for minimum contrast.

Use the Intensity knob to condense the beam, and use the trackball to center it.

Spread the beam by turning the Intensity knob **clockwise**.

Find some amorphous material to view.

Raise the screen with R1 


Turn on the camera with Workset → Camera → CCD/TV → Search

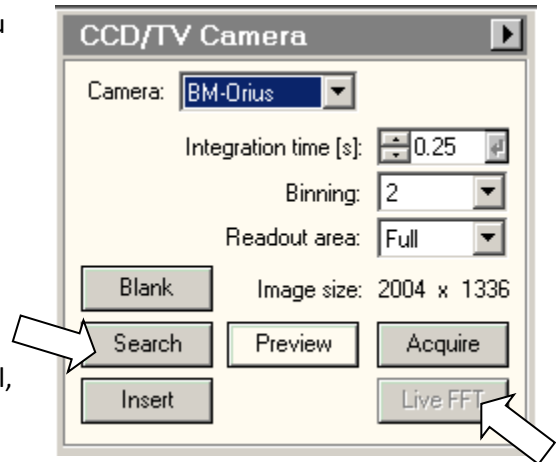
(This should insert the camera automatically, so you do not need to click on “insert”.)

Click on Live FFT

Go a bit out of focus, to see rings in the FFT.

Condense the beam a bit (counter-clockwise) if the rings are unclear.

Press the Stigmator  button on the left panel, or find it on the Tune tab. The multifunction knobs now control the objective stigmation.




Look at the FFT. The best focus is when all the rings spread out and disappear. But we need a few rings to check stigmation.

The multifunction knobs are controlling objective stigmation, right? Use those knobs to make the FFT rings more circular. Adjust the focus closer to ideal (no rings) and adjust the stigmation again. Iterate until you are happy. Or, less sad.

When you are done with stigmation, it's smart to click on None so you will not accidentally change the stigmators by turning the multifunction knobs.

Take some pictures already!

Find an interesting region, with the screen down (that is, using the flucam)

Lift the screen with  R1

Workset → Camera → CCD/TV → Acquire

Look at the **integration time** for image acquisition. If the image is not drifting, then use 1 second. If the image is drifting slightly, use 0.25 seconds.

If the image is drifting rapidly, try tapping on the sample rod again. Or, you might just have to wait 20 minutes or so for the sample to reach equilibrium.

Save the image on the support PC ("TecnaiD601PC") in your own directory.

Move your image files to the YINQE server (there is a shortcut on the PC desktop) or use a web browser to dump them into your Yale Box account.

The support PC is NOT backed up. Files on the support PC will be deleted randomly, without notice.

STEM OPERATION FOR XRAY MAPPING

If you are looking at a crystal, use TEM mode first to set the alpha and beta tilt.

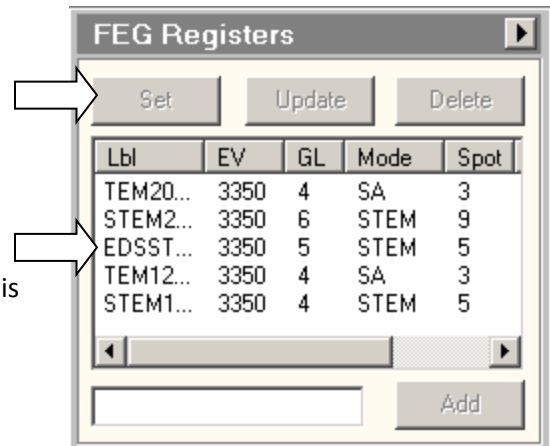
In TEM mode, position the sample over an open region. Later we will need to view an amorphous region, such as carbon film or glue. Set the magnification somewhere in the M or SA range, so that you'll see an edge of the sample when you switch to STEM mode.

Load standard STEM conditions with

Workset → Setup → FEG Registers → EDSSTEM200Kv

and click on "Set".

In the STEM Detector box, make sure the HAADF camera length is set to **220 mm**.



Do you need to do any alignments? Probably not!

But let's just do a quick check:

Press the "eucentric focus" button on the right panel.

Lower the magnification. Your sample will be out of focus.

Focus with the Z buttons. This will be a "coarse" focus. Fine focusing can be done with the "focus" knob.

Zoom in on your sample and look for astigmatism just as you would on a SEM. Pressing the "stigmator" button will attach the multifunction knobs to the condenser stigmation control.

XRAY SPECTROSCOPY

You don't need to use STEM mode to get an x-ray spectrum! If you do not care about resolution, then simply condense the beam on a region of interest and grab a quick and dirty spectrum.

Turn on the x-ray detector with

Workset → STEM → Super X EDX  Pulse processor → On

→ Acquire

The spectrum should appear in the TIA window, on the right. After a few seconds you can click on Acquire again to stop. Did you see that count rate? Pretty amazing.

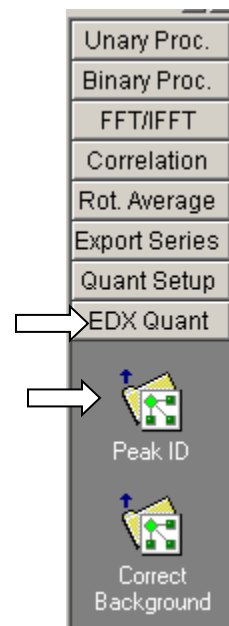
IF you are starting from STEM mode, then acquire a STEM image and freeze it.

Move the reference point  to a place of interest on the image

Super X EDX → Acquire

Now the spectrum should appear on the TIA window.

To identify the elements, choose "EDX Quant" on the left side of the TIA window and then click on "Peak ID".



XRAY MAPPING

First get STEM mode running, so you can use the x-ray detector while scanning the beam. If you need a high count-rate, then choose the FEG Register “EDSSTEM200Kv” instead of the high-resolution register “STEM200Kv”.

Refer to the section “STEM Operation” above for aligning the column.

You probably do not have to...
Turn on the x-ray detector with

Workset → STEM → Super X EDX  Pulse processor → On

Esprit is probably running already – look on the task bar. If not...
Fire up Esprit from the lower-left icon tray



Click on Login. The user name and password are both “edx”.

Click on **Hypermap**

New

Image stabilization!

Acquire

Acquire again to stop

Click on the little bed icon that is supposed to look like a periodic table.

Point Line scan Mapping HyperMap

Image scan Preview New Map data Acquire QMap Quantify

Free regions

H	F1	F2	F3	F4	F5	F6	F7	F8	He								
Li	Be	Inputs						B	C	N	O	F	Ne				
Na	Mg	I1	I2	I3	I4	I5	I6	I7	I8	Al	Si	P	S	Cl	Ar		
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	Ac	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	
			Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lw	

C New element Clear all Auto

Choose two or three elements to display in the xray map

Click on Acquire

Of course there are all sorts of parameters you can set. Feel free to poke around Esprit.

Esprit has a very strange menu structure. Many important functions, such as “save” can be found by clicking on



Once you have an xray map, you can have Esprit display an average spectrum of selected regions. Use the **eye dropper** (lower right) to select a region and then click on the “Spectrum” tab to see the averaged spectrum. Who would have guessed to use the eye dropper!

Use the “Auto” button next to the periodic table to identify elements in the spectrum.

Be sure to save

The composite image

The individual images

The complete database

WHEN YOU ARE DONE

0. Delete all images in TIA, using window → close all
1. If you are in STEM mode, go back to TEM mode using the FEG Register box.
2. Remove the objective and SA apertures, if necessary
3. Lower the screen with R1

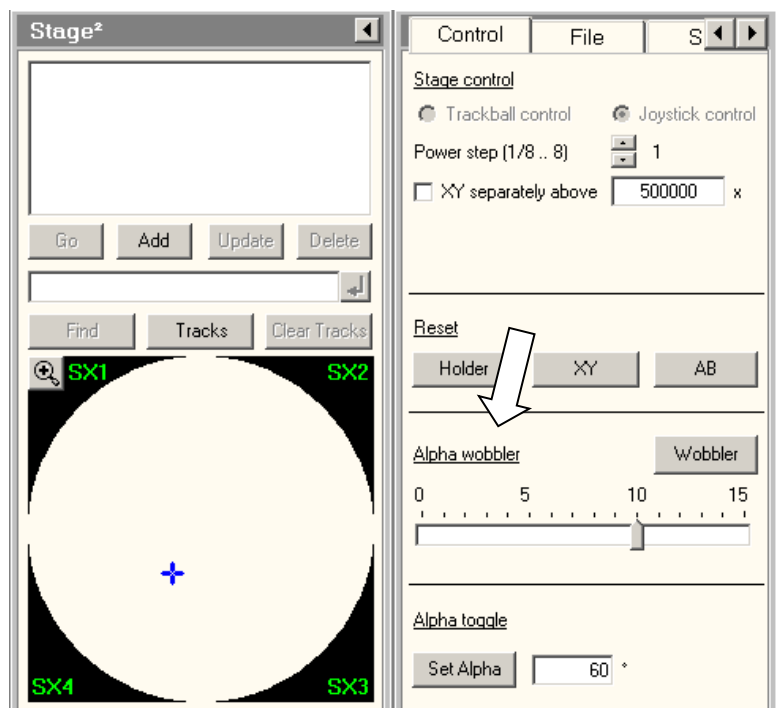
4. Close the column valves with

Workset → Setup → Vacuum →
Col. Valves Closed

5. Reset the stage with

Workset → Search → Stage ▶
Reset Holder

This is very important, to avoid
breaking the rod as your remove it.



6. Remove the sample rod (see below)
7. Remove your sample
8. Leave the rod in the plasma cleaner, pumping.
9. Log out of Badger (unless you want to spend lots of money)

REMOVING SAMPLE ROD

1. RESET THE STAGE

Workset → Search → Stage → Flapout → Reset Holder

2. Pull the rod straight out until it stops, about 3 inches.
You'll see the blue line on the rod.
Turn the rod clockwise until it stops – GENTLY

3. Very gently pull the holder out a little way more,
until it pops loose from vacuum.

5. Pull the rod out the rest of the way. Try to avoid hitting
anything on the way out.

Leave the rod pumping in the plasma cleaner.

