OSIRIS TEM CHEAT SHEET

V.6

BEFORE YOU START

0. Log on with Badger. This will turn on the monitors. Everything else is always running (hopefully).

1. Check the vacuum status, which should be "COL VALVES".

2. Check the high voltage (far right tab). The "high tension" button should be yellow and the voltage should read 200 kV. The "operate" button should also be yellow. If the high voltage is not on, click on the "high tension" button, then click on the "operate" button. The system will not let you push these buttons until the pressure is low enough.

Load your sample in the holder and then...

TURN ON THE BEAM

Workset \rightarrow Setup \rightarrow Vacuum \rightarrow 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.

Vacuum (Use	r)		Þ
Status: CO	L. VAL	VES	5
Pressure			
Gun Column Camera Buffertank Cold trap LN2		1 6 20 34	Log Log Log Log 56 %
Col. Valves Closed			

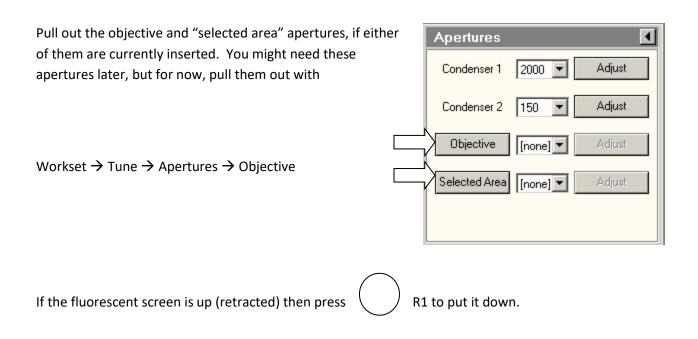
Load standard TEM conditions with

Workset \rightarrow Setup \rightarrow FEG Registers \rightarrow TEM200Kv then click on "Set".

Do NOT click on "update", "delete", or "add".

FEG Registers				
Set	(Jpdate	:	Delete
LЫ	EV	GL	Mode	Spot
TEM20	3350	4	SA	3
STEM2	3350	6	STEM	9
EDSST	3350	5	STEM	5
TEM12 STEM1		4 4	SA STEM	3 5
•				Þ
				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). Go back to the top of this cheat sheet and re-read "Before You Start".



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

Press the "Eucentric Focus" (

) button.

This will set the objective lens to a standard value.

IF you have a lacy grid, then simply adjust Z to minimize the contrast. That's right: *minimize the contrast*.

If your grid is a continuous film or if you are looking at a crystalline cross-section then you will have to

find the eucentric height by wobbling the sample and then adjusting Z to minimize the image shift

Workset \rightarrow Search \rightarrow Stage \rightarrow 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. Note that the contrast also decreases when you reach the eucentric height.

Stage ²	Control File S
	Stage control
	C Trackball control 💿 Joystick control
	Power step (1/8 8) 📑 1/2
	XY separately above 500000 x
Go Add Update Delete	
4	
Find Tracks Clear Tracks	Reset
€ SX1 SX2	Holder XY AB
+	Alpha wobbler Wobbler
	Alpha togale
SX4 SX3	Set Alpha 60 *

COLLECTING IMAGES

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

Set the focus for MINIMUM contrast.



OPTIONAL: 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 meet the circle evenly, you should adjust the C2 aperture position

	Apertures	•		
	Condenser 1	2000 💌 Adjust		
Adjust the C2 condenser position by using	Condenser 2	150 💌 Adjust		
Workset \rightarrow Tune \rightarrow Apertures	Objective	[none] Adjust		
workset 7 fulle 7 Apertures	Selected Area	[none] Adjust		
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.

Focus and Stigmation

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

Make sure the Intensity knob (the condenser) is set so that the beam <u>spreads</u> when the knob is turned <u>clockwise</u>.

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



Use the Intensity knob (the condenser) 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.

Using the Camera

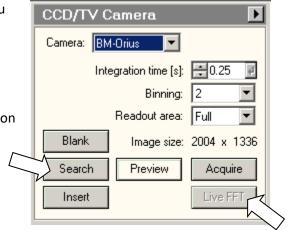
		<u>۱</u>
Raise the	screen with R1)

Turn on the camera with Workset \rightarrow Camera \rightarrow CCD/TV \rightarrow 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. It is easier to see the rings at higher magnification, and on thicker amorphous material.



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

Press the Stigmator \bigcirc button on the left panel. 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, be sure to click the stigmator button again, so you will not accidentally change the stigmators by turning the multifunction knobs.

Take some pictures!

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

Lift the screen with () R1

Workset \rightarrow Camera \rightarrow CCD/TV \rightarrow Acquire

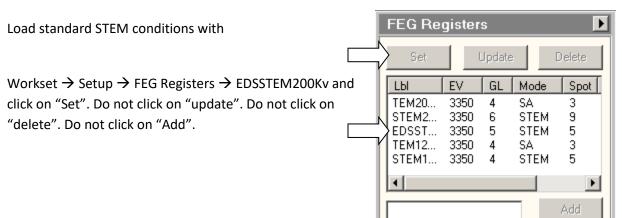
Look at the **integration time** for image acquisition. If the image is not drifting, then use 1 second. If the image is drifting slightly, use 0.25 seconds.

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 (the Z: drive) in your own directory. **Right click** on the image (in TIA) then "Export Data", then choose the format "PC TIFF with scale marker (full res)".

Move your image files from the support PC to your favorite network storage. Do not use USB memory sticks. Images on the Z: drive will be deleted without notice.

STEM OPERATION FOR XRAY MAPPING



Workset \rightarrow STEM \rightarrow Search button

This will insert the HAADF detector and will start scanning the beam. On the right monitor, TIA should display the image.

The image should have a **dark** background and your sample should be **bright**. If not, you need to adjust the position of the beam:

Skip this step if the image is ok. Otherwise...

On the STEM tab, in the Direct Alignment menu, choose Diffraction Alignment.

Now use the multifunction knobs to put the beam in the center of the HAADF detector (you can usually see the shadow of the detector in the middle of the screen.) The important thing is to adjust the beam position so that the background of the image is uniformly **dark**.

Click <u>Diffraction Alignment</u> again to release the multifunction knobs.

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

Lower the magnification all the way. 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. If the image gets streaky when out of focus, correct the stigmation just as you would on a SEM. If you do not know how to operate a SEM then you should be ashamed. Chances are that the stigmation needs very little or no correction.

XRAY SPECTROSCOPY

You do not need to use STEM mode to get an x-ray spectrum! From TEM mode, you can simply turn on the x-ray detector (on the STEM tab) and then collect a spectrum using a high current (spot 3).

Turn on the x-ray detector with

Workset \rightarrow STEM \rightarrow Super X EDX Pulse processor \rightarrow On \rightarrow Acquire

The spectrum should appear in the TIA window, on the right. When you get tired of collecting x-rays, you can click on Acquire again to stop. Did you see that count rate? Pretty amazing.

IF you are starting from STEM mode, then you can collect a spectrum the same way. In STEM mode we normally use spot 5, which is a lower beam current than TEM mode's spot 3.

XRAY MAPPING

First get STEM mode running, so you can use the x-ray detector while scanning the beam

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

Workset \rightarrow STEM \rightarrow **I** Super X EDX Pulse processor \rightarrow On

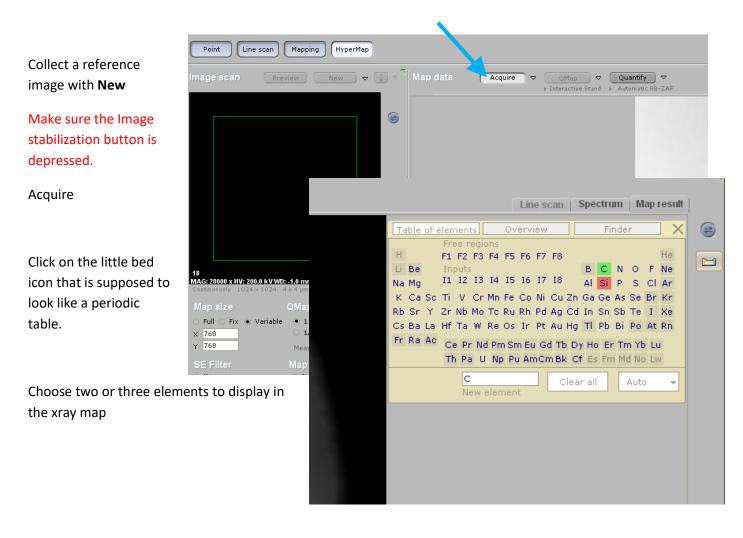
Click on "search" again to **stop** the scan. Now we want to scan with Esprit instead.

Find Esprit on the task bar. Click on Esprit to bring it to the front of the right-side monitor.

XRAY MAPPING WITH ESPRIT

Click on Hypermap

Test the connection by clicking on **preview.** If the preview window does not show a scanning beam, then you must ask for help to reboot the computer. You cannot reboot by yourself. Really.



Esprit has a very strange menu structure. Many important functions, such as "save" can be found by clicking on the round menu buttons.

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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.

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 \rightarrow 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 \rightarrow Setup \rightarrow Vacuum \rightarrow Col. Valves Closed
- 5. Reset the stage with

Workset \rightarrow Search \rightarrow Stage Reset Holder

- 7. Remove the sample rod (see below)
- 8. Remove your sample
- 11. Log out of Badger

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Go Add Update Delete	
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Find Tracks Clear Tracks	Reset
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SX4 SX3	Set Alpha 60 °