

**Portland State University**

**Department of Physics**

**Operational Procedures for**

**Tecnai F-20 TEM**

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## **Table of Contents**

Prep Steps:.....	4
Prep Table Guidelines:.....	4
Double Tilt Holder:.....	4
Single Tilt Holder:.....	5
Status of Microscope: .....	7
Logging In:.....	7
Microscope Operation: .....	7
Control Hints:.....	8
Adjust Condenser Lens Astigmatism .....	10
Adjust Eucentric Focus (Wobbler).....	10
Gun Alignment and Beam Alignment (for advanced level of operation only!) .....	11
Adjust Objective Lens Astigmatism:.....	11
IMAGING:.....	12
Take Digital Image .....	12
Take Diffraction Pattern .....	13
Bright Field Imaging.....	13
Dark Field Imaging.....	14
STEM in the Analytical Mode and EDX Spectrum .....	15
EDX: Line Scan.....	16
EDX: Mapping .....	17
EDX Shut Down Procedure:.....	19
TEM Shut Down Procedure:.....	20

**The followings are a short list of must-do processes (bottom line) for the usage of TEM. You must be able to perform it without looking at the instruction sheet.**

To start:        Make sure: V7 valve closed; Stage at home. Insert the specimen holder to the column. Select the type of holder you are using. If the double tilt holder is used, one should connect the B cable following the instruction from the computer. Wait until the red light on the specimen stage is off and the Process Information Timer in the Vacuum Overview window has run to zero and the message reads “Column Valves Closed”. ***Press the purple part of the stage during the following whole process.*** Rotate holder counter-clockwise until the stopper position. You will feel that the vacuum is trying to pull the holder in. Gently let the holder slide into the column

To end:        Make sure: V7 valve closed; Stage at home. ***Press the purple part during the following whole process.*** Gently pull the specimen holder out until the stopper position. Rotate clockwise until stopper position. Pull the holder all the way out.

Do the Cryo Circle if you are the last user. Do not forget to cover the computer keyboard, STEM detector when you do the Cryo Circle. Fill the liquid nitrogen full if somebody will use it after you. Be aware that the cold trap will last for less than 5 hr when filled full, implying that the next user must show up within reasonable time.

### ***Prep Steps:***

1. Sign the Log sheet. Check to see if there are any issues with the microscope before you continue.
2. Fill LN2: Fill the small LN2 canister. Check the LN2 container to see if there is any left. If there is condensation built up inside, pour out the LN2 and refill. Water left in canister and combined with fresh LN2 will cause the LN2 to boil over. It is best to start with fresh LN2. When filling the LN2, use paper towels and move keyboard, monitor out of way to ensure that no LN2 on them. Cover the hardware buttons, keyboards with the plastic sheet.
3. Fill the LN2 to about 50% and mount the LN2 canister to the TEM slowly letting the copper coils into the LN2 canister. Use the second LN2 canister to top off the 1<sup>st</sup> canister and place the Styrofoam top on LN2 canister.

### **Prep Table Guidelines:**

**NOTE:** Be careful not to loose any of these pieces. There are three. The wrench, screw and washer.

1. Turn on the optical microscope and place the holder up against the scope. Carefully remove the plastic end piece cover.

### **Double Tilt Holder:**

2. There is a small tool used to remove the top cover hex screw on the sample holder. The tool is stored in a plastic vile.
3. Use the small tool “wrench” and looking through the microscope unscrew the small screw on the top of the sample holder. Place the wrench and screw in a safe place.
4. Then turn the sample holder upside down and let the washer fall out.
5. Turn the sample holder back upright and load your TEM sample grid to the sample holder with the copper side up.
6. Now place the washer back in the sample holder. Notice that it goes in a specific orientation.
7. Then place the screw back on top and using the wrench, screw it back in. Make sure that screw is all the way in and does not extend above the sample holder. Also be careful not to tighten the wrench so much as to break your sample or holder.

8. Now turn the sample upside down and tap on it to make sure the sample is secure and does not fall out in the TEM or on the way to the TEM.
9. Remove the sample holder from the plastic protective sleeve. Now while looking under the microscope, check for debris on the O-Ring. If the O-Ring is dirty use a Kleen wipe and clean the O-Ring.
10. Now the sample holder is ready to be loaded into the TEM.

**Single Tilt Holder:**

1. Using a small pin type tool. Place the pin into the end piece of the sample holder and gently lift up the top of the holder. There is no washer as is with the Double Tilt holder.
2. Place your sample into the holder and then using the tool, close the holder again.
3. Now turn the sample upside down and tap on it to make sure the sample is secure and does not fall out in the TEM or on the way to the TEM.
4. Remove the sample holder from the plastic protective sleeve. Now while looking under the microscope, check for debris on the O-Ring. If the O-Ring is dirty use a kleen wipe and clean the O-Ring.

Now the sample holder is ready to be loaded into the TEM.

**Cryo Transfer Holder:**

1. Pump down the Cryo Transfer holder on the station: Insert the holder to the station; connect the pumping line to pump the vacuum for the liquid nitrogen dewer; open V1 and V2 valves; when the vacuum reaches high degree, open the valve on the holder to pump the vacuum for liquid nitrogen dewer. Holder must be pumped overnight before usage.
2. Close the valve on the vacuum line; Close V1 and V2 valves; Pull holder out; Pull the vacuum line out of the holder.
3. Load specimen to the holder: load specimen to the holder with the main face up; rotate screw in
4. Rotate the screw that is used to clamp B cable off the stage: because the existence of this screw prevent the lever of the holder lying perfect horizontal;

Send the holder to the parking position and send the holder into the column in a usual way;

5. Connect the temperature controlling/monitoring and rotation controlling cable to the holder; Turn on the powers to both of the control unit; For the temperature controlling/monitoring unit, press “set” and “raise”/”decrease” button simultaneously to set the desired temperature. Knowledge test: what is the liquid nitrogen boiling temperature (-196C). Quiz: What temperature can we obtain with this cryo holder? (-182.6C). If you set temperature lower than -196C, the specimen will be cooled down simply by liquid nitrogen. If you set it to a relatively higher temperature, e.g., -100C, the heater inside the holder will be turned on to obtain the desired temperature.

## **Status of Microscope:**

### **Logging In:**

1. Sign in with your user account. Press CONTROL – ALT – DELETE and sign on.
2. Click the “Application Launcher”: It is the little rocket Icon on the taskbar. The launcher will open up three applications and they are as follows:
  - a. Tecnai User Interface
  - b. Gatan Digital Micrograph
  - c. TIA Tecnai Imaging & Analysis
  - d. Gatan Filter Control
3. Launcher will show auto start checks next to these four applications and if you just click Start they will start automatically.
4. WAIT ! WAIT ! WAIT ! ( 3 ~ 5 min ) It will take a while for the applications to boot up. So it is not wise to start pressing buttons while they boot up. Remember this is a delicate machine and patience is a virtue.
5. You will see an ERROR Message: “Could not load Plug in Gatan CCD.dll” Just click OK.

### **Microscope Operation:**

1. Check various parameter before operating microscope. In the bottom right corner of the screen is Tecnai G<sup>2</sup>. Use the pull out menu and select Vacuum Overview so you can see the vacuum system as you make changes to the microscope.
2. Click “Vacuum” tab in Workset Window(Upper left screen) to make sure the following numbers are met:

* Gun	1	Log
* Column	6	Log
* Camera	~24	Log
* Buffer tank	~33	Log
* Backing Line	~50	Log
- 2.5 From TecNai G<sup>2</sup> pull down menu, select vacuum overview menu.
3. Turn on “Turbo on” button to accelerate the turbo pump.

- 3.5 Make sure that the stage is at home.
- 3.6 Make sure that V7 is closed.
4. Check specimen holder: before inserting the specimen holder, one has to check the holder to make sure that the specimen is properly mounted (tap the specimen holder a little bit) and be sure that the rubber “O ring” of the specimen holder is clean.
5. Insert the specimen holder then select the specimen holder on the screen and then click “Enter”. If the double tilt holder is used, one should click “Connect B-tilt Cable” and then click “Enter”. Wait until the red light on the specimen stage is off and the Process Information Timer in the Vacuum Overview window has run to zero and the message reads “Column Valves Closed”.
6. ***Press the purple part of the stage during the following whole process.*** Rotate holder counter-clockwise until the stopper position. You will feel that the vacuum is trying to pull the holder in. Gently let the holder slide into the column
7. Check “Column Vacuum” in the vacuum page, it should be  $\leq 10$  log. If the vacuum is higher than 10 log, one should wait the vacuum to recover before advance to next step. Click Turbo on to turn it off.
8. Click “Column Valve Close” button to open column valve. This step will allow the FEG to expose to the column atmosphere. If the column atmosphere is higher than 10 log, it may damage the FEG (Crying together is the only thing we can do if this happens). After the column vale is opened, the electron beam should appear on the viewing screen. If no electron beam appears on the viewing screen, it is possible that the specimen blocks the beam. One should move the specimen slightly away from the beam.

**Control Hints:**

**Magnification:** Make adjustments to the magnification knob slowly.

LM Low Magnification

M Medium Magnification

SA Select Area

MH Highest Magnification

**Spot Size:** Larger Spot Size # 9 = Small Spot, Smaller Spot size # 2 = Large Spot



**Focus Beam:** Use the focus knobs to adjust the focus. The beam size changed based on changes made by the focus knob. Use the track ball to center the beam. Use the intensity knobs to widen the beam.

### **Adjust Condenser Lens Astigmatism**

This and almost all the beam alignment procedure should be done in a hole area of specimen.

1. Select Objective Lens aperture # to 7.
2. Make sure the Selective Aperture (SA) is out. (Bar to the right)
3. Focus the beam and center it with the beam shift control track ball. Widen the beam with intensity knob. Center the beam by adjusting the Condenser Lens aperture, adjusting the X and Y knobs at the aperture (The condenser lens aperture control knobs are located above the specimen holder position).
4. In “Microscope Control”, find the Stigmator Window, Click the “Condenser” button. At this time, the multifunction X, Y buttons become active for adjusting condenser stigmatism. Set magnification higher than 100K to check the stigmatism and use multifunction X, Y buttons to correct the stigmatism. After the correction, the beam spot should appear perfect spherical. When done, click the None button.

### **Adjust Eucentric height (Wobbler)**

1. Select a distinguishable feature in the specimen.
2. Focus the image over the binoculars.

Control the Z height: On the stage tab, click on the flip out and then you will be using the wobbler control to correct the Z height. Start out at a lower magnification (SA mode) and click the Wobbler. Use the intensity to fill the screen. Then the object is to minimize the movement of the image by pressing the + or – buttons.

Gun Alignment and Beam Alignment (for advanced level of operation only!)

If you are a beginner, it is recommended that you escape this process and directly go to next step (Adjust Condense Lens Astigmatism).

1. **GUN SHIFT:** Select condenser lens aperture # 4. For Gun Alignment use a Gun Lens of 5.
2. **Select Gun Shift:** Find “Direct Alignments” window. Highlight “Gun Shift”.
3. **Then select spot size #9** (the beam will be very dim. To find the beam, you have to reduce the magnification). **Use the track ball** on the left panel to move the beam to the center of the viewing screen.
4. **Change the spot size to #2.** At this time, the beam will be very bright. You should first focus the beam using the “Intensity” knob located on the left panel. Then **use multifunction knobs (X and Y)** to move the beam to the center of the viewing screen. You should repeat these steps for at least twice. Then click “Done” in “Direct Alignments”.
5. **GUN TILT:** Make sure spot size is 2. Find a vacuum area in the specimen. Increase the magnification to about ~100K. Focus the beam using the “Intensity” knob in the left panel. Go back to “Direct Alignments” page. Highlight “Gun Tilt”. Then **use multifunction knobs (X and Y)** to center the brightest small spot in the focused beam. Then click “Done”.
6. Check condenser stigmatism again and adjust it necessary.
7. **Beam Tilt PPX** – Click on Beam Tilt PPX and use the **multifunction knobs (X and Y)** to move the two spots until they superimpose one another.
8. **Beam Tilt PPY** – Click on Beam Tilt PPX and move the **multifunction knobs (X and Y)** to move the two images until they superimpose one another.
9. **Rotation Center (Objective)** –

#### Adjust Objective Lens Astigmatism:

1. Find and amorphous area of the carbon grid.
2. Increase Magnification to more than SA 100K X.
3. Insert the CCD Camera and search for an image.
4. In the Digital Micrograph Window, Click Process → Live → FFT.

5. Lift the screen
6. A correct objective stigmatism corresponds to a nice sphere FFT.
7. In stigmator window click Objective. When done, click “Done”. Use the multifunction X, Y knobs to adjust the sphere.
8. Defocus and continue to correct the stigmatism to get a better sphere.

### **IMAGING:**

1. ***Saving Specimen Locations:*** On the stage tab you can add and save the specific location settings so that you can easily visit them later.
2. You can easily locate and move to the positions by using the “MOVE TO” button.  
IMPORTANT NOTE: The “MOVE TO” button is safer to use than the “Go To” button. If the sample is tilted and moving between saved locations which are at different z heights or  $\alpha$  or  $\beta$  angles, the “MOVE TO” button will safely get you there. The Go To button may damage the microscope because it moves directly to the new location without choosing the safest way to get there.

### **Take Digital Image**

1. Insert Gatan CCD camera by clicking “Camera” on “Worksheet”. In “Controller” select “Gatan CCD” and then click “Insert” radio button. Flip out this page and select setting, make a full camera area for search, preview, and acquire mode.
2. Lift screen by clicking “R1” on right panel.
3. Press radio button “Search” to find the specimen area. One can move the track ball on the right panel to get the desired specimen area. Then stop the “Search” by clicking stop bar of the computer.
4. Click “Preview” to get an image on the computer monitor and adjust the fine focus to get the best focus. Then click the stop bar of the computer to stop “Preview”.
5. Click “Acquire” to get the final image.
6. In “Digital Micrograph” page, click “Edit” manual and select “Data Bar” to label the image.
7. Save the image: Click “File” manual in the “Digital Micrograph” page and select “Save As”. In the “Save in” window, search for “Off\_Line PC on D544b521” and

then select “Tecnai Files”. During the saving the image, one should save in Gatan Format (\*.dm3). Saving in this format, it will allow one to process the image off line in the computer located in the lobby of the TEM room.

8. Add a Scale Marker Bar: Select the image window, then click Edit, Databar, Add Scale marker.
9. Adjust Scale Marker Bar: You can move the marker bar around your image or even change its dimensions by grabbing a corner of the marker bar window and dragging to change it.

### **Take Diffraction Pattern**

This process is for someone who wants to take a diffraction pattern.

1. Specimen Tilt: There is a  $\alpha$  tilt (Front to back tilt) and a  $\beta$  tilt (Left to Right). The tilt is used to find the “Zone Axis”.
2. Adjust the Eucentric height and focus image to minimum contrast.
3. Press the Diffraction button on Right Control Panel.
4. Using the  $\alpha$  and  $\beta$  tilt to tilt the specimen to a desired Zone Axis.
5. Change the camera length to a desired value. 600 mm is a good setting
6. Use the needle on the right hand side to block out the bright spot. (transmitted spot). We block the spot because we do not want to burn out the camera or the phosphorous screen.
7. The multifunction X, Y knobs can be used to move the Diffraction pattern on the stage screen.

### **Bright Field Imaging**

1. Select Diffraction Mode.
2. Tilt sample to the zone axis.

3. Use the smallest objective aperture, (with the Select Area out) to see the transmitted spot in the center only.
4. Switch to image mode.
5. Just under focus the image and this is the Bright Field image using the transmitted beam.

### **Dark Field Imaging**

1. Select the Diffraction Mode.
2. Tilt sample to the zone axis.
3. Move the transmitted spot to the screen center by using the multifunction knobs. Selected the smallest objective lens aperture, and adjust its position (with the Select Area out) to see the transmitted spot in the center only. Then remove the objective aperture.
4. Press Dark Field Button and use the Multifunction X and Y knobs to move a desired diffraction spot to the center of the phosphorous screen.
5. Switch to image and take a picture.

## **STEM in the Analytical Mode and EDX Spectrum**

STEM ALIGNMENT (make sure HRTEM alignment is done properly before attempting)

IN Microprobe mode...

1. Withdraw US 1000 CCD camera before proceeding to align for STEM
2. Set C2 aperture at #2 and center it (C2 is smaller than C3, which results in more parallel beam) and center it.
3. Move to a hole area in specimen and align condenser lens and condenser stigmatism
4. At FEG Control box set Gun lens at 5 and extraction voltage at 4300V.
6. At STEM box press STEM button.
7. Flip out STEM window and choose Focus Using at "Intensity & Objective"
8. Deselect Diffraction button on RH microscope control panel
9. Recenter the beam using the beam position track ball
10. Set the spot size for (a) imaging mode at 9, (b) EDS analysis at 7
11. At as high a magnification as possible focus the image to minimum contrast (if loose beam decrease the mag and find the beam)
12. Select rotation center in the Direct Alignment Tab, and make the bright spot to the center of the beam by using multifunction X and Y.
13. Press Diffraction button on RH microscope control panel.
14. Flip out the STEM box and set "Intensity & Objective" Focus back to "Intensity"
15. Insert either the BF (single circle) or DF annular detector (detector is in when the button is backlit)
15. Acquire the STEM image by
  - a) pressing R1 to lift the screen
  - b) At STEM box hit Search Image and adjust the image brightness and contrast by clicking Auto C/B
  - c) if image is out of focus use the Focus knob on the RH microscope control panel (this reduces the size of the beam on the specimen - the smaller the beam size, the sharper the image)
  - d) At STEM box press Acquire button to obtain STEM image
16. Acquire EDS Spectrum
  - a) tilt alpha tilt angle on sample to 15deg (At Stage box flip out set tab, enter 15deg for Alpha, click GOTO)
  - b) Search and Focus the image. Acquire an image.
  - c) Go to Analysis window, select beam position marker and put beam on an area to be analyzed
  - d) view and acquire the EDS spectrum
17. Go to STEM box and press Blank button
18. Save the image and spectrum.

### ***1. To Acquire an EDX Spectrum:***

- Tilt the sample to 15 degree at A angle. In Workset, Stage, Flip out Set Tab,.Enter 15° for Alpha. Click GOTO.

- Search and Focus the image. Then Preview and Acquire.
- Go to the Analysis tab.
- Put the beam onto the area to be analyzed.
- Reduce spot size or adjust spot size to obtain appropriate counts/s, typically 1000.

### **EDX: Line Scan**

1. Acquire a STEM image;
2. Go to Analysis window;
3. Insert a reference point into the image;
4. View and Acquire an EDX spectrum from EDX User.
5. Select a region for drifting correction, using square box icon.
6. Insert a Line profile, with the line Icon. Drag a line on Sample.
7. In Workset, Analysis tab, Experiments window, Select Component:
  - Spectrum Collection
8. Select Experiment:
  - Drift Corrected Spectrum Profile
9. Set up the scan parameters. Click on the flip out window.

### **Line Scan Settings:**

- Acquisition Settings
  - Profile size 10-100
  - Dwell time 2000 – 10,000
- Connection Setting
  - Acquire EDX Spectra            Yes
  - Acquire STEM Images            Yes
  - Elemental Process                No
- Correction Setting
  - Number of Acquisitions in slice        2
  - Reference Image Size (X & Y)        64
  - Reference Image dwell time (μs)        10



- Number of slices per reference 2
- Predict draft rate Yes
- Cross correlation filter start 0.05
- Cross correlation filter end 0.80

10. Highlight the line.
11. Press Acquire button in the Experiments window. Wait until the experiment is done.
12. Go to STEM window and press Blank button.
13. Go to TIA window.
14. Use “Image Position Marker” and click on the line in the STEM line profile image, press Alt key, drag the mouse to the spectrum to link the profile image with the EDX spectrum.
15. Make and Rearrange a new window.
16. Click the Peak ID.
17. Use “Energy window tool” to select an element peak.
18. Paint the peak using this Energy Window Tool.
19. Go to Process, select Extract map profile, click and add a profile in the new window.
20. Double click the left key of the mouse on the top of the profile. A window pops up. Click Show Legend.
21. You can change Name and Style of the profile.
22. Use Marker tool to make a line in the above new window, press Alt key, drag the mouse to the spectrum to link the new profile with the EDX spectrum.
23. If more elements are added on the above new window, repeat the procedure 16-20.

### **EDX: Mapping**

- The Mapping procedure is similar to the Line Scan procedure except some differences at the experiment parameters.
- In general, for Mapping experiment parameters, the image size is 20 \* 20. Non fixed direction.

1. Acquire a STEM image.
2. Go to Analysis window.
3. Insert a reference point into the image.
4. View and Acquire an EDX spectrum from EDX User.
5. Select a region for drifting correction, using square box icon.
6. Insert a Box profile for Mapping, with the box Icon. Place the box on the image.
7. In Workset, Analysis tab, Experiments window, Select Component:
  - Select Spectrum Collection
8. Select Experiment:
  - Drift Correction Spectrum Image
9. Set up the scan parameters. Click on the flip out window.

**Mapping Settings:**

- Acquisition Settings
  - Image Size X            20
  - Image Size Y            20
  - Fixed Direction        none
  - Dwell time              1000
- Connection Setting
  - Acquire EDX Spectra    Yes
  - Acquire STEM Images    Yes
  - Elemental Process        No
- Collection Setting
  - Number of Acquisitions in slice    2
  - Reference Image Size (X & Y)    64
  - Reference Image dwell time ( $\mu$ s)    10
  - Number of slices per reference    2
  - Predict draft rate            Yes
  - Cross correlation filter start    0.05
  - Cross correlation filter end    0.80

10. Acquire a new STEM image to check the image drifting, and highlight the box.
11. Press Acquire button in the Experiments window. Wait until the experiment is done.
12. Go to STEM window and press Blank button.
13. Go back the Analysis window.
14. Go to TIA window.
15. Use “Image Position Marker” and click on the box in the STEM image, press Alt key, drag the mouse to the spectrum to link the image with the EDX spectrum.
16. Make and Rearrange a new window.
17. Click the Peak ID.
18. Use “Energy window tool” to select an element peak.
19. Paint the peak using the Energy window tool.
20. Go to Process, select Extract map profile, click and add a profile in the new window.
21. Double click the left key of the mouse on the top of the profile. A window pops up. Click Show Legend.
22. Name and Style the profile.
23. Use Marker tool to make a line in the above new window, press Alt key, drag the mouse to the spectrum to link the new profile with the EDX spectrum.
24. If more elements are added on the above new window, repeat the procedure 16-20.

**To Export the EDX Data and Files:**

Click File save as, off line PC on (D544b521Z:), Tecnai files, File name “XXXX”

**STEM Shut Down Procedure:**

- Press button to withdraw detector
- In Workset
  - Stem Tab

- Stem Imaging Window
- Click STEM to make button gray.

**TEM Shut Down Procedure:**

1. Extract the camera.

- In Workset
- Camera tab,
- Click “Insert.” It should be Gray when extracted.

**EDX Shut Down Procedure:**

- Press “OUT” button to withdraw STEM detector.
  - In Workset
  - Stem Tab
  - Stem Imaging Window
  - Click STEM to make button gray.
2. On Workset, Vacuum tab, Click “Column Valve Closed”. This is the button that is the exception to the rule. This button should be gray and when you click it and close the column valve it will be Yellow.
  3. Put the Magnification in SA mode (M4000X is ok).
  4. Set the Gun Lens = 5.
  5. Set Spot Size = 2.
  6. Set Objective Lens aperture = 7.
  7. Make sure SA (Select Area) is out. Bar to right = OUT.
  8. On Workset, Stage tab, click the Flip out on Control tab and Click on Reset “Holder” to home the sample holder stage back to the (0, 0, 0, 0, 0) position.
  9. Remove the plastic cover over the sample holder stage. Unplug the B cable if using a double tilt.
  10. You can lower the FEG extraction voltage to 3800V and then continue on to next step.

**Remove Sample Holder**

11. Pull out sample holder until it reaches the park position.

12. Turn Clockwise.
13. Pull out the sample holder the rest of the way.
14. Put sample holder back over at the sample prep table.
15. Cover the stage with plastic cover.
16. Cover the glass windows on TEM with rubber mats.
17. On Workset, Vacuum tab, Click the flip out for Cryo Cycle.  
Cover the key board and STEM detector using plastic sheet.
  - a. Remove the LN2 canister from the side of the TEM and place on prep table.
  - b. Make sure the flip out is out, then Click the “Cryo Cycle” button.
18. Up to this point we are ready to shut down. Wait until the time in the cryo cycle starts and reduces by at least a couple of minutes. This ensures that the cryo cycle does actually start without a problem before you decide to log off.
19. Once you are certain that the Cryo Cycle has started you can close the 4 application windows. One of the windows will ask you, Do you want to save changes? Yes / No. If you have a good alignment on the microscope, then you can click yes.
20. Always say “NO” when the computer asks: “Do you want to save the changes to GIF.2000?”
21. Now you can Click Start, Log Off.

**DO NOT DO A SHUTDOWN OF THE COMPUTER. IF YOU SHUT DOWN THE COMPUTER YOU SHUT DOWN THE TEM AND ALL THE ELECTRONICS THAT CONTROL IT. This will damage the Gun and cost money and take time to bring back up. You can't just turn the PC back on.**

## **GIF Operation Manual**

General advice: GIF is a very sensitive machine, e.g., movement of your steel chair could cause a change in your spectrum. Try to use it when everything seems stable and no one will bother you. Using a wood chair (or do not use any chair at all) might be a good idea.

To start: You need to turn on the TV monitor.

### **I Tuning**

- (1) Do routine TEM alignment.
- (1.2) Go to Camera/Plate Tag and select GIF MSC.
- (2) Go to EFTEM page, make sure that imaging mode is checked.
- (3) Find a hole (vacuum) area and adjust the beam intensity to a suitable extent. This is because we will tune GIF and if you do not adjust the beam intensity properly, the software will come back and bother you.  
Sample condition: Gun lens=5; Extraction voltage=4400; Spot size#=2; Condenser aperture#=3; Magnification (with phosphorous screen down)=4300X; Spot size on screen=1 cm.
- (4) Make this beam centered on the 9:00 o'clock in the small circle and lift up the screen.
- (5) Tune GIF
  - (5-1) Click Align ZLP. This takes approximately 1 min..
  - (5-2) Click Tune GIF. This is a long process (3-5 min.), just wait. Digital Micrograph might ask you to adjust the beam intensity. In that case, adjust the intensity so that it falls to the green region, then hit enter on the key board.
  - (5-3) Repeating (5-1) and (5-2) for the second time is suggested.

### **II Energy filtered imaging**

- (1) Focus the image by using the GIF camera.
- (2) To take an elemental mapping image:
  - (a) Find out the suitable exposure time: Check the custom button and enter a proper value corresponding to the element you are interested. Adjust the binning, exposure time and the entrance aperture diameter of GIF so that the account of each pixel on the image is around 4000.

To change the pixel# for elemental mapping: Go to Auto filter; Flip out page; Choose imaging(filtered)/mapping/Filtered imaging; click view; change binning; click ok.

(b) Perform elemental mapping: Click the elemental mapping button and follow the setting procedures. Enter an elemental symbol/atomic number and click other items to apply the change. Make sure that the slit width is the same as that you used in (a) and enter the same exposure time you found out in (a).

Then, the mapping starts, which takes a couple of minutes. One post-edge and two pre-edge images will be taken. After mapping, the computer will ask you to select an area on pre-edge 1 image. The purpose is that computer will use this region to align the pre-edge 1 image to the post-edge image. Go to DM (Digital Micrograph) “region of interest” tool box, select square tool and draw a rectangular on pre-edge 1 image, click OK. Attempt should be made to choose areas including easily distinguishable features. Then, the computer will go through the alignment procedure. Sometime, the computer will ask you whether to accept computer alignment. Accept it or do it yourself. For almost of the cases, accepting computer results is suggested. When the computer alignment goes well, you will not be asked. Finally, an elemental mapping image will be displayed.

(c) You can take more than one elemental mapping image.

(d) You can color mix the results of different elemental mapping results. For this purpose, click the elemental mapping images sequentially. Go to process and choose color mix. After aligning, click generate. If there are some areas that the images did not overlap each other, click crop to cut the extra margins.

### **III Spectroscopy mode**

(1) Do routine TEM alignment; remove US100 Camera (Camera Page); ensure STEM/DF/BF detectors are out (control box next to TEM); select GIF MSC (Camera Page).

(1.5) Do Energy filtered imaging mode alignment.

(2) Find a hole area and reduce the beam intensity to such extent that you barely see any brightness on the phosphorous screen. This is a **MUST DO** step, otherwise you risk burning the GIF CCD camera. Be sure to center the hole on the screen.

(3) With the phosphorous screen down, go to Auto filter page. Select spectroscopy mode. Microscope control will ask you to confirm that you reduced beam intensity. Confirm it since you have reduced beam

intensity (hit enter). Select zero loss. Set: dispersion=0.05 eV/pixel; all the exposure times=0.01S; GIF entrance aperture=2 mm (2 mm is for normal use, can also set to 3 or 0.6 mm). This is a MUST DO step.

- (4) Lift the screen (R1) and take a search view (or preview) spectrum. If you do not find the zero loss peak, adjust energy shift back and forth.
- (5) Increase the exposure time or beam intensity if necessary. Make sure that the spectrum color stays on blue. Other colors (yellow or red) indicate that the intensity is too high and you should decrease the intensity quickly.
- (6) Put the zero loss peak to the zero position: Read the energy position of the ZLP on the energy axis, type this value to the energy shift window, and click enter. Click align ZLP.
- (7) Do the fine tuning of ZLP (zero loss peak) if you want. There are two methods to do the tuning: either using the TV rate camera or ZLP.

(7-1) Alignment by using the TV rate camera. Check the TV image button and you will see the ZLP image on the TV monitor. Adjust the following parameters so that the TV monitor image appears as a sharp, straight vertical line, which is a good image of the spectrum. Usually, first order focus correction should be enough. Occasionally, second order focus is necessary. Correction of AC components A and B is not encouraged though not prohibited. Note the following:

- (a) First order focus X is bad if the line is broadened; Focus Y: bad if it is oblique.
- (b) Second order focus X: bad if it is curved; Focus SY: bad if it is propeller shaped.
- (c) AC component A and B: bad if the line is broadened or even doubled.

(7-2) Alignment by looking at the ZLP spectrum. Uncheck the TV image button and optimize the spectrum to maximize the zero loss peak height and minimize its width. Spectrum tuning can also be done with the auto filter tuning function.

- (8) Put the zero loss peak to the zero position again. Read the energy position of the ZLP on the energy axis, type this value to the energy shift window, and click enter. Click align ZLP.
- (9) You can take a spectrum from the object you are interested. A few methods are possible for this purpose. Examples, which are widely used, are given in the following. In (9-1), adjustments of the



spectrometer will be illustrated, which will not be repeated in (9-2) and (9-3).

#### (9-1) TEM image mode

(a) Move your interested object to the entrance aperture of GIF. The center of the GIF entrance aperture is on the small circle of the phosphorous screen, at 9:00 o'clock.

(b) Select Custom. Set the energy shift and dispersion value, based on the following ideas.

*Val.Disp*: value of energy dispersion

*Ene.Shift*: Energy shift you entered

Your spectrum will start from  $Ene.Shift - 100 * Val.Disp$  and ends at  $Ene.Shift + 924 * Val.Disp$ . Make sure this range covers your interested energy region.

Be advised that if you lower the energy dispersion, e.g. from 0.1 eV/pixel to 0.5 eV/pixel, the spectrum intensity will increase approximately 5 times. Keep this in mind and pay attention to the adjustment of beam intensity and exposure time so that you do not cause damage to the MSC camera.

(c) Probably, you need to increase the beam intensity and exposure time. Do this gradually and with care. You can click acquire to get a still spectrum instead "Search" or "Preview".

#### (8-2) Diffraction mode

Find an interested area (spot) in TEM mode, focus the beam. Switch to diffraction mode. Put transmission spot to the entrance aperture. Collect spectrum.

#### (8-3) STEM mode

In this case, use only the Dark Field STEM detector. Move the transmission spot to the entrance aperture of GIF. Take a STEM image, put the beam on a feature you are interested. Take a spectrum.

To end: make sure that imaging mode is selected in Autofilter; Turn off TV monitor; When you quit "Filter control" software, never choose "yes" to the question of "Save changes to GIF.2000Data before closing"