INSTRUCTIONS

JEM-2010F

FIELD-EMISSION TRANSMISSION ELECTRON MICROSCOPE WITH STEM CAPABILITY

August 2011

OPERATION

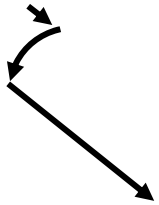
PRELIMINARIES

1. Ensure that **EMISSION** and **HT** are on: The **HT READY** and **FEG READY** lights (left panel) should both be green. The **EMSN** button on the same panel should be bright and the LED display should show an emission current of ~130 to 150 μA. The **HT** LED display should indicate a non-zero value. Take note of the HT value shown on the small monitor to the right of the column. It is normally 200 kV, in which case the **HT** LED display will read ~ 97 μA. If it is not 200 kV, see step 8 below.
2. Check the gun vacuum: All ion pump gauges for the gun should read ~zero.
    a. **GUN: 60 l/s:** This should read ~0 on the 10^{-6} Pa scale (red scale 75 l/sec)
    b. **EMITTER: 15 l/s** (actually 3x 5 l/s pumps located at emitter) Typically reads ~1 μA.
    c. **GUN V1 Valve: 20 l/s** SIP (located at gun isolation valve) - < 0.1 x 10^{-6}Pa
3. Check that the pressure reading on the column SIP power supply is ≤ 2 ×10^{-5} Pa.
4. Make sure the V1 **VALVE** is closed: The **VALVE** push button will not be brightly illuminated and the green LED for V1 on the vacuum schematic will be out. This valve isolates the gun vacuum from the column vacuum and should be closed whenever the microscope is not in use and must be closed during specimen exchange.
5. Ensure that the x-ray EDS detector is retracted.
6. Check that Gatan Instrument Bin is powered up, camera controllers (Ultrascan and Enfina cameras) are on, and the Ultrascan camera is at -25 C.
7. If the ACD has not already been cooled by a previous user, fill the dewar with liquid nitrogen. (Make sure the viewing screen window is covered.) When boiling stops, fill it again. If it was cold when you arrived, top it off.
8. Ensure that on the JEOL computer FasTEM Server and FasTEM Client are running. If not, launch the Server. When the server GUI minimizes, restore it and enter a password (jem-2010f). Launch the Client (username = user, password = jem-2010f). If the HT is not at 200 kV, use the FasTEM Client to increase it.
9. Login to the Gatan computer using your PSU credentials (dce domain).
10. Launch the web browser and log in to the Research Instrumentation Management System (http://www.rims.psu.edu/) using your PSU Access credentials.
    a. Login to Lab Time. (Choose Lab Space (MCL).)
    b. Login to Equipment.
    c. Close the web browser.
11. Launch the software applications. (Note: The FasTEM computer and the Emispec computer share the same keyboard, monitor and mouse. To switch between them hit the “Scroll Lock” key twice and then press “1” for FasTEM or “2” for Emispec.)
    a. Launch Filter Control
    b. Launch DigitalMicrograph (DM).
    c. If you need to use the X-ray EDS system, on the Emispec computer launch RTEM Control and launch ES Vision. (Due to software bugs, the Emispec computer is normally run with the Admin account: username = admin, password = jem-2010f.)

LOADING THE SPECIMEN

Wear gloves when handling the sample holder. Do not touch any part of the holder rod with your bare hands.

Load your TEM samples in the specimen holder you wish to use. There are four ambient temperature holders available:



Plug Removal

1. Ensure that the switch is set to AIR.
2. Pull back on lever and pull plug out (~1 mm). Turn to the left until it stops. ***DO NOT PULL OUT!!***
3. Wait for green light to go out. Then remove plug.

- Standard (single-tilt) JEOL holder: This holder has two parts: the rod portion and the end piece that holds the sample. Before assembling the two parts, load the sample into the end piece, which should be positioned on its plastic support. Sample is secured with a spring clamp that is secured with two small screws. Then use the special pin to open the clamp at the end of the rod. Using forceps, place the end piece in the clamp and allow the clamp to close.
- Gatan double-tilt analytical holder: Using the magnifier lamp to better view the cradle, gently place the sample in the cradle. Carefully (and gently) screw the hex nut into the holder. Insert the nut with the large end up. Be very careful not to cross-thread the nut and do not use excessive force. The nut will bottom out after a small fraction of a turn (typically less than ~1/4 of a turn). Turn the sample holder upside down and tap gently on the back end (away from the sample) to make sure your sample is secure. *Note, the hex nut is Be and should be treated with care. Beryllium is highly toxic.*
- JEOL double tilt analytical holder: (This holder is shared between the two JEOL microscopes.) The cradle mechanism on this holder is extremely delicate. Be sure to situate the black plastic support piece under the cradle mechanism before loading the sample. Use a screw driver to loosen the clamping tabs on either side of the sample location and swing them out of the way. Insert the sample. Insert one or two spacer rings if necessary. Insert the Be piece. Swing the clamping tabs back in place. *Gently* tighten the screws.
- Gatan double-tilt/rotate holder. (Do not use this unless you have received instruction.)

The remaining holder listed below requires special instruction.

- Gatan double-tilt cooling holder (shared between the two JEOL microscopes)

REMOVING AND INSERTING THE ENTRY-PORT PLUG

When no sample is in the scope, a plug is normally left in the goniometer entry port to allow the diffusion pump to continuously pump on the vacuum buffer chamber, thus maintaining cleanliness.

To remove the plug, first verify that V1 (**VALVE** button) is closed. Then proceed as follows (See figure):

1. Set the switch to AIR. (It may already be in that position.)
2. Pull back on lever and pull plug out (~1 mm). Turn to the left until it stops. *DO NOT PULL OUT!*
3. Wait for the green light to go out. Then gently pull the plug out.

INSERTING THE SAMPLE HOLDER

Check sample and O-rings: Check to insure your specimen is mounted properly in the specimen holder using the optical microscope, also make sure the O-rings in the specimen holder are clean

Check stage coordinates: Before putting in specimen holder, make sure the X, Y, Z, and X-tilt coordinates displayed by FasTEM Client are zero. If they are not at zero, contact a staff member.

Objective aperture: Check that the motorized objective aperture is retracted.

Holder Insertion: To insert the holder, proceed as follows (See figure):

1. Align the guide pin and insert the plug as far as it can go.
2. You will hear two sounds indicating valves operating. Then set the switch to PUMP.
3. After a minute the green light will turn on (the red may light still be on). Carefully turn the holder CW a small amount; allow the vacuum to pull it in (about 0.5 cm).
4. While keeping a firm grip, turn it CW again so that the complete rotation is 90° and allow the vacuum to *slowly* pull it completely in. Do not let go of the holder before it is completely in, otherwise it may damage the X-Y movement mechanism.

If you are using one of the double-tilt holders, connect the Y-tilt cable to the connector below the goniometer.

Holder insertion will cause the column pressure (as read on the SIP meter) to increase. It should recover quickly and return to ~2×10^{-5} Pa or lower. If it does not recover, remove the holder and check the O-rings.

Holder Insertion

1. Align guide pin, push in.
2. Wait for valving. Then set switch to PUMP.
3. Wait for the green light. Then turn a few degrees clockwise and the rod will partially slide in.
4. Turn completely while keeping a firm grip. Allow the holder to slowly and completely slide in.

FINDING THE BEAM

Check again that the column pressure is less than 2×10^{-5} Pa, and open the isolation valve V1 (**VALVE** button). If you can't see any beam, the most likely reason is that your sample is in the way. Lower the magnification and try moving the sample with the track ball. DO NOT move apertures or gun tilt/shift or beam shift to find the beam!

If you can't see the beam, try the following (approximately in order):

- Check the **SPOT SIZE** is 1 and the PROBE CONTROL is in TEM mode.
- Decrease the magnification and turn the **BRIGHTNESS** control clockwise.
- Check that the objective and selected area apertures are out. (Do not adjust the CONDENSER aperture X-Y position.)
- If your sample is on a grid or if you have bulk sample with a hole, the beam may be blocked by a grid bar or by a thick part of the sample. Move the specimen a bit until you see the hole. You can monitor the sample X-Y coordinates on the FasTEM Client window.
- Go to **LOW MAG**. mode and spread the illumination by turning the **BRIGHTNESS** control clockwise.
- Move your sample to find an appropriate area.
- If you still can't find the beam, close V1, press the stage "**N**" button to the left of the column and take out the sample holder. Put the entry-port plug in, open V1 and find the beam. If you find it, then you must close V1 and remove the plug and reinsert the sample holder.
- If you still can't see the beam, get assistance.

BEAM ALIGNMENT (TEM MODE)

Note: During any period of work interruption, close the isolation valve by pressing the **VALVE** button.

There is more than one way to align the instrument. The following is one method. If you are familiar with different method that works for you, use it. In general, the system should require very little, if any, alignment.

Set mode to TEM 1 – 3: For initial alignment, set the **SPOT SIZE** to 1 and the **α-SELECTOR** to 3. Start with the 150-micron aperture and ensure that it is roughly centered.

Perform the following 5 checks.

1. Check gun tilt (Should require little or no adjustment):

Choose a magnification of ~100K×. Find the beam crossover condition and then slightly strengthen the **BRIGHTNESS** control to partially fill the screen. Activate **ANODE WOBBLER**. Adjust the **GUN TILT** so that the illumination expands and contracts concentrically.

2. Align gun/condenser lens system (Should require little or no adjustment):

1. Change **SPOT SIZE** to 5. Obtain crossover condition and center with beam **SHIFT**.
2. Change **SPOT SIZE** to 1. Obtain crossover condition and center with **GUN SHIFT**.
3. Repeat 1 and 2 until beam movement is minimized.

3. Adjustment of condenser stigmators (Usually requires little or no adjustment):

Check the condenser stigmation and correct it if necessary. (Activate **COND STIG** and use the **DEF X** and **Y** controls.) Judge the presence of astigmatism by noting the roundness of the illuminated area as you turn **BRIGHTNESS** clockwise. Or, observe the shape of the beam crossover at very high (~500K ×) magnification.

4. Refine the centering of the condenser aperture:

1. Ensure that the beam crossover is centered. If not, center it with the beam **SHIFT**.
2. Turn **BRIGHTNESS** clockwise. If illumination shifts, center it with condenser aperture X-Y knobs.
3. Repeat 1 and 2 until aperture is centered.

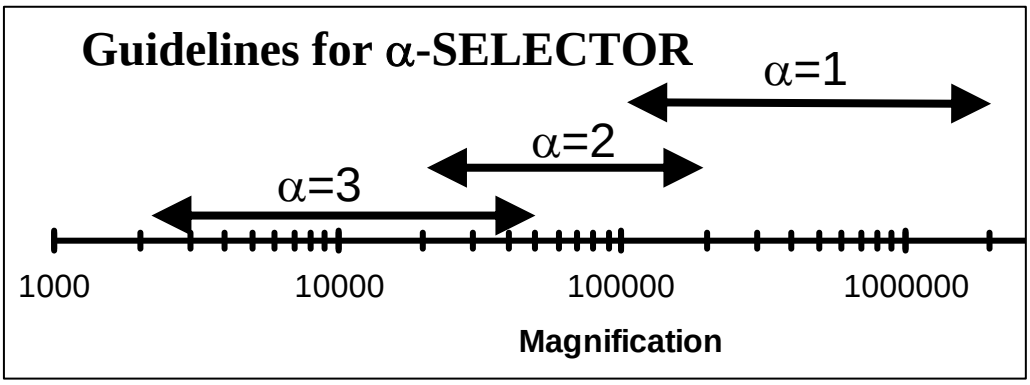
5. Check tilt balance (pivot point) (Right hand drawer):

1. First make sure that DV (displayed on PAGE-1 of microscope monitor and also on the FasTEM Client) is 0.
2. Activate the **COND DEF ADJ TILT** button and set the **TILT X/Y** switch to X. Adjust **FREQ** and **AMP** to maximum if necessary.
3. Adjust **SHIFT-X** to make the two spots overlap. If necessary (not likely), adjust **DEF-X**.
4. Repeat this with Y direction.
5. Repeat for **α-SELECTOR** settings 2 and 1.

BRIGHT FIELD IMAGING

Find an area of interest and bring it into approximate focus by adjusting Z. (Keep DV ~ 0.)

Select an appropriate setting for the **α-SELECTOR**. See the diagram below. Guidelines are as follows: For magnifications ~ 50K× or less, use setting α = 3. Between 20K× and 200K×, α = 2 is appropriate. α = 1 can be used for 100K× and above.



When changing the **α-SELECTOR** adjust the condenser stigmators if necessary. Also, bring the illumination back to the center of the field of view by using the beam **SHIFT** if necessary.

Before recording an image, check the following three alignments: 1) beam tilt; 2) objective aperture centering; 3) objective stigmators:

1. Beam tilt

Refine the beam tilt by finding the current center (**OBJ WOBBLER**) or voltage center (**HT WOBBLER**). Alternatively, if you are familiar with coma-free alignment, use that method before recording high resolution images.

2. Insert and center an objective aperture

If an objective aperture is desired: go to diffraction mode, insert an aperture, and center it. Note that the motorized objective aperture is approximately in the true back focal plane; if this aperture is in focus, the back focal plane is in approximate focus. The lower objective aperture, if you use it, will be slightly out of focus unless you strengthen the diffraction lens. Then you will no longer be exactly focused on the true back focal plane. Also, note that the motorized objective aperture will not insert if the Z coordinate has too large a negative value or if the X- or Y-tilt values are too high.

3. Adjust objective stigmators

In image mode, select the desired magnification and make final adjustments of the focus using the **OBJ FOCUS** knob. (DV should remain close to 0.) Use the high-resolution camera (Gatan camera).

Adjust the objective stigmators using one of the following methods:

Method 1: Fresnel Fringe Method: (Perform at magnification of ~20K× or greater with Ultrascan camera.) While viewing a small hole in the sample, adjust the stigmators to ensure that the Fresnel fringe is of even thickness around the edge of the hole. If you do not have a hole available, a curved edge will usually suffice. Use the **OBJ FOCUS** to go through focus and ensure that the fringe disappears evenly and reverses contrast.

Method 2 (Preferred): Judge the phase contrast image of an amorphous sample. (Perform at magnification of ~200K× or greater with Ultrascan camera.) Adjust the stigmators and the objective focus to find the minimum contrast and ensure that the image at slight over and under focus shows the graininess of the amorphous material with no preferred texture or streaking. Use the FFT feature of the Gatan software to judge the stigmation.

Notes on high resolution imaging

- To improve lateral beam coherence, make sure the beam divergence is not too large. For high resolution imaging (above ~200kX), use an **α-SELECTOR** setting of 1.
- Carefully check the beam tilt (either voltage center or coma free method).
- Astigmatism correction: The setting of the stigmator is critical at high resolution.
- The sample should be tightly secured in the holder and should be stable under the beam. Also, any compromise in mechanical stability (e.g., a torn support film) could be detrimental.
- If sample appears to be unstable, it could be that the settling time parameter in the Gatan software needs adjustment. (See below.)
- The contamination rate must be acceptably low. Do not waste time trying to record images of a rapidly contaminating sample.
- Do not use a magnification higher than necessary. It is rarely necessary to go above 500 or 600 K×. Higher magnifications require longer exposures thereby increasing the chance that sample drift may ruin your exposure.

DIFFRACTION

Put the microscope in diffraction mode (press **DIFF** button). Select the camera length you wish to work with by using the **SELECTOR** switch.

Adjust intermediate lens stigmators as follows: With the intermediate and objective apertures out, C3 (**BRIGHTNESS**) strongly excited, and **α-SELECTOR** at 3, adjust the **DIFF FOCUS** to observe the caustic figure. If the caustic is too intense to observe comfortably, adjust the **SPOT SIZE** to reduce the intensity. Center it with the **PROJ SHIFT** (right-hand drawer) if necessary. Adjust the intermediate stigmators to correct the astigmatism. (Select **INT STIG** in right-hand drawer and use the **DEF** knobs in the drawer.)

If you later decide to work with a different camera length, repeat the above correction for the new camera length.

In image mode, find an area of interest.

Insert an intermediate (selected area) aperture and center it with X-Y knobs on the aperture assembly.

(If you want to minimize the diffraction error caused by the objective lens spherical aberration, use the **SAM** mode and adjust the **DIFF FOCUS** knob to focus the intermediate aperture.)

Make final adjustments to the image focus using the **OBJ FOCUS** knob.

Spread the illumination. (Turn **BRIGHTNESS** CW.) (Note, in mode TEM 1 – 3, cylindrical illumination occurs with C3 at ~90% of maximum: reference voltage at about 7.2.)

Go to diffraction mode and use the camera length for which you corrected the intermediate stigmation. Focus the pattern with the **DIFF FOCUS** knob.

If necessary, center the diffraction pattern with the **PROJ SHIFT**.

Before recording a pattern with the CCD cameras, be sure to first insert the beam stop to block the central beam. If you want to avoid the use of the beam stop, decrease the beam current by using a smaller spot size and/or a smaller condenser aperture.

BEAM ALIGNMENT IN NANOPROBE MODES (CBD, NBD, EDS)

Select **CBD** (or **EDS** or **NBD**) illumination mode. Adjust **α-SELECTOR** to the desired setting. Confirm that the tilt balance is properly adjusted. If not, correct it.

At a magnification of ~100K×, obtain crossover condition and center it with the beam **SHIFT**. Check the condenser aperture centering and correct the condenser astigmatism.

Activate the **HT WOBBLER** and use the **BRIGHT TILT** to ensure that the illumination expands and contracts concentrically.

Convergent Beam Diffraction (CBD)

By simply eliminating the use of the diffraction aperture and using focused illumination at the sample (crossover at sample plane), you will obtain a convergent beam electron diffraction pattern when you select **DIFF** mode.

You can obtain smaller probe sizes and/or have more control over the convergence angle by selecting **CBD** mode or **NBD** mode. The convergence angle is then controlled by adjusting the excitation of the condenser mini-lens (**α -SELECTOR**).

If you are concerned about precise focusing of the CBD pattern, the following are three ways to locate the back focal plane of the objective lens.

1. If the sample is crystalline and exhibits a Kikuchi pattern, adjust **DIFF FOCUS** to make the Kikuchi pattern sharp.
2. Alternatively, the back focal plane will be in focus if, in TEM mode with **SPOT SIZE** 1 and **α - SELECTOR** set to 3 and **BRIGHTNESS** control ~90% of maximum (Reference voltage ~7.2), the caustic spot is minimized.
3. The motorized objective aperture is approximately in the back focal plane. If it is in focus then the diffraction focus should be approximately correct.

RECORDING DATA (USE OF THE GATAN SOFTWARE)

It is impractical to present full details of the Gatan software (DigitalMicrograph) in this manual. The software itself has a useful “help” feature. A few important and often-used features will be summarized.

The camera pull-down menu allows selection of one of the two cameras: the Ultrascan (high-resolution) camera (retractable) and the Enfina (EELS detector). For images and diffraction patterns make sure the Ultrascan camera is selected and inserted.

Note that there should be two floating windows associated with the camera: the “View” window and the “Acquire” window. The View window offers the options of search and focus. The Acquire window offers only the record function. Note that the tool box icon offers the user the option to change camera parameters such as

binning and active CCD area. This is important because any user may change parameters and the changes apply to all users. Be sure to adjust the parameters to your needs.

Adjusting the binning and/or active area can greatly increase the readout speed thereby allowing something like "live" viewing of the image. This may be useful when searching for an area of interest.

An important feature of the camera is a parameter called "settling time". The beam shuttering occurs above the sample, and with many samples it is necessary to allow the sample to settle for a tenth of a second or longer before recording an image. Adjust the settling time as necessary.

Recording diffraction patterns:

When recording diffraction patterns, please be sure to use the beam stop to block the central beam. Its intensity is typically so high that prolonged exposure of the camera phosphor to this beam can ultimately result in premature damage to this phosphor. For diffraction from single crystals in high symmetry orientations, be aware that the Bragg reflections may also be very strong. Recording such reflections may require smaller spot size (strongly excited C1) and/or smaller condenser aperture. Also, be aware that the phosphor exhibits an "after glow"; any strong signal falling on the phosphor may result in artifacts in subsequent recordings.

STEM OPERATION

1. The scope should be reasonably well aligned in TEM mode – especially the gun tilt. Take note of the gun deflector settings.
2. Decide whether you want the DC offset on or off. It can normally remain on but should be off for the best performance when doing high resolution STEM.
3. Click the STEM button on the FasTEM Client. This puts the microscope in the "STEM Alignment" (AL) mode. (The illumination system is in CBD mode.) Check that the gun deflectors remain the same as in TEM mode.
4. Little if any alignment is required. If the DC offset is on and you wish to check the illumination system alignment before going to STEM mode, you can proceed as follows, although it is rarely necessary:
    a) On the left-hand control panel, maximize the 1st condenser excitation (**SPOT SIZE**).
    b) Set the magnification to ~50K×.
    c) Maximize the 2nd condenser excitation (**BRIGHTNESS**).
    d) Set **OBJ FOCUS** step to 3 and weaken the objective lens so that the caustic spot is visible on the screen. Minimize the caustic spot. (DV will be about -16 to -18.)
    e) Center the caustic spot with the **BRIGHT TILT** controls
    f) Adjust the objective excitation (**OBJ FOCUS**) so that DV = 0, and focus the illumination with the 2nd condenser (**BRIGHTNESS**).
    g) Center the focused probe with the left and right **SHIFT** controls.
    h) Repeat steps c through f until there is little or no change.

    As an alternative to the above, simply center the caustic with the **BRIGHT TILT** controls and, without changing the objective lens, center the illumination with the **SHIFT** controls.
5. Go to STEM mode by clicking the SM button on the FasTEM Client and make sure the magnification is 100K× or greater. Make sure the spot size indicated on the microscope monitor is S (not M or L).
6. Remove the condenser lens aperture
7. Through the FasTEM Client, degauss the illumination system, and choose the probe size you want.
8. Again, through the FasTEM Client, degauss the projector lenses and choose a temporary camera length of ~ 20 to 25 cm.
9. Press the **SPOT** button on the ASID unit to make the probe stationary. Magnification will read ××××. (You may have to press the **SPOT** button two times.)
10. It is best to find an amorphous area on the sample. You can see a shadow image of your sample if you make coarse adjustments to **OBJ FOCUS**, either stronger or weaker, and the shadow image will invert as you pass through the focused probe condition.
11. Adjust the **OBJ FOCUS** to observe the ronchigram. If your sample was in focus in TEM mode with DV ~ 0, it should require little adjustment of **OBJ FOCUS** to see the ronchigram.

12. To provide a point of reference, use the **PROJ SHIFT** to center the ronchigram on the black dot on the small viewing screen.
13. Adjust the **COND STIG** to obtain a round ronchigram
14. Insert a condenser aperture and center it. The 40-μm or 20-μm aperture is recommended.
15. Obtaining a ADF STEM image:
    - Choose a camera length from the FasTEM Client. (Degauss projectors first.)
    - Observe the CBD pattern on the screen and ensure that it is approximately centered.
    - Choose a detector: (Fischione, or Gatan) and insert it. Raise the screen and be sure that the Ultrascan camera is retracted.
    - Press the **PIC** button on the ASID unit to start the beam scan
    - Align the beam with the detector using **PROJ SHIFT**. (In ADF STEM, the image signal will be minimized for thin samples.)
    - Images may be recorded using Digital Micrograph or ES Vision.
16. Obtaining a BF STEM image:
    - Insert the Gatan BF detector and raise the screen.
    - Choose a camera length from the FasTEM Client. (Degauss projectors first.)
    - Press the PIC button on the ASID unit to start the beam scan.
    - Align the beam with the detector using **PROJ SHIFT**.
    - Images may be recorded using Digital Micrograph or ES Vision.
17. When finished with STEM mode, return the microscope to TEM mode. Click on “TEM” on the FasTEM Client.

RECORDING STEM IMAGES

STEM images can be digitally recorded using the Gatan software, DigitalMicrograph, or the Emispec (FEI) software, ES Vision.

In DigitalMicrograph, make sure that the floating window called “Digiscan” is open. Clicking on the toolbox allows you to select the number of pixels, the pixel dwell time and the bit depth for the three modes of Search, Preview, and Record.

In ES Vision, several of the available workspaces are configured with a STEM image window. Number of pixels and dwell time can be selected using the tools on the right hand panel of the ES Vision GUI.

X-RAY ENERGY DISPERSIVE SPECTROSCOPY (EDS)

EDS data is recorded using the ES Vision software on the Emispec computer. The detector insertion and retraction is controlled by the RTEM software application on the same computer.

The preferred method of collecting EDS data is in STEM mode, although the ES Vision software contains a workspace that allows EDS data collection without going to STEM mode. One, of course, sacrifices spatial accuracy with this option.

ES Vision has a workspace especially configured for STEM and EDS, and it has pre-programmed options for doing EDS line profiles, maps, or a set of discrete points. Any of these three options can also be implemented with drift correction. There is also a time series option.

The pulse processor time constant can be adjusted through the graphical menu on the right side of the ES Vision GUI.

EDS data are saved in ES Vision format, but can be exported in two-column text format that can be imported into any graphing software (such as Excel).

ELECTRON ENERGY LOSS SPECTROSCOPY (EELS)

EELS is typically done in STEM mode. This allows excellent spatial resolution for chemical analysis. However, if spatial resolution is not important in your EELS measurements, you can use TEM mode with the microscope in diffraction mode.

The system consists of a bending magnet, focus-correcting quadrupoles and aberration-correcting sextupoles used to produce the spectrum. In addition there are four quadrupole lenses to magnify the spectrum and a detector to collect the data. The detector consists of a YAG scintillator optically coupled to a rectangular CCD chip (1340 pixels by 100 pixels) that is 26.8 mm long in the dispersive direction and 2 mm wide in the nondispersive direction. It has a very limited dynamic range and is easily saturated by the main electron beam.

Avoid signals that are strong enough to saturate the detector with short (millisecond) integration times. The spectrum as displayed in DigitalMicrograph will appear yellow if it close to saturating and red if it is saturating.

The spectrometer (not including the detector) is controlled by the Filter Control software. However, the user rarely needs to interface with the Filter Control. Rather, DigitalMicrograph features two floating windows -- EELS Acquire and EELS Tuning -- to interface with Filter Control as well as the detector.

Note that the camera view or acquire windows allow you to see the full two-dimensional read-out of the detector. The EELS floating window will automatically integrate the signal in the nondispersive direction.

Locating the beam (Can be done in STEM mode or TEM imaging or diffraction mode)

- Go to the camera menu, go to the sub-menu Camera and select “Enfina”.
- Locate a hole in the sample.
- Retract the Ultrascan camera. If you are in STEM mode you must use the DF STEM detector.
- Illuminate the center of the TEM screen.
- Lift the TEM screen.
- Verify that you can detect the zero-loss beam
- Finally click on Align ZLP and the beam should be aligned.

Watch the DigitalMicrograph progress bar to see when it has finished.

You may wish to check the alignment. Choose an integration time of a few milliseconds then click on View and the spectrum should be displayed. Pressing the space bar stops the view.

Preparing a gain reference (Can be done in STEM mode or TEM imaging or diffraction mode. Just be sure to fill the 5-mm aperture with the beam.)

At this point you should take a gain reference image.

Gain reference procedure

- Go to the AutoPEELS view floating window, set the dispersion to 0.0eV/ch. This is a special setting of the quadrupole lenses that projects a distorted image of the spectrometer entrance aperture onto the detector. Set the start energy to 0 eV and set the aperture size to 1mm.
- Set up a view from the Camera View floating window with a 0.1-sec exposure. Choose Search mode and click on the tool box and select “unprocessed” for the data correction. Binning should be 1 and the full CCD should be selected. Click “Start View” on the Camera View window.
- If the image does not show a centered beam then adjust the “`Start Energy`” until the beam is centered as shown below.

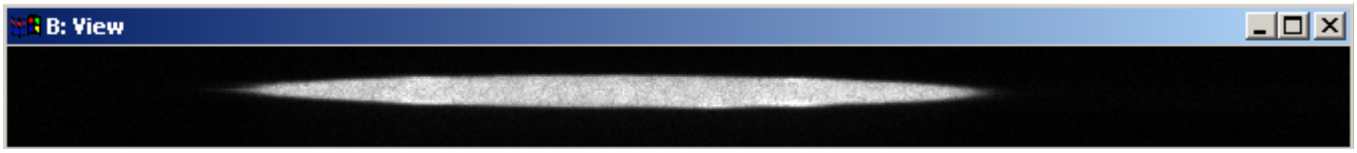


Figure: 1mm aperture view at 0.0 dispersion

– From the AutoPEELS view floating window increase the entrance aperture until the whole of the detector is illuminated (5-mm aperture), adjust the beam on the TEM to attain a count level of about 20,000, and leave the system viewing for 5 minutes. Increase the exposure time to ~1 sec or longer to get a more efficient duty cycle.

The reason the beam is left over the whole detector for five minutes is to "anneal" the YAG scintillator. This removes any "history" and reduces the memory effect of future bright beams.

– Next select "`prepare gain reference`" from the `camera` menu.
– Make sure the target intensity and frames to average are set to 30,000 and 10 respectively

Figure: 5mm aperture view at 0.0 dispersion

The choice of the parameters in the "Prepare Gain Reference" dialog has an effect on all of the images that you acquire later. All images that you subsequently acquire are divided by the gain reference image. If the noise in this gain reference is greater than the noise in a subsequent image then the noise in the gain reference will dominate in the resulting image.As a simple rule the total number of counts in the gain reference should be more than in the final data. If you intend to take 100 frames averaged later then make sure the gain reference has 100 frames averaged.
The target intensity is set at a level well below the saturation level so that the detector is unlikely to saturate in a single frame. On the ENFINA saturation varies from CCD to CCD but is normally between 40,000 and 65,000 counts.

Focusing the system
Choose the dispersion you wish to work with and the entrance aperture you wish to work with.
Ideally the system should be focused using the same camera length you will use to collect data and with the entrance aperture filled with intensity. Note that it is often not possible to focus under these conditions because the beam will saturate the detector. In this case, adjust conditions such that the detector is not saturated and focus the spectrum.
To focus the system click on the focus button in the Auto-tune floating window. Normally only FX and FY need adjustment. For a more complete focusing, press Alt-Focus and you can select the sextupoles (SX and SY) as well as the AC compensation coils.
Manual focusing is described below.
Acquiring a spectrum
To record a spectrum, locate an area of interest on the sample. To obtain good EELS data it is important that the area you look at be thin and clean.
If you are in normal TEM mode (not STEM mode), adjust the illumination conditions as desired (usually one uses a focused probe) and put the microscope in diffraction mode. Use a camera length between 2 and 20 cm. (Camera lengths less than 8 cm must be uploaded using the Free Lens Control feature of the FasTEM Client.)
In STEM mode, you should likewise choose a camera length between 2 and 20 cm.
Make sure the center spot (disc) is in the center of the screen. If you are looking at the low loss region, choose a 1 msec integration time. For higher energy losses adjust the integration time as appropriate (~0.1 sec). Then click the view button to start the system.
Now adjust the **PROJ SHIFT** setting on the TEM by a small amount to optimize the amount of signal entering the detector.
On most microscopes changing the magnification or mode causes small changes in the focus and apparent energy of the beam; repeating the alignment of the zero loss and focusing procedure should improve the spectrum.

Assess the spectrum acquired in view mode and optimize the integration time to record a high quality spectrum. The "Acquire" option allows you to specify the number of integrations you desire. Also, note that you can specify and save acquisition parameters in the EELS Acquire menu to be recalled later when you need them.

Manually adjusting the EELS system

1 Aligning the system by hand

To adjust any of the spectrometer controls by hand.

- Start a view in the normal way.
- Go to the program FilterControl.
- Double click on an element/lens.
- Moving the mouse right or left changes the lens value. Pressing the + or – key increases or decreases the strength of the mouse.
- Mouse click exits the mouse control mode leaving the lens at the new value.
- ESCAPE exits without changing the lens value.

2 Finding the beam

Adjust the value of the lens marked ADJUST from –2000 to +2000 the beam should be in this range, typically it is within a few hundred volts of where you start.

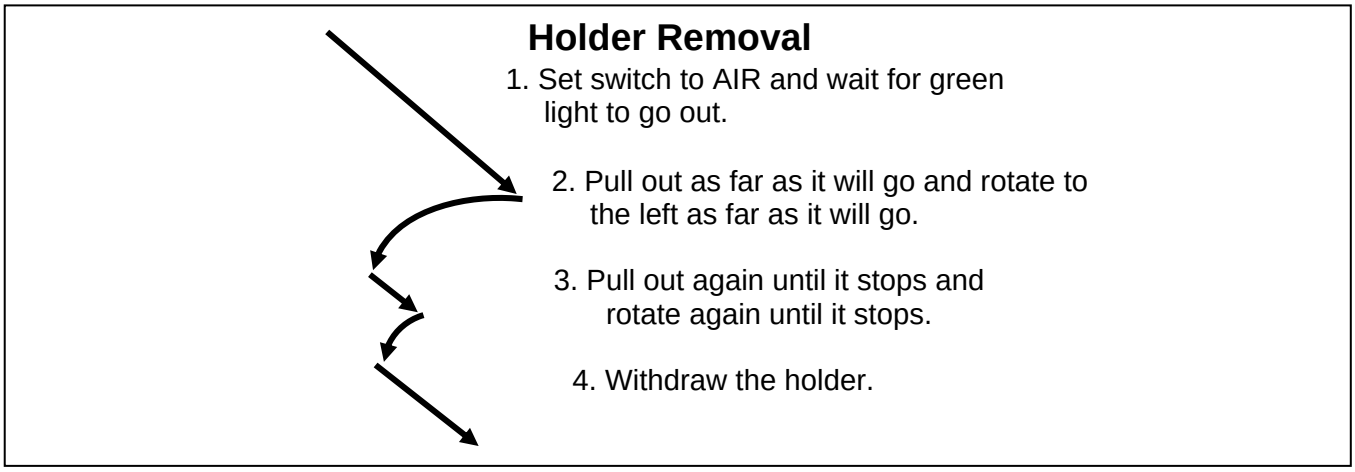
3 Focusing lenses

- Select a 1mm entrance aperture
- Make the spectrum as sharp as possible by adjusting FX and FY.
- Select 3mm entrance aperture
- Focus with SX and SY
- Go to the aperture you intend to work with
- Refocus FX and FY.

END OF SESSION

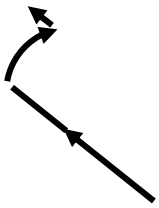
At the end of your session:

- Set magnification to 40K× or higher, and spread the illumination to fill the screen.
- Press the **VALVE** button to close the isolation valve.
- Ensure that the EDS detector is out.
- Make sure the motorized objective aperture, lower objective aperture, and diffraction aperture are retracted.
- Press **N** button to left of viewing chamber to return stage to origin. Check the X,Y, Z, X-TILT, and Y-TILT coordinates on the FasTEM Client to be sure that they have been reset to 0.
- Remove holder. (See figure.)
  1. Pull out as far as it will go and rotate to the left as far as it will go.
  2. Pull out again (~0.5 cm) as far as it will go and rotate to the left again.
  3. Set the switch to AIR and wait for the green light to go out.
  4. Carefully withdraw the holder using a gloved hand to guide the rod.



– If the next user has not arrived, or if you are the last user, insert blanking plug as follows: Align airlock pin, push in, turn to the right, and push in again (~1 mm). (See figure.) Make sure that the spring loaded tab goes into the slot. (It is not necessary to set the switch to PUMP. Pumping will commence as long as the tab is in the slot.)
– Remove sample from holder.

Plug Insertion



1. Align airlock pin, push in
2. Wait for valves to operate, then set switch to PUMP
3. When green lamp comes on, turn to the right and allow the plug to slide in (~1 mm).

This must be done by the last user of the day

– Plug ACD heater in Dewar and make sure it is connected well.
– Press **ACD HEAT** on the lower left panel.