Notes

1. The JEOL 2010F STEM/TEM is an analytical electron microscope with a field-emission gun normally working at 200 kV. The field emission gun is sensitive and may easily be damaged by incautious use. The gun is also expensive to replace (about $100,000.00 not including labor) and the replacement procedure often requires several weeks of downtime. Therefore, users should obey the following instructions when operating the microscope and play strict attention to warnings concerning the electron gun system.

2. The microscope is operated via the JEOL FasTEM system. This runs on two computers that are located on the right hand side of the scope. One computer runs server software that communicates directly to the microscope (FasTEM Server) and the other computer runs client software that serves as the interface to the user. The client machine also runs the data acquisition software from Gatan (Digital Micrograph) and the Gatan Imaging Filter (or GIF) control software (Filter Control).

There are three main programs that run on the FasTEM client machine:

i. FasTEM client, which communicates with the server and displays the operating conditions for the user.

ii. Filter Control, the software that communicates with the GIF and allows direct control of the GIF.

iii. Digital Micrograph, the Gatan software that controls the microscopes cameras, scanning system and has a more user friendly interface to the GIF.

These applications need to be stated in the same order as they are listed above because they communicated with one another and each expects certain software to be activated so that it can operate correctly.

So, if any of the client software hangs or the client computer itself locks up, an application crashes, or something odd else happens, that appears to be software related, please try to note down as much information as you can about what you were doing at that time, so you can report the events to EMAL staff. Pay particular attention to the various operating conditions, e.g. the magnification, the mode you were in, perhaps the mode you were switching to etc.,

If any of the client software does crash, or hang, or behave oddly you should try the following steps (in order of severity):

• First try to close and reopen the offending application. Since the program probably works in conjunction with another application it may be necessary to resart that application too. You may have to use ‘Task Manager’ – accessible by pressing control+alt+delete – to close a program that is not responding.
• Second: close all the programs and go to the start menu and choose ‘log out’. This will reset the computer without powering it down.

• Third (last resort): close all the programs and restart the computer.

3. Because we need the computers to run the microscope, storing data on the data acquisition or server computers will have a negative effect on the operation of those computers. All data should be stored on the EMALServer, this is a large filer server running Mac OSX Server which is located in room 413. The server has over a terabyte of disk space and data from the 2010F should be stored on the volume “User Data 1” in the folder “_JEOL 2010F AEM”. You may create yourself a folder within “_JEOL 2010F AEM”, your folder should be named with your uniquname or username. We do not guarantee the security of your data on the server and strongly encourage you to back up your data to TWO locations that are separate from one another. This server is accessible from anywhere in the world via sftp. Any data saved on the instrument computers may be deleted by EMAL staff to maintain the full operability of the instrument.

4. Please pay a great deal of attention when you *change a specimen*. If the specimen loading procedure is not followed correctly the microscope will generate an error condition that causes the whole microscope to close itself down. The emission is turned off, the high voltage is turned off and the entire system will shutdown. Such an uncontrolled shutdown can cause damage to the gun and is therefore is completely undesirable. It will also take the best part of a whole day to restart the instrument, thus seriously inconveniencing other users.

5. Any irregularities and problems should be reported to EMAL staff:
   
i. **John Mansfield** jfmjfm@umich.edu
   734-936-3352 (work)
   734-994-3096 (home)
   734-834-3913 (cell)

ii. **Kai Su** kaisun@umich.edu
   734-936-3353 (work)

iii. **Haiping Sun** haipings@umich.edu
   734-936-3338 (work)

Please do not try to fix the microscope by yourself.

6. **Do not tilt both X-tilt and Y-tilt to greater than 20 degree.**
Summary of the basic alignment

1. **Check**: Vacuum: Gun < 0.1e⁻⁶ Pa, Column < 3e⁻⁵ Pa, V1 closed.

2. **HT ready**, meter = 75µA@160kV or 95µA@200kV. (If 160kV: increase voltage in FasTEM client: Target setting = 200kV, Step Voltage = 1kV, Step Time = 30sec. If the HT meter reads 97µA@200kV, restart Filter Control software)

   FEG ready and Emission = 140-170µA

3. **LOGIN**: Fill anti-contamination device with liquid Ni.

4. **Load Specimen**: Zero tilts and z height with ‘N’, and zero x,y position in ‘Goniometer’ tab in FasTEM client. Check before removing holder. Check V1 is closed and EDAX detector is OUT.

5. **Open V1**: Find beam.

6. **Set OBJ current to 5.75** (‘lens’ tab in FastTEM client). Check DV=0. **Focus specimen with z height.** (If DV ≠ 0 while OBJ current ~ 5.75, the DV memory needs to be reset. You should maintain OBJ=5.76, not DV=0).

7. **Center Condenser Aperture**: TEM 1-3, Mag. 100kx, OL=+0DV.

8. **Condenser Astigmatism**: shape of beam.

9. **Voltage Center**: Bright tilt, HT wobbler.

10. **Beam tilt purity** / Image wobbler: Mag. 100kx, OL = +0 DV, Cond Def Adj:Tilt, tilt X-Y

11. **Anode Wobbler**: Anode Wobb. Gun tilt, gun shift.

12. **Condenser alignment**: spot 1=gun shift, spot 5= beam shift

13. **Intermediate Lens Astigmatism**: apertures out, spread beam, Diff mode, Cam.Length=50cm, diff focus for caustic image. IL stigmators

14. **Correct Objective astigmatism**
1 Beginning the Session

1.1 Checking the Status of the Microscope

1.1.1 Make sure the Emission is on.

The "HT Ready" and "FEG Ready" lights (left panel, see Figure 1) should both be green. The "EMSN" button on the same panel should be bright. Do not touch the EMSN or HT buttons!

![Figure 1. Left hand panel – don’t touch.](image)

![Figure 2. Valve 1 (V1), Isolation valve.](image)

1.1.2 Check for correct Operating Voltage: HT meter = ~75µA@160 kV or 95µA@200 kV.

These are the normal values that indicate the Gatan Imaging Filter (GIF) is on. If the HT meter reads ~77µA@160kV and 97µA@200kV, then the GIF is off. If this is the case you need to restart the program ‘Filter Control’ after ensuring the GATAN electronics boxes all have power. The GIF is left on all of the time, but occasionally after an electrical storm or other power failure it may be off. The GIF actually drops the voltage to 197kV and the microscope is aligned with the GIF on. If the GIF is off and you try to use the microscope without restarting ‘Filter Control’, the microscope will be at a different voltage than it is aligned for and you will notice that the alignment will not be optimal.

1.1.3 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 telltale (see Figure 2 above) will be out. This valve isolates the gun vacuum from the column vacuum and is closed whenever the microscope is not in use and during specimen exchange.

1.1.4 Check the EDAX detector is out.

The dewar will be retracted and you will be able to see some of the metal bellows. In ‘RTEM control’ the out button is highlighted.

1.1.5 Normally a specimen holder is inserted in the column of the microscope.

![Figure 3. EDAX detector (a) retracted (b) inserted.](image)

1.1.6 Check the vacuum (see Figure 4.).
All gauges except the column vacuum should be pegged at zero, which is similar to how they look if the power is off. If the pump has power, the green power light will be on, regardless of the position of any power on/off switch (which you should not touch!).

**GUN:** 60 l/s Gun SIP-pegged at 0 (read from upper meter, red scale 75l/sec).

**COLUMN:** 150 l/s Column SIP - < 3 x 10⁻⁵ Pa (blue scale). With V1 closed the pressure is usually ~0.5 x 10⁻⁵ Pa, with liquid nitrogen cooling. With V1 open the pressure is typically ~1.0 x 10⁻⁵ Pa, with liquid nitrogen cooling.

**EMITTER:** 15 l/s (actually 3x 5L/s pumps located at emitter) SIP - (< 1µA).

**GUN V1 Valve:** 20 l/s SIP (located at gun isolation valve) - < 0.1 x 10⁻⁶ Pa.

### 1.2 Filling the Cold Finger Dewar.

1.2.1 Make sure the cover is on the glass viewing screen.

1.2.2 Fill anti-contamination device (ACD). See Figure 5. Fill the ACD dewar with LN2 using a funnel and beaker. (Refill in about 15-20 minutes, then every 5 hours or so) Column SIP will usually drop to ~ 0.5 x 10⁻⁵ Pa.

![Figure 5. Liquid nitrogen dewar on ACD.](image)

### 1.3 Open the FasTEM software if it has not been opened.

The FasTEM system consists of ‘FasTEM server’, ‘FasTEM client’, ‘Filter Control’, and ‘Digital Micrograph’. The FasTEM server is installed on the computer with the small monitor and usually is permanently active (Users should not close it). The other three programs are in a folder called ‘Start up the FasTEM’ which can be found on the client computer (with the large monitor). They should be started in order, i.e ‘1. FasTEM client’ (You need to login here using the usual common EMAL user and password (Figure 6.)), ‘2. Filter Control’ followed by ‘3. Digital Micrograph’.

### 1.4 Bringing up the HT (High Tension)

Check the HT meter (and the small monitor). If the meter reads 95µA, normally the microscope is already at 200 kV and you can skip this step. If the meter reads 97µA, the voltage is at 200kV but the GIF is off and you need to restart the software program ‘Filter Control’. If the meter reads 75µA, the voltage is at 160 kV, you need to bring it up to 200kV following the instructions below.

![Figure 6. FasTEM client login window.](image)
1.4.1 Go to the software program ‘FasTEM client’ and click on the top tab labeled ‘HT’See (Figure 7). The ‘Current Setting’ should agree with the voltage (160 kV) shown on the microscope screen and be consistent with the HT meter (75µA).

1.4.2 Set the ramp up conditions: Target voltage to 200kV; Set the step size to 1kV; Set the step time to 30 seconds; Total time should equal about 20 minutes; Start the ramp. Click on Up (Go).

1.4.3 Check the HT meter to see that the voltage is really increasing.

You will see the value on the HT meter change and you will also see the LED flicker when the voltage actually changes. Sometimes there is a communication problem and the voltage does not increase despite the readings on the computer and the microscope screen. You should stop the ramp, return the voltage to the beginning value, and then try to restart some of the software to establish communication, or ask for help.

Figure 7. FasTEM client HT control. Figure 8. Motorized Objective Aperture control.

1.5 Login to the EMAL 2010 Logbook.

At this point you should have established the condition of the microscope. If all is normal you may proceed, otherwise seek assistance. You need to enter your details and some details about the microscope in the logbook NOW. Don’t wait until the end and fill everything in – you will forget what the initial conditions were.

1.6 Specimen exchange (You may perform specimen exchange during the voltage ramps up).

Do NOT attempt to exchange a specimen before having been trained by our EMAL Staffs.

1.6.1 Before exchanging a specimen, you should check:

- **Check the motorized objective aperture** (Figure 8) is out (“0” position)-Do NOT try to use this unless you have been trained and authorized. It is very close to the specimen holder.

- **Normalize specimen position:** Make sure the Y-tilt cable is connected and press the “N” (button to the left of the column, the X-Y tilts and X, Y and Z should move to zero. Check to see that they do! Be sure to go back to MAG1 mode when you are doing so.

- **Make sure the column/gun "Valve" is closed (V1) and the EDX detector is out.**
1.6.2 Remove the specimen holder. **BE CAREFUL!** Study the diagram shown in the end of the manual before you start to change a specimen each time. It is also stuck to the microscope column.

You should have paid close attention to our instructions. If you have any doubts about the procedure you should check with us. A Quicktime Movie of the whole procedure is available on the EMAL Web Site (http://emalwww.engin.umich.edu/Quicktimes/).

(a) Set the switch on the goniometer to "Air" (see Figure 9.)

(b) Grasp the holder and pull it straight out until it stops.

(c) Rotate the holder counter-clockwise until it stops again (~70 deg). **When you are rotating the holder you should NOT be pulling it, and vice versa.** Once you start the rotation, the holder cannot be pulled back into the microscope by the vacuum. It is safe to release it and take a big breath and wipe your sweaty palms off before you continue.

(d) Now grasp the rod itself and with your forefinger and thumb, gently pull the holder straight out until it stops again. This is a very short pull, do NOT rotate.

(e) Gently rotate the holder counterclockwise until it stops again. This is a small amount of rotation (~20°). **You must NOT pull during this rotation** or you will skip past the micro-switch and wrench the holder out of the vacuum without venting it properly. This leaves the vacuum in an odd state and will crash the vacuum. To make sure you stay on the micro-switch while the holder is vented, push the holder in a little and keep a small amount of counterclockwise rotation pressure to keep the holder pin on the micro-switch.

(f) WAIT for the airlock to vent, indicated by the green LED on the pre-pump control (Figure 10) going out, and the LED for valve V16 (the one with an arrow and the legend N2 above) on the vacuum telltale will turn green. Carefully remove the holder the rest of the way by pulling it gently straight out of the microscope. Be careful not to crash the tip of the holder against anything.

1.6.3 Select a holder you wish to use and load your specimen into the holder.

**EMAL has 4 different holders for the JEOL 2010F.**

(i) **The JEOL double-tilt stage:** It is good for mounting odd shaped or easily broken specimens. This holder is a low background holder and is suitable for XEDS analysis.

(ii) **The Gatan #646 double-tilt holder:** This holder has the standard Gatan Hexring mounting system. The specimen cup of this holder is made from beryllium as is the hex-nut and anti-twist washer, which is therefore suitable for XEDS analysis. Great care should be exercised when handling the Be parts since beryllium oxide is very poisonous. It is also very brittle and will break if forced at all.

(iii) **The Gatan #636 double-tilt liquid nitrogen stage:** This holder can be cooled to near liquid nitrogen temperatures. This holder employs the standard Gatan Hexring mounting system. The specimen cup of this holder is made from beryllium as is the hex-nut and anti-twist washer, which is therefore also suitable for XEDS analysis.

(iv) **The Gatan #658 double-tilt hot stage:** This holder can be heated to ~1000°C. Above 500°C, however, it is necessary to use the supplied closed cycle water recirculator/chiller to prevent the holder from damage. The Hexring, anti-twist washer and specimen cup are fabricated from tungsten. Each holder has a separate box for its Hexrings and washers, please keep them separate.

When using the Gatan holders, the hexring should be placed in the holder with the larger face pointing up and should rotate smoothly in the threads. **DO NOT apply force to the hexring or you will jam it and/or break it.** Specimens should be mounted facing down in the holder - this is face up in the microscope and is optimum for EDS work.

1.6.4 Insert the specimen holder into the column. **BE CAREFUL!** Study the diagram again.

(a) Insert the end of the specimen rod into the airlock with the small pin on the rod aligned with the small slot in the goniometer.

(b) Push the rod in gently so that the pin pushes against the microswitch. You should hear the airlock start to vent again if you have contacted the switch properly. Be careful not to let the rod rotate past the
micro-switch, or you will crash the vacuum. By maintaining a slight counter-clockwise pressure on the switch you can safeguard against crashes.

(c) Hold the rod in place while switching the pre-pump switch (Figure 10) up to “PUMP”. You will hear several valves opening/closing and the rough pumping of the line begin.

(d) When the roughing pump begins to pump the airlock you should feel a small jolt of the specimen rod, it is then safe to release your hold on the rod. DO NOT ROTATE IT!

(e) Wait until the green LED on the pre-pump control (above the pre-pump switch, again see Figure 10) illuminates.

(f) Now rotate the rod about 20° clockwise and it will move into the column a short distance. Continue to rotate the rod and after approximately 70° you will feel the rod being drawn into the column, slowly guide it in until it stops. Do not let it go at this point as you could damage the microscope.

(g) The specimen is now inserted into the scope. Check the column vacuum before continuing.

2 Basic Alignment of the Microscope - The starting point for other alignments.

There is a FasTEM knob set with the column of buttons labeled ‘DETECTOR’.

<table>
<thead>
<tr>
<th>DETECTOR buttons</th>
<th>Functions</th>
</tr>
</thead>
<tbody>
<tr>
<td>EDS</td>
<td>Normal TEM mode</td>
</tr>
<tr>
<td>TV TOP</td>
<td>Currently no function</td>
</tr>
<tr>
<td>TV BOT</td>
<td>TV camera before the GIF. Choose Source ‘V1’ on the Samsung monitor</td>
</tr>
<tr>
<td>MSC</td>
<td>CCD camera before the GIF.</td>
</tr>
<tr>
<td>GIF/PEELS</td>
<td>Sets up lens conditions for the GIF.</td>
</tr>
<tr>
<td>STEM</td>
<td>Sets up lens conditions for STEM operation.</td>
</tr>
</tbody>
</table>

Figure 10. FasTEM knob set, detector buttons.

2.1 Find the Beam

• **Check the column vacuum:** make it sure that the column vacuum is better than $3 \times 10^{-5}$ Pa. It is usually better than $3 \times 10^{-3}$ Pa with no LN2 and $\leq 1 \times 10^{-5}$ Pa with the LN2 trap filled.

• **Open Isolation Valve (V1):** Open the column to the beam by depressing the "Valve" button. It should light up and the V1 LED on the vacuum telltale will be on. The valve will not open if the column SIP vacuum is $> 5 \times 10^{-5}$ Pa.

• **Find the beam:** If you can’t see the beam after opening V1, first check that the objective and diffraction apertures are out; then reduce the magnification in MAG1 mode or go to LOW MAG mode to find the hole in your specimen.

2.2 Alignment of the Illumination System

2.2.1 **Use a setting of "TEM 1-3" for the TEM alignment procedure:** The notation TEM 1-3 means "TEM Mode, Spot Size 1, alpha-Selector 3". Convergence angle is changed primarily by the CM lens current (alpha-Selector) or the condenser aperture. Changing in the Spot size from TEM 1-3
to TEM 5-3, increases the first condenser lens value but does not change the convergence angle significantly.

2.2.2 **Center condenser aperture:** First converge the beam and center it using beam shift. Then spread out the beam and watch for beam ‘swing’ from side to side as you go either side of brightness crossover (see Figure 11). Crossover is the point at which the beam size is the smallest on the screen. Check for beam ‘swing’ with the brightness spread out on the clockwise side of crossover and also the counter-clockwise side. If the beam swings, center the *spread out beam* using the appropriate condenser aperture knobs.

There are 3 adjustment knobs: a black knob at the front, and 2 silver knobs at the side (Figure 11). The black knob is always used. Which silver one you use depends on the position of the lever. There are two aperture strips, each with three apertures in. The lever selects the different aperture strips. If you move the wrong screw (and it seems like nothing is happening), you are misaligning the apertures you can’t see. With the lever pointed to the left (most common), use the front-most shorter silver knob. With the control pointed to the right use the hind-most longer silver knob.

![Condenser aperture alignment](image)

**Aperture sizes:**

**LEFT:** 100µm, 70µm, and 40µm

**RIGHT:** 2mm hole, 10µm and 30µm

![Condenser aperture alignment](image)

**Figure 12.** Condenser aperture alignment – (a, b, c) Beam ‘swing’. (d, e, f) Beam stays concentric around screen center ‘+’. (a,d) Brightness counter-clockwise, (b,e) converged, (c,f) brightness clockwise.

**Figure 13.** Condenser astigmatism. (a, b, c) Beam shape distortion. (d, e, f) Beam stays round. (a, d) brightness counter-clockwise, (b, e) converged, (c, f) brightness clockwise.
2.2.3 **Correct condenser astigmatism**: You should make the beam as round as possible when it is slightly defocused, or focus the beam and make sure that it expands and contracts symmetrically as you go slightly over and under focus. Depress the condenser stigmator button in the right drawer and use the deflectors to correct the shape of the beam.

2.2.4 **Anode Wobbler (Gun tilt)**: In TEM1-3 mode at a magnification of higher than 100kx. Converge the beam and center it using beam shift. Press the green "Anode Wobb" button (below Emission meter). Depress the "Gun" button in the right drawer and use the "X-Y Def" knobs in the right drawer to adjust the beam until it expands and contracts symmetrically about the optic axis. Turn off the anode wobbler after finishing.

2.2.5 **Align illumination down the optic axis of the condenser lens system**: By switching between "Spot Size 5" and "Spot Size 1" and centering the beam. Use the "Beam Shift" knobs to center the beam with "Spot Size 5" and the "Gun" button + "Shift" knobs in the right drawer for "Spot Size 1". Repeat until spot size 1 and 5 remain centered on the screen.

2.2.6 **Beam Tilt Purity (adjusts the current balance in the condenser deflect lenses)**: TEM1-3, Magnification: 300kx. Make sure the Obj. Lens is at DV=+0. Converge the beam and center it. Depress the "Tilt" button in right drawer (under Cond/Def/Adj). Wobble 2-cond beam deflect lens using the white toggle switch labeled "Tilt X-Y" on the lower right-hand drawer front. Set the switch to X (Y) and use the "ShiftX" and "DefX" ("ShiftY" and "DefY") knobs to make beam stationary. If you see two separate beams, make them into one beam first.

2.2.7 **Establish the voltage center**: In TEM1-3 mode at a magnification of higher than 100kx. Position the specimen so that a recognizable feature is on the reference dot in the center of the screen. Make sure it is in focus. Spread the illumination (clockwise with the brightness knob) to fill the screen and press "HT Wobbler" on the right panel. Press the "Bright Tilt" button on the left panel to activate the Cond-2 beam deflects. Adjust the X-Y bright-tilt knobs to obtain a stationary sample image. Turn off the "Bright Tilt" and HT wobbler buttons after adjustment.

2.2.8 **Find an area of interest on the specimen and center the illumination and spread it out to fill the screen**.

2.2.9 **Set the objective lens current to 5.75**: Use the "Objective Focus" knob. You can see the lens currents on the ‘Lens’ tab in the lower part of the FasTEM client. When the objective lens is at the correct current, DV (deviation voltage) visible on the ‘Status’ tab and also on the microscope screen, should be 0 at 100kx and will be an easy way to monitor changes in the Objective Lens current. Throughout your microscope session you should maintain DV = 0 and do all coarse focussing with the z-height adjustment.

*NOTE:* DV is a memory setting. If the microscope loses power it will lose the memory of where DV=0. If you set the Lens current to 5.75 but DV is a long way from zero (must be at 200kV with HT meter = 95), you should ignore DV and keep the Lens current at 5.76.

2.2.10 **Focus the specimen with the Z-height adjustment button**: Make sure the specimen is focused at a magnification of 100 kX (MAG1 mode).

2.2.11 **Adjust the Intermediate Lens stigmations**: Remove OL aperture and SAD apertures if inserted. Spread illumination to maximum in the clockwise direction (this sets the C2 lens = max =8.0 A). Go to DIFF mode. Set camera length, CL = 50cm. Adjust the DIFF FOCUS knob (IL1 lens) to see the caustic. Adjust the stigmators for the IL lens (right drawer, Stigmators, ‘INT’ – intermediate) to make the caustic image round.

2.2.12 **Align micro-area-illumination (if EDS to be performed)**: in the TEM mode, CBD switch on; select spot size 1-3; turn on Wobbler-HT and align it using Bright Tilt.
2.3 Gatan Imaging Filter (GIF) Alignment (If EFTEM to be performed)

2.3.1 **Pre-set**: Perform TEM routine alignment and center and focus the beam to about 1cm in diameter. Pull all detectors and pre-GIF camera out, lift the viewing screen, select TV monitor ‘V2’ with the GIF CCD camera selected.

2.3.2 **Set Filter parameters**: “Image” (*Spectroscopy for EELS mode, Dispersion to 0.0 eV/pixel*); slit width: 10.0eV or 20eV; TV camera: out (if the after-GIF TV camera used) or in (if the GIF CCD camera used); Aperture: ‘mask’; “Drift tube = 0”, Filter control in “TEM” mode.

2.3.3 **Centering the projector cross-over on the ‘mask’ using ‘Projector shift’ knobs.**

2.3.4 **Align ZLP** by clicking “Align ZLP” in the “Filter control” window.

2.3.5 **Click “Tune GIF” in the “Filter control” window**: The GIF system will be aligned automatically. If meet problem, ask John, Kai or Haiping for a help.

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**Figure 14. Gatan Image Filter (GIF) system (courtesy R.F. Egerton).**

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3. Working in the conventional TEM mode (CTEM)

In this mode, several techniques are available for different purposes. They are selected-area electron diffraction (SAED), microdiffraction (including Convergent Beam Diffraction (CBED) and nanodiffraction), diffraction contrast imaging (BF and DF), phase-contrast imaging (HREM), EDX spectroscopy, electron energy-loss spectroscopy (EELS both in imaging and diffraction modes) and energy-filtered electron microscopy (EFTEM). The following are details for each operation:

3.1 **Getting a diffraction pattern**

i. **SAED**: In image mode (MAG1), center an area of interest. Spread the beam clockwise to fill the screen. Insert a selective aperture and center it over your selected area. Go to DIFF mode, a pattern is obtained. You can focus the diffraction pattern using the DIFF FOCUS knob.

ii. **CBED**: In image mode (MAG1), center an area of interest. Converge the beam and center with beam shift. Go to DIFF mode. A crystalline sample may show disks if it is relatively thin. These should not be focused to points. If the sample is relatively thick and a single crystal where the beam is converged, you may also (or only) see Kikuchi lines.

iii. **Nanodiffraction**: In image mode (MAG1), press the Nano button and choose different spot size and focus the beam to the area interested. Press the DIFF button you would have a nanodiffraction pattern.

3.2 **Diffraction-contrast (BF and DF) and phase-contrast imaging (HREM)**
3.2.1 To get BF and DF images: In DIFF mode with a diffraction pattern, insert the objective aperture and center it to the transmitted spot for BF imaging or a diffraction spot for DF imaging. The smaller the aperture the more diffraction contrast you will get. Do not try to center the aperture if you cannot see it!

3.2.2 To get HREM images: For high resolution imaging where you want to see lattice planes from phase contrast, you need to use a large objective aperture or none at all. Usually you should align your specimen to make sure a low-zone axis paralleling to the electron beam by double titling. Aperture "1" is the largest and "4" is the smallest. Aperture #2 has a collection semi-angle of 14mrad.

3.2.3 Correct the objective lens astigmatism: There are several different ways to do so. Here only list one. Using the Digital Micrograph to view a live FFT of an amorphous area of your sample: The area chosen to do a live FFT must be fairly uniform and all amorphous. Adjust the objective stigmators to make the FFT round. You can select a small portion of your image using the dotted square selector tool with the option or alt key held down (Figure 15).

Figure 15. Objective astigmatism correction using FFT: Bad astigmatism showing a distorted FFT at (a) under focus, (b) at focus, (c) over focus. Good astigmatism showing round FFT at (d) under focus, (e) at focus, (f) over focus. (since you can see a slight shape difference between under and over focus FFT, there is actually still a little astigmatism).

3.3 Recording Images

The JEOL 2010 has several different camera systems and ways of viewing or recording images in the TEM mode. You can record images on film or you can capture images digitally. For TV viewing of the image and collecting unfiltered digital images there are two sets of TV and CCD cameras, a TV camera and a CCD camera before the GIF (Gatan Imaging Filter) and a second set of cameras (a TV and a CCD) after the GIF.

3.3.1 Recording Images on the upper camera system (before the GIF).

Press detector button ‘TV BOT’, Samsung: source ‘V1’.

This will raise the screen and insert the TV camera. The TV camera inserts after a delay. You can watch it underneath the column (Figure 16). The TV camera is centered on the screen center. You may need to increase the brightness to see the image.
When you select detector ‘EDS’ again, the TV camera will retract and the screen will lower. If you want to see the image on the screen temporarily without retracting the TV camera you can still use the screen button on the TEM right panel. Be sure to go back to detector ‘EDS’ if you are finished with the TV camera.

To view the image on the CCD camera, press detector button ‘MSC’: This will retract the TV camera (if inserted) and start the acquisition in Digital Micrograph in ‘view’ (turtle) mode. It will also raise the screen if it is not already raised. Press ‘EDS’ to return to normal TEM mode.

Modes:

Focus (rabbit): Binning 3, unprocessed, turbo. A fast preview used to position the sample. Pixels on the CCD are binned 3 x 3 for 1 pixel in the image to increase signal for fast acquisition. Not high quality, so usually unprocessed to keep it fast.

View (turtle): Binning 2, gain normalized. A better quality, slower preview which can be used to check the focus. Pixels on the CCD are binned 2 x 2 for 1 pixel in the image to increase signal for faster acquisition. Usually processed to remove artifacts. Should be a fair representation of the final image.

Record (camera): Binning 1, gain normalized. Highest quality image for final capture. One pixel on the CCD equals one pixel in the image. Image is processed to remove artifacts. Acquisition is usually a bit slower.

Gain Reference: Certain artifacts from the CCD detector are present in the unprocessed image. A background image captured with no sample in view is used to eliminate these artifacts from the processed image. If you see large rings in the image (Newton’s interference fringes from two closely parallel mating surfaces) or ‘chicken-wire’ like features (the fiber optic bundles) all over the image, you may need to re-capture the background image, or ‘Gain Reference’. However, first try to adjust the brightness in your image to see if the chicken-wire or rings disappear. In some cases if your current image is much brighter than the image used for the background, the processing cannot completely remove the artifacts.

To capture a gain reference, first remove your sample completely from the field of view. Any features visible will become part of the background image and will be processed back in to all subsequent captured images. The beam should be spread out to evenly illuminate the screen. Go into Camera/Setup and set the view (turtle) mode to unprocessed. Then choose ‘Prepare Gain Reference’ from the ‘Camera’ menu. Digital Micrograph will first adjust the exposure time between 0.1 to 10 seconds in order to get an image with >8000 counts. Then it will capture 5 bright (shutter open) and 5 dark images (shutter closed) to get the signal from the CCD when the beam is on and off. The averaged dark image will be subtracted and the averaged bright image will be divided through your image during processing. Go back into Camera/Setup and set view (turtle) mode to gain normalized. Capture some images without the sample in view to check that you get even gray illumination.
3.3.2 Viewing and recording images on the GIF CCD camera.
In the DM pull down window “Camera”, select the GIF CCD camera. Make sure beam is not blocked.
For TV mode: choose technique – TEM, “Search” mode, then “Active”
For CCD: choose “Focus” (fast = rabbit) or “Preview” (slower, better quality = turtle), then click “Active”.

Note: Diffraction patterns should be recorded on film unless you have blocked the transmitted spot and weaken the intense diffraction spots in the diffraction pattern or you will damage the digital cameras.

3.3.3 Recording Images on Film (not so often used)
Make sure Detector ‘EDS’ is chosen on the knob set. This will ensure all cameras are retracted out of the way of the film. In some cases the problem is not the camera blocking the film, but the DM digital exposure control keeps the shutter closed while the screen is up unless there is a digital acquisition in progress.
Select automatic or manual exposure time on Page 1 of the microscope screen.
The full screen or the small screen can be used to estimate the exposure time. Insert the small screen using the lever located on the right side of the viewing chamber. The automatic exposure is usually OK for images. You must use manual exposure for diffraction patterns.
Press "Photo" on the right panel to manually advance the next negative under the screen.
The "Photo" light will illuminate when a plate has advanced.
Press "Photo" to start the exposure.
You will hear the screen lift up and the LED next to the “Photo” button will light up during the exposure. The screen will lower and the film will drop into the receiving chamber after the exposure.
If you press the photo button again after exposure but before the plate moves away, it will stay beneath the screen ready for a double (or triple…) exposure.

3.4 X-ray energy dispersive spectroscopy (XEDS)
3.4.1 Pre-settings:
• Edax Genesis software: There is an EDX detector attached to this microscope. You can use it to determine what elements are in your sample. The Edax Genesis software will allow you to collect spectra and do semi-quantitative composition analysis from particular places on your sample. Double click it to open the software.
• Perform TEM routine alignment and Micro-area illumination modes alignment.
• Open the RTEM icon and insert the Detector by clicking In and retract by clicking OUT with EDS, button enabled and Focus the beam to whatever interested and then start to collect a spectrum.
• Choose a proper amp together with beam size to set the DT% below 30%.

3.4.2 Use the Edax Genesis program for spectrum collection:
• Choose the Spectrum mode in the EDAX Genesis software.
• Click the Clear and Clear All icons in the tool zone of the software to clear both the old spectrum and its index.
• In the preset column to define a collection time or set to None (no limit).
• Click the Collect icon to start a spectrum acquisition.
• Once a good spectrum has been obtained (good SNR), stop the acquisition by click one more time the Collect icon.
• Index the peaks by click the Peak ID icon to auto index or manually by putting the curse at the peak positions and selecting a suitable element that match the best of the peaks.
• Click the HPD icon to highlight the spectrum and check how good the curves fit for the peaks. If not re-calibration of the spectrum is needed.
• Click the Quant icon to get a semi-quantitative results (Method: ZAF; Sel: EDAX; Type: element or oxide; Stds: none).
• The spectrum can be saved as different types. *.spc, *.tif and *csv are recommended.

3.5 Electron energy-loss spectroscopy (EELS)

3.5.1 EELS in image mode (Diffraction-coupled):
  • Perform GIF routine alignment.
  • Make the beam illuminate on Vacuum and select a suitable magnification.
  • Press the PEELS/GIF button in the FasTEM knobs set.
  • “EELS” function in the AutoFilter selected (Turbo view a spectrum can be set in the “Filter” window”.
  • Viewing the “beam” on the Sumsung screen (V2) with the GIF MSC camera selected (Check “Insert the camera”) and inserting an “aperture” and center it to the “beam”.
  • Set suitable acquiring parameters and Align ZLP (Using Shift X & Y to set the spectra at a proper position and make the zero-loss-peak as narrow, high & symmetric as possible by Focus X (focus only) & Y(rotate the peak); then open “Collocate” using Focus X (sharpen the zero-loss peak) and Focus Y to adjust the spectrum and make ZLP sharpen and parallel to the slit edge; and SX (make the peak straight) or SY (make the peak like an aircraft propeller); Adjust AC CompA and B if beam split at the largest dispersion available and spectrum not stable, to narrow the peak etc.).
  • Focus the beam to the interested area and select a suitable magnification and insert a suitable Objective aperture (which determines the acceptance angle together with the entrance aperture).
  • Set an energy range (three methods) and acquiring time and then Start to acquire an EELS spectrum.

  Note: Check: “Camera” – “Remove Dark References” if the spectrum seems funny!

  Check the energy resolution and thickness! Collect ZLP and calibrate it; select “Compute Thickness” in “analyze” (0.2~ 0.3 mean free distance; or check the low–loss integrated intensity is less than 1/3 of the integrated zero-loss intensity).

3.5.2 EELS in diffraction mode (“Image-coupled”):
  • Press the PEELS/GIF button in the control penal.
  • Select Vacuum using the selective aperture (or focus the beam) and select a suitable Camera Length to obtain a SAED (or nanodiffraction) pattern.
  • Centering the direct spot of the pattern in the middle of the entrance aperture using projector shift on the Sumsung (TV2) screen with the “TV camera insert” selected and a “selective entrance aperture (SEA, 3mm, 2mm, or 0.6 mm)” inserted.
  • EELS function in the AutoFilter selected.
  • Select suitable acquire parameters and Align ZLP (see above).
• Select an interested area and obtain a SAED (or a nanodiffraction) pattern.
• Set an energy range (three methods) and Start to acquire EELS spectra (pay attention to “Accumulated Readouts” settings). “Turbo” viewing spectrum image; “Corrected” for ZLP, “Accumulated” for Core loss.

3.6 Energy-filtered transmission electron microscopy (EFTEM)

3.6.1 Energy-filtered image and diffraction using low-loss

• **Using Zero-loss electrons (EF-BF imaging and EF-SAED):** Typically performed with the 3.0 mm aperture selected and a 10 eV or less wide energy window centered on the zero-loss peak.

• **Using Plasmon-loss electrons:** To image the local variation of plasmon excitation, which can be used to characterize the particle and can also be employed for selective imaging when different phases or precipitates and the matrix show separated sharp plasmon losses.

• **Thickness map:** ZL image via unfiltered BF image \((\lambda/n) = \ln(I_e/I_o)\).

3.6.2 Elemental mapping: **Using High energy-loss electron**

* **Using the Gatan AutoFilter package:**

  a. Perform regular microscope;
  b. PEELS/GIF selected;
  c. GIF and EELS alignments;
  d. Choose EFTEM on the Autofilter with zero-loss selected to center the slit (viewing on the screen with TV camera inserted);
  e. Acquire Gain and reference (settings, frames) for the MSC camera and then acquire a BF image;
  f. Focus the filtered image at low energy loss (100eV or 200 eV) before collecting high energy filtered images
  g. Select “Alt” + Zero-loss, Plasmon or Custom to set up mapping parameters and start collection (Doing drift correction manually “control + drag the image”).

* **Using the (EFTEM-SI) package:**

  a. Perform regular microscope; PEELS/GIF selected;
  b. GIF and EELS alignments;
  c. Choose EFTEM on the Autofilter with zero-loss selected to center the slit (viewing on the screen with TV camera inserted);
  d. Choose setup on the EFTEM-SI menu and input filter parameters;
  e. Choose Acquire on the EFTEM-SI menu (several different options);
  f. Artifact correction: i) Removing X-rays; ii) Spatial drift correction.

4. Working in the STEM mode

Figure 17 shows the electron-optical settings and how atomic resolution EEL spectra can be acquired.
4.1 Probe alignment using “Ronchigram”

1) Go to STEM mode directly by pressing “STEM” button twice; Set magnification higher than 1M in “Pic” mode and then switch to “SPOT” mode (“xxxx” mode). Make sure the spot is in small “S” (nor M or L) if high resolution imaging to be performed.

2) Setting up “probe size” (0.2 ~0.5 nm) and “camera length” (depending on detector to be used);

3) Find an amorphous area and focus the image to see “Ronchigram” (shadow image, or nanodiffraction pattern) using the ‘PROJECTOR SHIFT’ to center it in the middle of the screen;

4) Correcting the Condenser (objector) Stigmatism: COND on + GUN DEF to make the Ronchigram a circle at all focus settings;

5) Illumination beam alignment: Locate the optic axis (‘coma-free axis’); press Condenser Wobble+Condenser Shift: ‘Bright tilt + Beam Shift’ to make the pattern oscillate in and out on the ‘coma-free axis’;

6) HT tension correction: HT wobble + BRIGHT TILT make the feature oscillate in and out on axis.

7) Insert a suitable Condenser aperture and center the aperture to the Ronchigram.

BF field imaging:

• Insert the Gatan detector (BF).
• Choose TEI detector on ASID.
• Go to PIC mode and adjust brightness and contrast: Using TEI detector controls below the STEM monitor.
• Use Digiscan in GATAN Digital Micrograph to capture STEM images.

HAADF (Z-contrast) imaging:

• Insert an Objective aperture (condenser aperture in TEM mode); Choose a suitable ‘Objective aperture’ and center it to the ‘coma-free axis’.
• There are two annular detectors that can be used for STEM imaging through the Gatan DigiScan or EDAX systems: Joel annular dark-field detector or Gatan (BF and DF). Center the probe in the middle of either of the two detectors and HAADF, ADF or BF images can be recorded using either the DigiScan or the Edax Genesis software.

4.2 EELS in the STEM mode
- Performing normal TEM and GIF/PEELS alignments;
- Go to STEM mode and Optimize the probe;
- Center the probe in the middle of an aperture (at spectroscopy mode, dispersion: 0.0eV/pixel);
- Obtaining a DF reference image using the Gatan dark-field detector;
- Optimizing the ZLP spectrum:
  1. Select “PEELS” in ‘AutoFilter’;
  2. Setting up a suitable acquiring time, Dark count & gain selected etc.; put the probe in a hole (in DigiScan STEM choose ‘control beam’);
  3. At Spectroscopy Mode, set dispersion, and quantify EELS spectrum;
  4. Check the energy resolution and thickness! Collect ZLP and calibrate it; select “Compute Thickness” in “analyze” (0.2~ 0.3 mean free distance; or check The low–loss integrated intensity is less than 1/3 of the integrated zero-loss intensity);

4.3 EDS in the STEM mode

4.3.1 Edax Genesis: There is an EDX detector attached to this microscope. You can use it to determine what elements are in your sample. The Edax Genesis software installed in the nearby computer will allow you to collect spectra and do semi-quantitative composition analysis from particular places on your sample. You can also do elemental mapping/linescan to show the distribution of particular elements. Double click to open the software.

4.3.2 Pre-Settings:
- Performing normal TEM and GIF/PEELS alignments;
- Go to STEM mode and Optimizing the probe (using a probe size larger than 0.5 nm);
- Obtaining a DF or BF reference image;
- Insert the EDS detector;
- Choose a proper amp together with beam size to set the DT% below 30%.

4.3.3 Use the Edax Genesis program for collecting spectra: This can be done by leaving the microscope in full frame, select area or spot modes.
- Select a collection mode in the Scan window (Full, sel. Area or spot) to acquire a spectrum from the whole viewed or selected area or a specific site.
- Choose the Spectrum mode in the EDAX Genesis software.
- Click the Clear and Clear All icons in the tool zone of the software to clear both the old spectrum and its index.
- In the preset column to define a collection time or set to None (no limit).
- Click the Collect icon to start a spectrum acquisition.
- Once a good spectrum has been obtained (good SNR), stop the acquisition by click one more time the Collect icon.
- Index the peaks by click the Peak ID icon to auto index or manually by putting the curse at the peak positions and selecting a suitable element that match the best of the peaks.
Click the **HPD** icon to highlight the spectrum and check how good the curves fit for the peaks. If not re-calibration of the spectrum is needed.

Click the **Quant** icon to get a semi-quantitative results (*Method: ZAF; Sel: EDAX; Type: element or oxide; Stds: none*).

The spectrum can be saved as different types. *.spc, *.tif and *.csv are recommended.

### 4.3.4 Elemental mapping (2D elemental distribution):

- Align the microscope and switch to “**External XY**” mode by click it in the Scan window.
- Click and Switch to the **Map/Line** mode in the EDAX Genesis.
- Set resolution (Res.) and strips by right click the mouse button in the area right to the **Collect image** icon in the tool zone.
- Click the **Collect image** icon to collect an SE image.
- In the spectrum window below the image window, click the far top-left clock icon or the **Spectrum collection** icon in the tool zone to collect a spectrum.
- Index the spectrum. Index only those peaks for which elemental mapping will be acquired (the indexed elements will be **Region-of-interest (ROI)**).
- Click the **Map** icon in the tool zone. Check **live** and **drift** (for drift correction).
- Define resolution, dwell time and frames (better set more like 1024) etc. by right click the mouse and select.
- Click the **Collect maps** icon and give a name of the collection and click save and Ok to start a collection.
- Once the collection has been finished, the data will be saved in the folder automatically.

### 4.3.5 Elemental Linescan (1D elemental distribution):

- Align the microscope and switch to “**External XY**” mode by click it in the Scan window.
- Click and Switch to the **Map/Line** mode in the EDAX Genesis.
- Set resolution (Res.) and strips by right click the mouse button in the area right to the **Collect image** icon in the tool zone.
- Click the **Collect image** icon to collect an SE image.
- In the spectrum window below the image window, click the far top-left clock icon or the **Spectrum collection** icon in the tool zone to collect a spectrum.
- Index the spectrum. Index only those peaks for which elemental mapping will be acquired (the indexed elements will be **Region-of-interest (ROI)**).
- Click the **line** icon in the tool zone. Check **drift** (for drift correction).
- Choose the line tool (under the maps icon) and draw a line in the interested area of the image.
- Define point, dwell time etc. by right click the mouse and select.
- Click the **Collect line** icon and give a name of the collection and click save and Ok to start a collection.
- Once the collection has been finished, the data will be saved in the folder automatically.
*Ending*: Retract the EDS detector.

### 4.5 Nanodiffraction
- Optimizing the probe;
- Align crystal in the TEM mode or using Ronchigram in the STEM mode;
- Set a suitable camera length;
- At “Spot” mode using the pre-GIF CCD camera to record the pattern.

### 4.6 To exit STEM: Retract the JEOL ADF detector if used. Press EDS (or other) button on FasTEM controller.

### 5 Finishing the Session
- Return magnification to a relatively low setting (~ 100kX), spread the illumination to fill the screen and remove any objective or SAD apertures.
- Close the gun isolation valve V1.
- Check that the EDAX detector is OUT.
- Neutralize the stage position and tilts: Reset X-Y tilts and X-Y and Z translates to the 0 positions by pressing the green "N" button to the left of the column. FasTEM server will pop up a window when you neutralize the stage. In order to regain control of the stage you need to click on the ‘close’ button.
- Remove the specimen rod.
- Remove your sample from the specimen rod.
- Insert one of holders back into the microscope: Typically this will be the holder you were just using. If you were using the hot stage or the cold stage, you should store that holder on the Gatan pumping station and choose a double-tilt hold for insertion into the column.
- Exchange film if used: Reset number of unexposed plates in the FasTEM client. The vacuum in the camera chamber should reach ~30 uA level fairly quickly.
- Log out: Complete your entries in the log book and note any problems.
- **LAST USER ONLY**: Lower the high tension to 160 kV: To lower the High Voltage, go to the ‘HT’ tab in the FasTEM client. Set the following ramp down conditions:
  - Target voltage to 160kV;
  - Set the step size to 1kV;
  - Set the step time to 3 seconds.
  - Start the ramp. Click on DOWN (Check the HT meter to see that the voltage is really decreasing).
- **LAST USER ONLY:** Boil off Liquid Nitrogen: Insert the ACD heater into the LN dewar and connect the plug to the ACD. Depress the red "ACD" button located in the lower left panel on the microscope console. The light will turn bright and the heater will boil the LN out of the dewar. You cannot exchange the specimen or film once the ACD heater is activated, so it should be the very last thing that you do. If you change the film first, make sure that the camera reaches ~30 uA level before you run the ACD.

- Note that the camera is not also pumped while the specimen is being pre-pumped so the camera vacuum will increase during that time as well. The camera leak rate is approximately 6uA per hour (on a Log scale 30uA ~ 10^{-5} torr (diff pump) and 250uA ~ 760 torr).

**FINISH:**
- Close V1. Check that **EDAX detector is OUT**.
- Zero sample x,y,z position and tilts with ‘N’ and FasTEM client. Remove sample and reinsert holder.
- **You MUST change film** if used more than 5 plates.
- Make sure specimen and camera chamber are pumped down to 30 µA. (Gauge on front)
- **LOG OUT.** Leave microscope in MAG1, not LOW MAG so that Objective lens stays warm.
- **LAST USER:** reduce HT to 160kV. (FasTEM client: Target setting=160kV, Step Voltage=1kV, Step Time= 3sec)
- **LAST USER:** boil off the liquid nitrogen using ACD heater.

**Appendix: Removing and Inserting the Specimen Holder**

* Please perform accordingly while removing and reinserting the holder!!

When you insert and remove the specimen holder, the pin on the rod touches a microswitch 😄 (like a button) that tells the microscope vacuum system to do something. What the microscope does will depend on the position of the ‘pump/air’ switch. Keep a counter-clockwise pressure on the holder while you are on the microswitch 😄 to ensure the pin on the sample holder keeps good contact. Otherwise you will crash the vacuum and shut down the microscope. Be especially careful for highlighted steps.

OUT          IN
1. Insert the holder straight in and push it against the microswitch ☻. Keep a counter-clockwise pressure to maintain good contact. When you hear some valves opening to vent the holder again, change the switch to ‘PUMP’. Keep holding the holder until you feel the vacuum grab it. You can then release it, BUT DO NOT rotate it or ☹.

2. WAIT for the green light to come on before rotating clockwise, otherwise ☹.

3. Allow the specimen holder to go in to the stop.

4. Rotate clockwise until you feel a stop. Do NOT pull or push.

5. Allow the holder to go in slowly. You need to resist a little. Do NOT resist a little.

6. Check the column vacuum before opening.

5. Set switch to ‘AIR’

2. Rotate counter-clockwise until you feel a stop. Do NOT pull!

3. Pull straight out until you feel a stop (a small pull only. Be careful!!).

4. Rotate counter-clockwise until you feel a stop. Do NOT pull! If you pull here, you will be ☹.

6. WAIT until the green LED goes out to indicate the specimen chamber is vented. Then pull out the holder. If you remove it without venting correctly, ☹.

1. Pull straight out until you feel a stop. Do NOT release!

2. Rotate counter-clockwise until you feel a stop. Do NOT pull!