JEM 3010 Manual (2011 Edition)

Basic Alignment Instructions

Check the vacuum (power supply closet) and write on the log book**:**

- Gun pressure SIP2 < $2x10^{-5}$ Pa (usually 0.8 - $1.0x10^{-5}$ Pa)
- Column pressure SIP1 < $2x10^{-5}$ Pa (usually 1.5 - $2.5x10^{-5}$Pa)

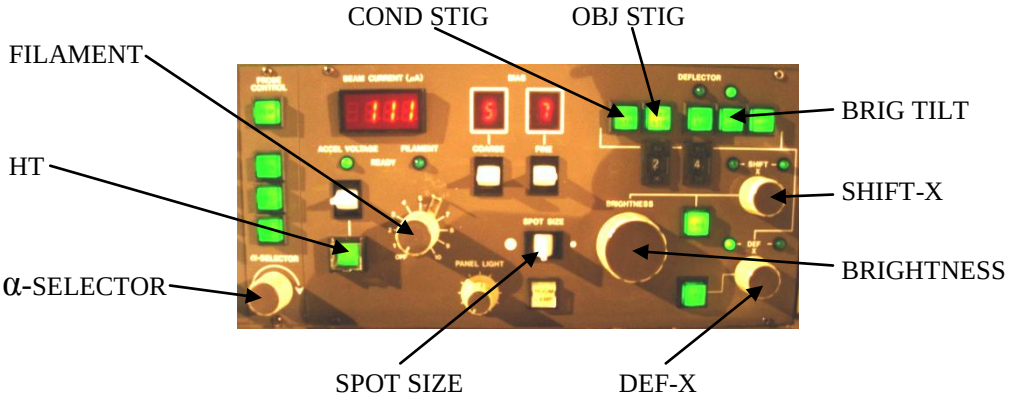
If the vacuum is not good, please contact someone from the staff.

If you are not sure about anything, PLEASE READ THIS MANUAL FIRST.

FIRST STEP: Complete the small Dewar (ACD) with liquid nitrogen. The microscope viewing port must be covered before pouring the LN2. The thick viewing port will crack under thermal shock. Wait for a few minutes and complete the Dewar after it stops boiling.

Turn on the TV/CCD cameras if necessary:

- TV Camera (TV Control Unit, Gatan 622SC, red button **POWER**).
- Video Processor Unit (Gatan Model 750, black button **POWER** at the back). Turn off **ADAPT** (red light off) and select two (**2**) frames using **AVERAGE** switch.
- The CCD camera (Gatan MSC794) should be always ON. If not, turn on right now (black button **I/O**). Check if the **PELTIER COOLER** is set to **COOL**. It takes one hour to stabilize the temperature.



Turning on the High Tension (HT)

At the microscope, **BEAM CURRENT** (red indicator on left control panel) must read: **"000"**. If the red indicator is off, please contact the staff. If the microscope is already set to 300kV and the **BEAM CURRENT** reads **~110μA**, skip this section.

Verify that the acceleration voltage has been set to **200 kV** (**ACC VOLT** on the TEM computer screen). If not, type the following instruction in the TEM computer keyboard: **htset 200** ↵

The green lamp over **ACCEL VOLTAGE** (left control panel) should be on.

1) Press **HT** button (left control panel). The current will start to increase and the **BEAM CURRENT** (red indicator) will show **~73μA** at 200kV.

2) It is time to increase from 200kV to 300kV. To increase voltage, type the following Instructions in the TEM computer:
load ht ↵
run ↵
>start ht ?(kV):
200 ↵
>stop ht ?(kV) v:
300 ↵
>ht step ?(kV/10):
10 ↵
>time limit (Minutes):
20 ↵ (after weekends or long holidays, use 40 minutes)

The voltage will gradually increase from 200 to 300kV. The TEM computer should lock-up until the end of the process. Wait until the microscope reaches 300kV. At 300 kV, the **BEAM CURRENT** reads: **~110μA**. If it is too high, contact for the staff.

Remember to fill the log book located on the table:

User Name: ???????????
Sample Holder: ?????????
Sample name, type material, etc.

While you are waiting the HT, start loading your sample in the sample holder (SH).

Sample Holder (SH)

Important: Always use glove when you are manipulating the SH or your sample. The gloves are not for your protection. You are the main source of contaminations.

Load your sample in to the right SH (ST1, ST2, DT and DTB). Check the TEM computer if the sample positions, **Specimen X**, **Y**, **Z**, **Tilt X**, and **Tilt Y,** are at **zero**. Before inserting the SH into the TEM, confirm the SH to the TEM computer by typing:

>**selwt 1** ↵ (selects **DT**, two tilting angles appear on the screen)

or

>**selwt 0** ↵ (selects **ST**, one tilting angle appears on the screen)

Important: After loading your sample, always check that the SH´s o-rings are completely free of dust or lint under the optical microscope. Use tweezers to remove the contamination.

To insert or remove the SH from the microscope, follow carefully the instructions on the last two pages of this manual. One copy of it is always on the TEM console. While you are loading, remember to keep one eye open on the vacuum meter.

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Remember: after the green light is on at the goniometer, ALWAYS wait for at least another extra 10 minutes before finish inserting the SH.

After the SH is inserted, press **LOW MAG** (right control panel) and **CRS** (left of the column). Type in to the TEM computer:

>**init** ↵

The TEM will calibrate the SH position by locating its extremes for X and Y and finding the center (0, 0). During the process you will hear the gears working on the goniometer. If you don´t hear this noise and the X and Y position in the TEM computer are moving slowly, please call the staff. You probably skip pressing **LOW MAG**. After finishing the process press **MAG 1** (right control panel).

(Check the vacuum)

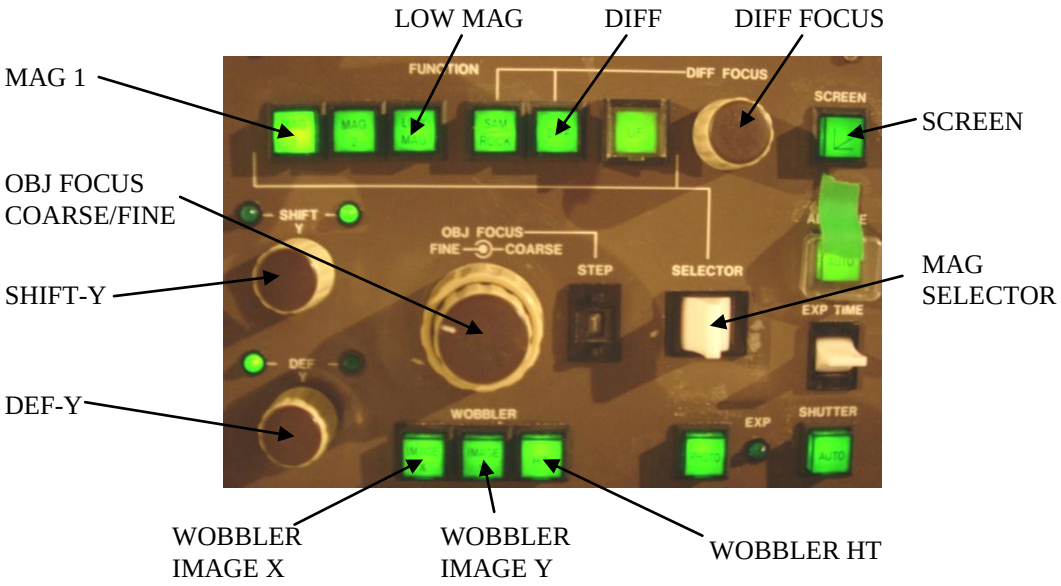
Starting the Electron Beam

Check that the green lamp below **FILAMENT** is ON (filament enabled). Set the **FILAMENT** knob to the preset value (around **6**). The stopper prevents you from going beyond the proper setting. Do not readjust this stopper.

Heat the filament using the **Filament Heating Ramp Control** (device box over left TEM control panel). The red lamp below **FILAMENT OFF** must be ON. If the red lamp is flashing, wait until it stops.

Push down the **FILAMENT ON/OFF** key (right hand side on the control unit). A yellow light will turn on, and a green lamp (below **FILAMENT ON**) start flashing. This process should take 16 minutes. Only the green light (**FILAMENT ON**) at the left end will be ON at the end of the process. The **BEAM CURRENT** indicator must be around **~120μA**. The electron beam is ON, to check remove the TEM window port cover and look at the phosphor screen.

Check that the **TEM** button under **PROBE CONTROL** is on (left panel). Set **SPOT SIZE** to **1**, and **ALPHA SELECTOR** to **3** (left panel). This displays as **1-3** on the upper right corner of TEM computer screen.



MICROSCOPE ALIGMENT

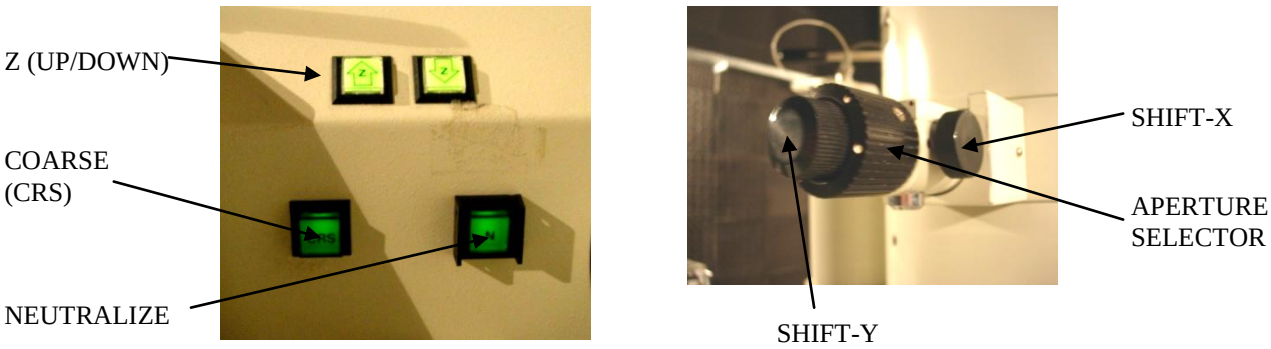
1) Finding the Electron Beam

If the beam is not visible on the phosphor screen, set to a lower magnification using the **MAG SELECTOR** switch (right panel) and move the sample around using the trackball. If the beam is not on the screen, press the **LOW MAG** button. Adjust the illumination using **BRIGHTNESS**. After finding the beam, press **MAG 1**. Set magnification back to **50K** to start aligning the TEM.

2) Centering the Condenser Aperture

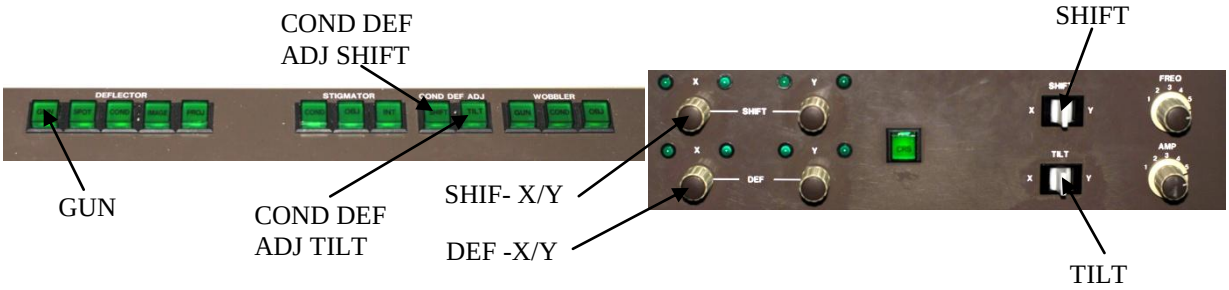
The second biggest aperture is the standard. If you need to use a different one, you must always return to the standard at the end of your session and leave it aligned.

Remember to always correct for the hysteresis of the condenser lens (**BRIGHTNESS** knob, counterclockwise-crossover-clockwise). Focus the beam using the **BRIGHTNESS** knob to obtain the crossover and bring it to the center of the phosphor screen using the **BRIGHTNESS SHIFT** knobs **X** and **Y**. Defocus clockwise the beam to have approximately the same diameter of the screen. If the beam is not centered, use the mechanical knobs of the aperture holder to center it.
Important, remember to check the condenser aperture alignment often. It is a mechanical device and it will misalign more often than you imagine.



3) Sample Height (Eucentric Center)

Adjust the objective lens focus (**OBJ FOCUS**) knob to the finest step (**1**) using the small switch next to it. Using coarse (outer) and fine (inner) **OBJ FOCUS** knobs, set to **DV = +-0** on the TEM computer. Press **MAG 1** to reset **OBJ FOCUS** to **0 nm**. Spread the beam and find a good area for observation in your sample. Focus your sample using **Z** control buttons (left of the TEM column). At the focus the contrast should be minimal. Another alternative way is to use **WOBBLER IMAGE X** or **Y** (right panel). The image will start to oscillate. You need to stop it from moving by adjusting **Z** at the right position.



4) Optimizing the Gun Tilt

Focus the beam with **BRIGHTNESS** knob, and center it on the screen using **BRIGHTNESS SHIFT** knobs **X** and **Y**. Press the **GUN** button (under the TEM keyboard). Adjust **GUN DEF X** and **Y** knobs (under the **GUN** button) to max out the current at the phosphor screen (**CURR DENS** reading at TEM computer).

Alternative way: de-saturate the filament using the **FILAMENT** knob. Adjust **GUN DEF X** and **Y** knobs (under **GUN** button) to obtain a bright spot with a symmetrical shape around it on the phosphor screen.

5) Aligning the Optical Axis (illumination)

Center the condenser aperture. Focus the beam with **BRIGHTNESS** knob. Change the **SPOT SIZE** from **1** to **5** (white switch on left panel). Bring the beam close to the center with the **BRIGHTNESS SHIFT** knobs and refocus the beam with **BRIGHTNESS** knob (disable **CRS BRIGHTNESS**). Re-center the beam.

Change **SPOT SIZE** back to **1**, and refocus the beam with **BRIGHTNESS** knob. This time press **GUN** button (under the TEM keyboard) and re-center with **GUN SHIFT X** and **Y** knobs (under **GUN** button).

Repeat this step (5) until you don´t see much change.

6) Condenser Astigmatism

Center the condenser aperture. Focus the beam with **BRIGHTNESS** knob. Slowly de-saturate the filament by turning the **FILAMENT** knob counterclockwise (less than one division) until you see some detail of the filament on the phosphor screen. Press **COND STIG** button (left panel) and adjust the image sharpness using **DEF** knobs (left and right panels**).** Slowly, saturate back the filament by turning the **FILAMENT** knob clockwise. Go to a higher magnification if this makes easier to you to adjust the **COND STIG**. Remember to return the TEM to the original mag. Make the beam's image on the screen as sharp as possible. Make sure it expands and contracts symmetrically as you focus and defocus the beam with the **BRIGHTNESS** knob.

7) Beam Tilt Purity

Check if **DV = -+0** on the TEM screen. Check the eucentric center and re-adjust it if necessary (step 3). Focus the beam on the phosphor screen using **BRIGHTNESS** knob. Press **TILT** button (under the TEM keyboard) and set the white switch marked **TILT** to **X** position (under **TILT** button). Stop the movement of the beam from oscillating using **SHIFT X** and **DEF X** knobs (both under **GUN** button). Move the TILT switch to **Y** position and now repeat with **SHIFT Y** and **DEF Y** knobs. If the beam moves out the screen, center it using **BRIGHTNESS SHIFT** (left/right panels).

8) Voltage Center

Defocus your beam (**BRIGHTNESS**) until you have a uniform illumination over the screen and look for a good area on your sample to get an image. Particle-like objects are the best. If you have adjusted the eucentric center correctly, the image should be very close to the right focus, but check it again anyway.

If you prefer, you can use the TV camera to adjustment the voltage center. But remember to never image the diffraction pattern or the focused beam directly on the camera.

To use the CCD/TV cameras adjust the "BRIGHTNESS" knob until the illumination larger than the phosphor screen. Also lower the screen before you change the TEM magnification or its mode.

Press **WOBBLER HT** (right panel). The image will start running on the phosphor screen. Press **BRIT TILT** button and using **DEF X** and **Y** (left and right panels). Use these controls to stop the movement from running. If properly adjusted, the image

should pulsate symmetrically. Press **WOBBLER HT** button again to turn it off. Adjust the voltage center at a magnification close to the one you plan to take your images.

9) Repeat steps **6**-**9** until you don't need more adjustments. Do it at **250k.**

10) Finish the Alignment

Do center the condenser aperture to finish the alignment.

Now the TEM is aligned and ready to start taking images. Good luck.

Taking Images on the TEM-3010

To take an image of your sample, adjust the image focus using the **OBJ FOCUS** knob (right panel). Correct the objective astigmatism pressing **OBJ STIG** (left panel) and using the **DEF X/Y** knobs (left/right panels). Adjust the focus and the astigmatism, one at a time until you get the sharpest image. The astigmatism will cause the image to smear in one particular direction when you focus/defocus the image on the screen. Start the adjustment at a low magnification and then move to a higher one. You can use the little phosphor screen to do the rough adjustments, and then move to the TV/CCD camera to do the fine adjustments. To use the TV/CCD camera, the illumination is spread until it covers an area slightly larger than the whole phosphor screen. To focus the beam directly on the detector may cause permanent damage to the detector´s scintillator. Press **SCREEN** (right panel) to access the TV/CCD. The little screen can be positioned by using the metal lever located at the right side of the TEM viewing port.

Hint: To adjust the focus or the astigmatism, you can count the clicks of the knob. Start turning the knob in one particular direction until the image start to losing sharpness. Turn the knob in the opposite direction until the image start again to lose sharpness. Count the number of clicks of the knob. Go back half of the number of click to get the best image.

Hint: One alternative to do the fine adjustment of the astigmatism and the focus is to use the Fourier Transform of the image obtained with the ccd camera. Once you have done the rough adjustment of the image, start acquiring continuously the image with the **Gatan DigitalMicrograph** on the computer using **Camera View** >> **Start View**. Select **Process** >> **Live** >> **FFT**. Adjust the focus and the astigmatism to obtain a FFT image (power spectrum) similar to (e).

An amorphous material will image as below. If the astigmatism is properly corrected, it will image as (d-f). The astigmatism creates the asymmetry of the FFT. Remember that this method works better if you are already corrected most of the astigmatism and the focus. On the image, at condition (e) you will be close to the minimum contrast. Outside this condition, the granularity of the image will increase. On edges

you will see a light fringe if you are under-focus. At over-focus the contrast will reverse and you will see a dark fringe.

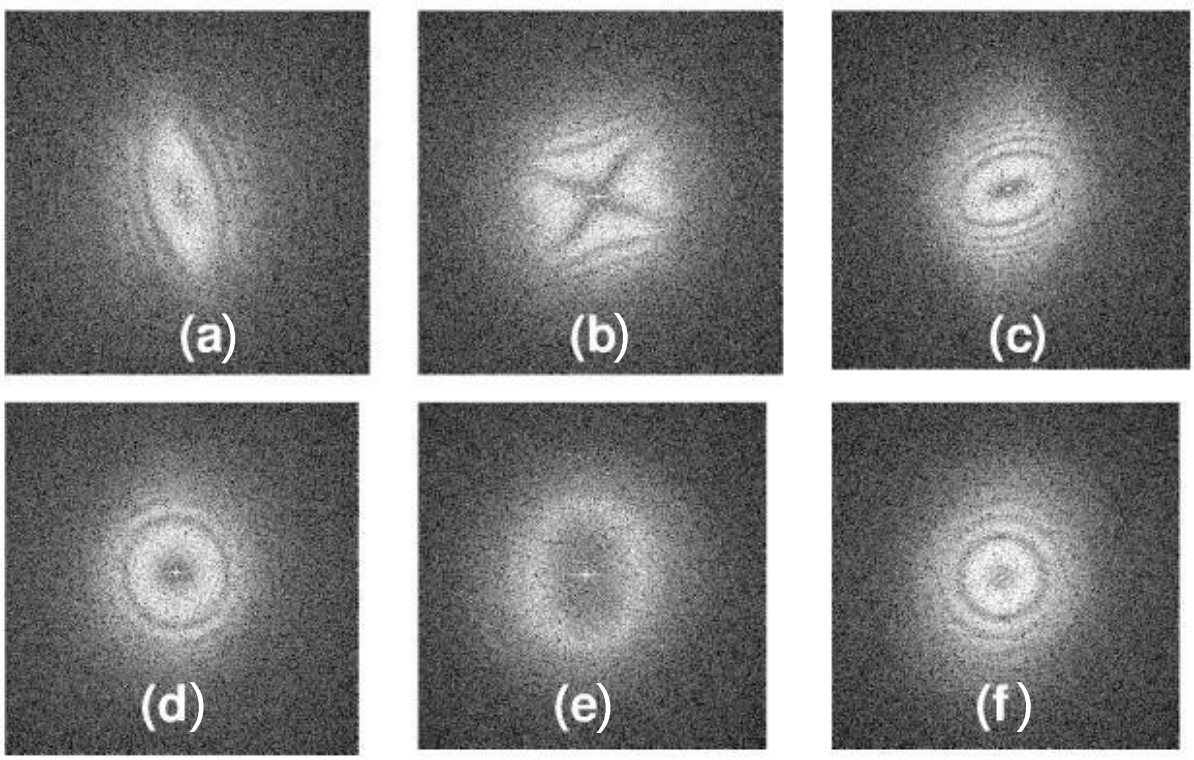


Figure 1: Image with strong astigmatism: (a) under-focus, (b) focus, and (c) over-focus. After almost fully correcting the astigmatism: (a) under-focus, (b) focus, and (c) over-focus.

Finishing your Session (Middle of the Day)

Just turn off the filament. You can leave the TEM HT at **300kV.**
Use the Filament Heating Ramp Control to turn off the filament (over left panel). The green lamp below **FILAMENT ON** must be on. Press down the **FILAMENT ON/OFF**. A red light will start flashing (below **FILAMENT OFF**), and subsequently a yellow lamp will light up and move from left to right. This process takes about one minute. Be patient. Only the red lamp at the right (**FILAMENT OFF**) is on. The **BEAM CURRENT** indication must be around **110μA**. If the red light is still flashing wait until it stops.

Turn the **FILAMENT** knob counterclockwise to set to **OFF**.

TEM phosphor screen must be down (no beam going to the cameras). Remove the objective and the SAD apertures if you did use them.

CCD Camera (Gatan MSC794): Leave it on, but the **CAMERA** key must be down (position **OUT**). Leave the TV camera on.

Removing Sample Holder (SH)

IMPORTANT: Before removing the SH, neutralize! 

To neutralize, press the **LOW MAG** button (right control panel). Select the coarse setting by pressing **CRS** button (left of the TEM column). Then press the **N** button to neutralize (left of the TEM column). This process automatically moves the SH to the position x=0, y=0, and also cancel the tilt angles. Wait until it finishes**.** Press the **MAG 1** button (right control panel). Now you can start removing the SH without risking damage the SH or the objective pole piece.

To remove the SH follow the instructions on the page at the end of this manual. A copy of it is also on the TEM. **Revisit the instructions before removing the SH.**

REMEMBER: The vacuum will pull the SH into the TEM. You should never rotate and pull the SH simultaneously. Do only one movement at time. If not, you may leak air and crash the TEM. 

Write down the following information in the log book:
Filament: ??????????? **Power: ???????????**
ΔFil: ????????

Before leaving the room, **PLEASE CHECK!**
- **FILAMENT** knob is **OFF**.
- TEM phosphor screen is down.
- TEM viewing port is covered.
- TEM telescope eyepiece is capped.
- Optical microscope is covered (table).
- **MAG 1** is on, and magnification set to **50k**.
- **PANEL LIGHT** is off.
- Contrast (objective) and Selected Area Diffraction (SAD) apertures removed.
- Second condenser aperture is in position.
- TEM computer screen is off (contrast and brightness to the minimum).
- **VACUUM IS OK.**

Finishing your working session (End of the Day)

Turn off the filament and the HT.

Use the Filament Heating Ramp Control to turn off the filament (over left panel). The green lamp below **FILAMENT ON** must be on. Press down the **FILAMENT ON/OFF**. A red light will start flashing (below **FILAMENT OFF**), and subsequently a yellow lamp will light up and move from left to right. This process takes about one minute. Be patient. Only the red lamp at the right (**FILAMENT OFF**) is on. The **BEAM CURRENT** indication must be around **110μA**. If the red lamp is still flashing, please wait until it stops.

Turn the **FILAMENT** knob counterclockwise to **OFF**. Remove the objective and the SAD apertures if you did use them.

Turning OFF the High Tension:

Check if the filament is off. Depress the **HT** button. The **BEAM CURRENT** reading will decrease (left panel) and at V=0 kV the **BEAM CURRENT** will read "**000" μA**.

Set the TEM acceleration voltage to **200 kV** using the TEM computer.
Type: **htset 200** ↵

Things to Turn Off:

- The TV camera (TV Control Unit, Gatan 622SC, red button **POWER**).
- Video processor (Gatan Model 750, black button **POWER** at its back).

The CCD camera (Gatan MSC794) is always left on, but the **CAMERA** switch has to be down (position **OUT**).

Removing Sample Holder (SH)

IMPORTANT: Before you remove the SH, neutralize the goniometer! 

To neutralize, press the **LOW MAG** button (right control panel). Select the coarse setting by pressing **CRS** button (left of the TEM column). Then press the **N** button to neutralize (left of the TEM column). This process automatically moves the SH to the position x=0, y=0, and also cancel the tilt angles. Wait until it finishes**.** Press the **MAG 1** button (right control panel). Now you can start removing the SH without risking damage the SH or the objective pole piece.

To remove the SH follow the instructions on the page at the end of this manual. A copy of it is also on the TEM. **Revisit the instructions before removing the SH.**

REMEMBER: The vacuum will pull the SH into the TEM. You should never rotate and pull the SH simultaneously. Do only one movement at time. If not, you may leak air and crash the TEM. 

Write down the following information in the log book:
Filament: ??????????? **Power: ???????????**
ΔFil: ????????

Before leaving the room, **PLEASE CHECK!**
- **FILAMENT** knob is **OFF**.
- TEM phosphor screen is down.

- TEM viewing port is covered.
- TEM telescope eyepiece is capped.
- Optical microscope is covered (table).
- **MAG 1** is on, and magnification set to **50k**.
- **PANEL LIGHT** is off.
- Contrast (objective) and Selected Area Diffraction (SAD) apertures removed.
- Second condenser aperture is in position.
- TEM computer screen is off (contrast and brightness to the minimum).
- **VACUUM IS OK.**

Summary

- **Check the vacuum (power supply closet) and start the TEM (HT and filament).**
- **Check if the TEM is neutralized and load the SH.**

1) **Find the Electron Beam (MAG and LOW MAG). Start at 50k.**
2) **Center the Condenser Aperture (aperture knobs).**
3) **Set DV=+-0. Find the Eucentric Center (Z height).**
4) **Optimize the Gun Tilt (GUN DEF).**
5) **Align the Gun and the Condenser (spot size 5 and 1, GUN SHIFT, BRIT SHIFT). Repeat as much as necessary.**
6) **Correct the Condenser Astigmatism (COND STIG).**
7) **Beam Tilt Purity (COND DEF ADJ and TILT).**
8) **Voltage Center (HT WOB).**
9) **Repeat steps 6-9 at 250k.**
10) **Center the Condenser Aperture.**

- **Start taking images.**
- **Shut-down the TEM accordingly (Filament, HT, neutralize SH, apertures, and TEM settings).**

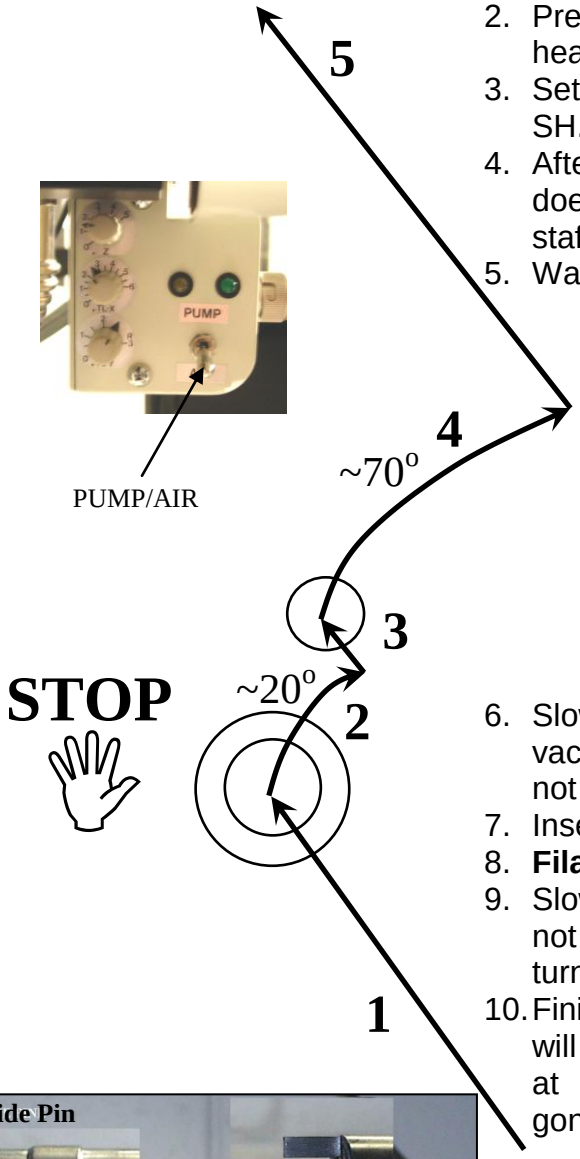
IMPORTANT: Know the instrument. Safety first.

Ionizing Radiation Generation

Magnetic Field

High Voltage Hazard

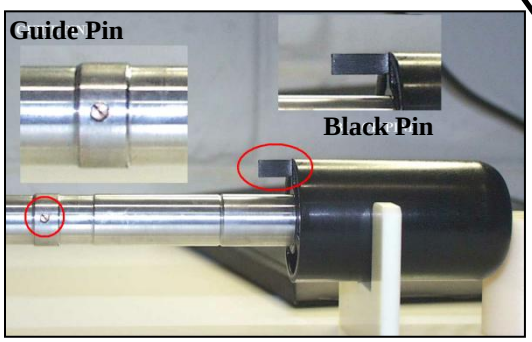
Inserting the Sample Holder into TEM Column



1. Align SH guide pin with the notch on the goniometer located at 9 o´clock to insert. Insert the SH.
2. Press the SH toward the column (step **1)**. You will hear the valves from the TEM.
3. Set the switch to **PUMP** position to pre-vacuum the SH. The yellow lamp will light on. Wait.
4. After about 10 minutes, the green light will turn on. If it does not turn on, please contact somebody from the staff.
5. Wait for more 10 minutes.

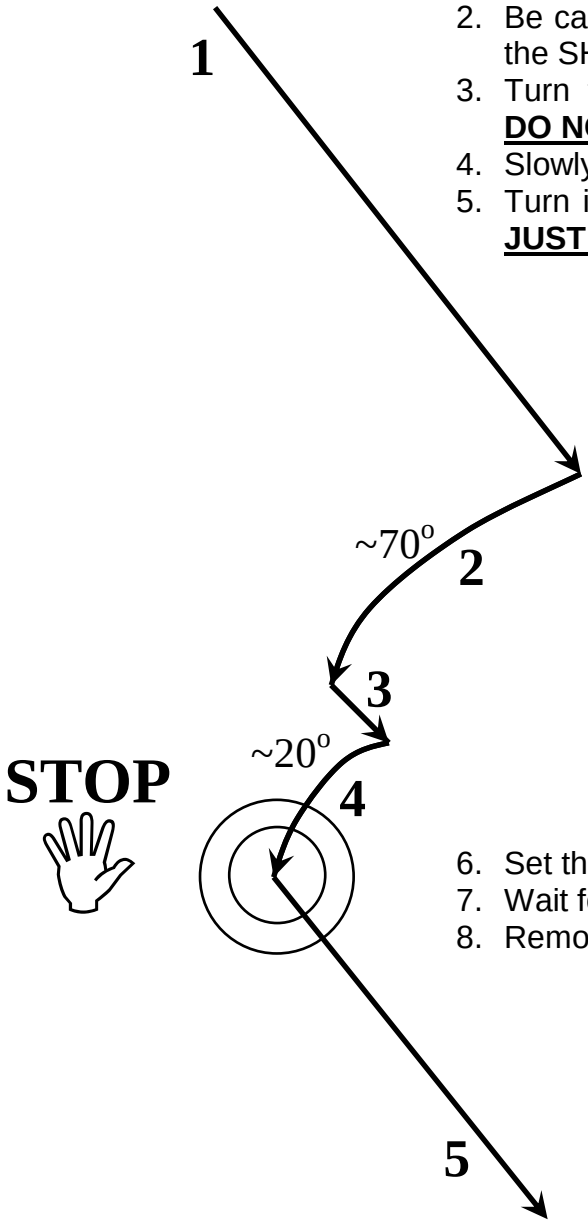
Never, never turn the SH clockwise before finishing the PUMP step.

6. Slowly turn the SH clockwise ~20° (step **2**). The vacuum will pull the SH after completing this step. Do not let it go and smash into the TEM.
7. Insert the SH carefully (step **3)**. It is less than 1cm.
8. **Filament** lamp will turn ON (left panel).
9. Slowly, turn the SH clockwise ~70° (step **4)**. Again, do not let it go and smash into the TEM after finishing turning the SH.
10. Finish inserting the SH (step **5)**. The SH´s black pin will fit in its place (small slot on the goniometer located at 3 o´clock). The gap between the SH and the goniometer is ~3mm.



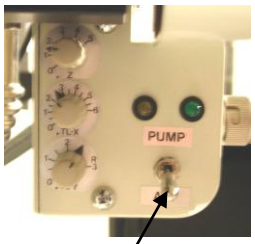
All the steps have be done slowly and always observing the column vacuum.

Removing the Sample Holder from the TEM Column



1. Neutralize the SH before start removing.
2. Be careful!! Slowly pull out the SH (step **1)**. Do not let the SH smash against the TEM column.
3. Turn the SH counterclockwise (ccw) ~70° (step **2**). **DO NOT PULL, JUST TURN IT CCW.**
4. Slowly pull out the SH (step **3**).
5. Turn it ccw ~20° (step **4**) and stop. **DO NOT PULL, JUST TURN IT CCW.**

Never, never remove the SH before finishing venting the TEM (AIR switch).



PUMP/AIR

6. Set the switch to **AIR**.
7. Wait for ~30 seconds.
8. Remove the SH (step **5**).

These procedures are done slowly and always observing the column vacuum.