Center for Nanoscale Systems
Harvard University
FAS / SEAS

Standard Operating Procedure

Hitachi TH7800 TEM (TEM-12)

Version 1.0 (October 2018)

Only users who have completed training by CNS staff are authorized to use this tool.

Emergency

In the event of an emergency, contact the nearest CNS staff member.
In *urgent* cases, such as

- fire or medical: call 911
- public safety: call Campus Police 5-1212
- all other: call University Operations center 5-5560

Safety

Instrument specific safety information:

- No eating or drinking in B15H, the HT7800 room
- Please return tools to proper location, leave the RT holder in the column, and clean up after yourself.

Contact

Please notify a staff member immediately if you encounter problems on the instrument. For assistance, please contact

\

Nicki Watson nwatson2@fas>harvard.edu

Acknowledge

CNS should be acknowledged in any publication resulting from work done using CNS facilities, staff or other resources:

This work was performed in part at the Center for Nanoscale Systems (CNS), a member of the National Nanotechnology Coordinated Infrastructure Network (NNCI), which is supported by the National Science Foundation under NSF award no. 1541959. CNS is part of Harvard University.

Start-up procedure

Log into CLEAN system
Hitachi7800 room B15H.

1. Check the front panel of the TEM.
   a. ***EVAC*** button should be lit
   b. ***GUN*** button should be lit

2. Check the lights on the lower panel to the right of the desk-
   a. ***EVAC*** should always be on;
   b. If ***COL*** of off, turn it on now.

3. The PC Should be left on. If it is off, press computer ***Power button***

4. Double Click CNS connect. This establishes connection to Z drive for storing data

(COL is the high voltage and lens power supply switch; software won't launch if COL is not on)

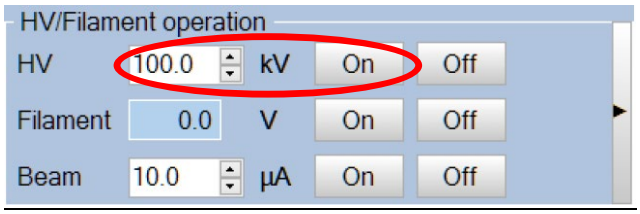
5. Launch [HT7800] software
6.

a. ç

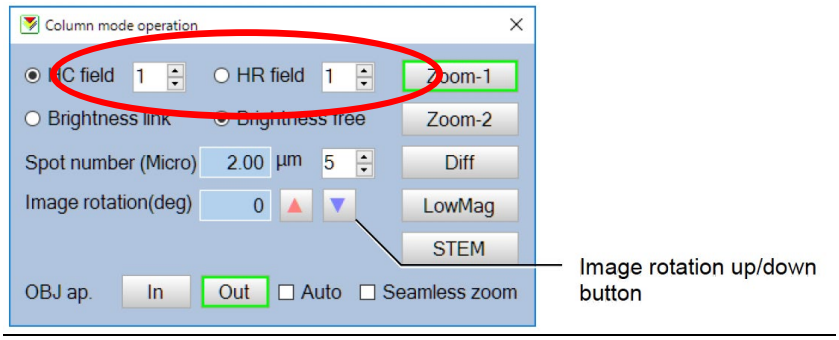
Software buttons are noted with [ ] while hardware features are noted with ***bold italics***

Set TEM conditions in 'HT7800' software

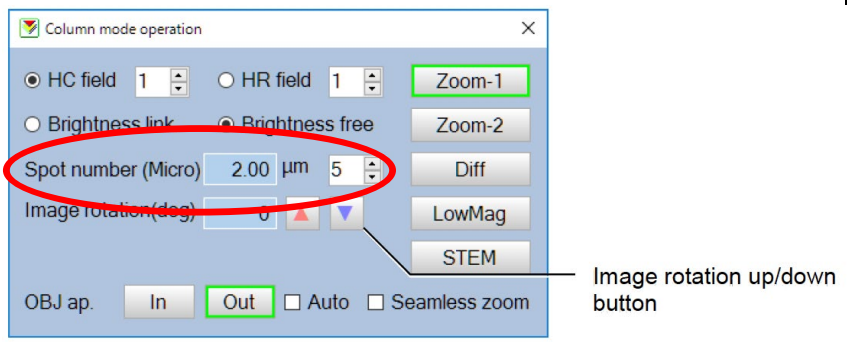
1. Go to the [HV/Filament operation] Window
2. Select KeV
   a. 80 kV: biological and low contrast materials
   b. 100 kV: thicker and high atomic mass materials
3. turn [On] the HV
   a. The [Beam] will automatically turn on with the [HV]
   b. Both [HV] and [Beam] will increase slowly to the set value



4. Check the [Column Mode Operation] window
   a. Decide if contrast or resolution is more important for your imaging then choose HC or HR
   b. [HC field] = high contrast imaging
   c. [HR field] = resolution favored over contrast



5. Select [spot number] appropriate for your work.
   a. Low magnification, high contrast [HC field] start with [spot number] 5
   b. High magnification, better resolution [HR field] start with [spot number] 1



Column mode operation	Spot 1	Spot 2	Spot 3	Spot 4	Spot 5
HC (high contrast)	0.60 µm	0.70 µm	1.00 µm	1.50 µm	2.00 µm
HR (high resolution)	0.60 µm	0.65 µm	0.70 µm	0.80 µm	1.00 µm

6. Load appropriate Personal Data Set [PDS] for the voltage you have selected
   a. Click [PDS] icon (lower left corner of software)
   b. In the new window, click on alignment that matches the accelerating voltage and field type previously selected
   c. Click [load] button

7. Load samples (pages 4-7)
   a. Prepare for sample holder removal
   b. Remove holder from the goniometer
   c. Load sample grid(s) into standard holder
   d. Load holder into goniometer

See pages 4-7

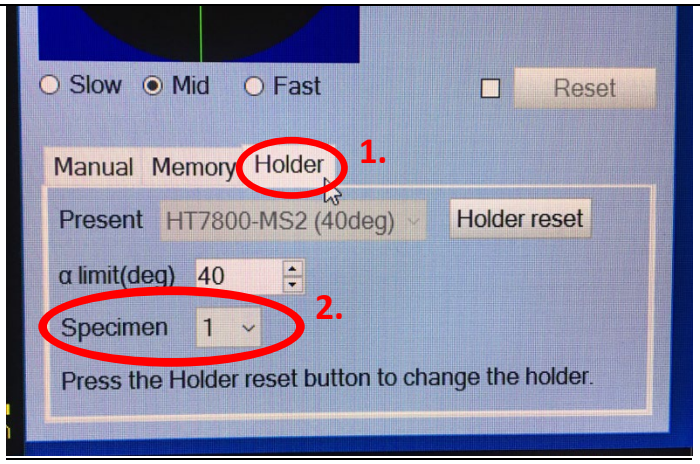
Sample Exchange:

Prepare for sample holder removal

1. Make sure the holder matches the setting on the [holder] tab of the [Stage Operation] pop-out window
    a. Press the [Holder reset] button
    b. Make sure the holder you are using is listed under the [Holder] tab

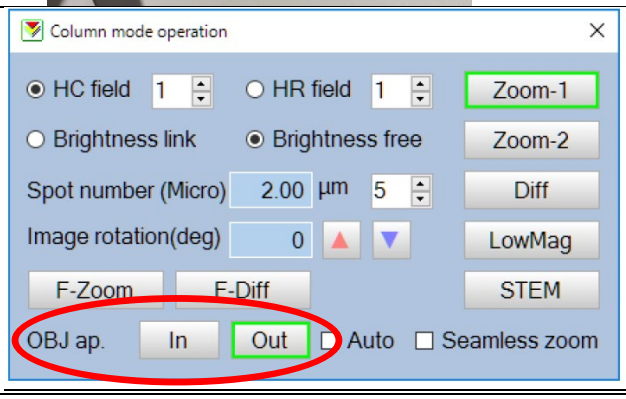
	holder	Max tilt	X-axis range
Standard	HT7800MS2	40deg	400µm
Tomography	HT7800-SS	70deg	250µm

2. When using the standard holder, an option to select the specimen number appears in the holder window. Use this to switch between specimens

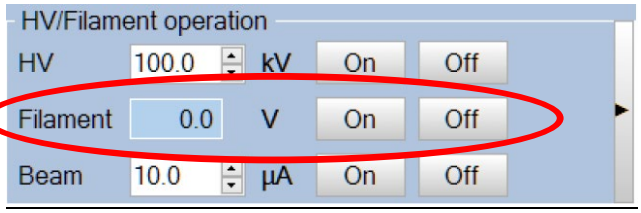


3. Reset Objective lens voltage
    a. Press ***LENS RESET*** on the hard panel

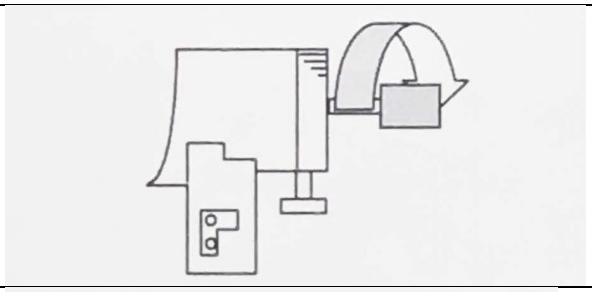
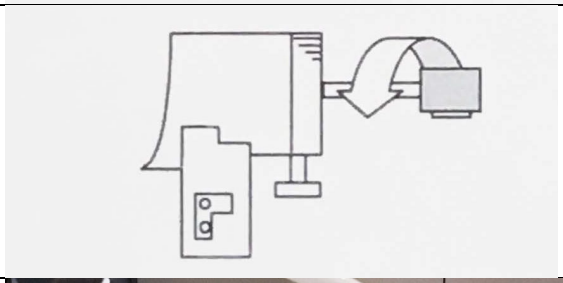
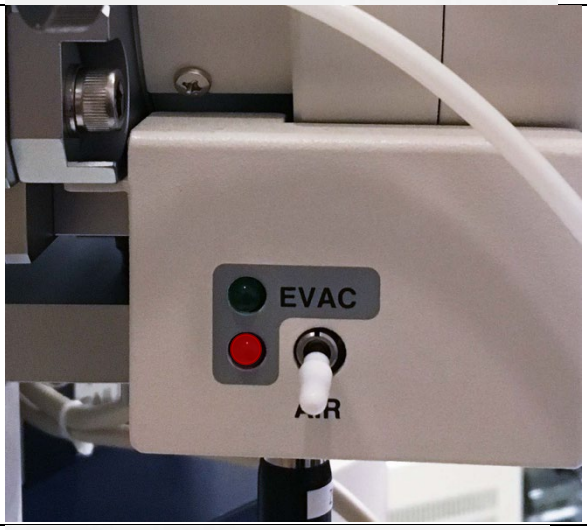
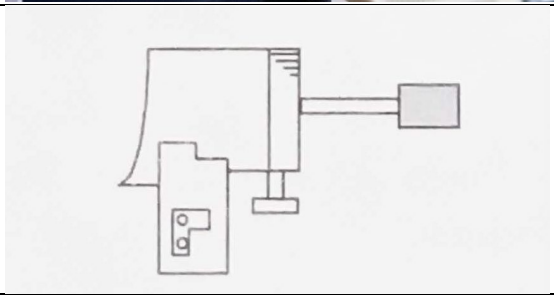
4. Check [Column mode operation] pop-out window and make sure the [OBJ ap.] Is [Out] F1



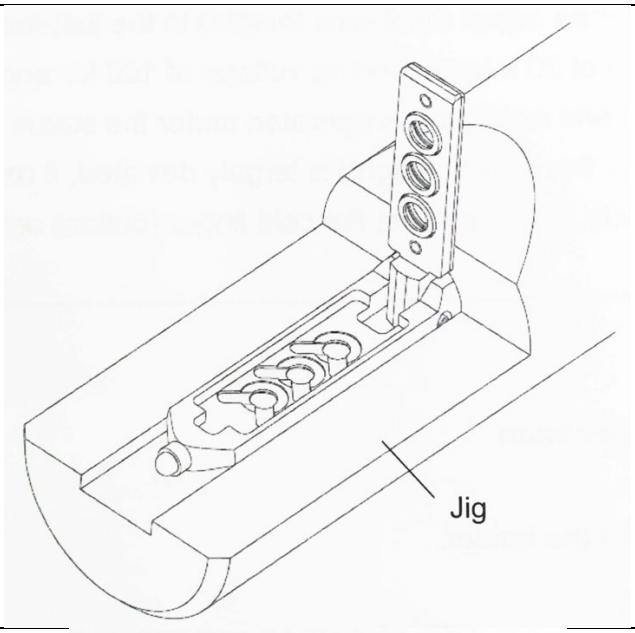
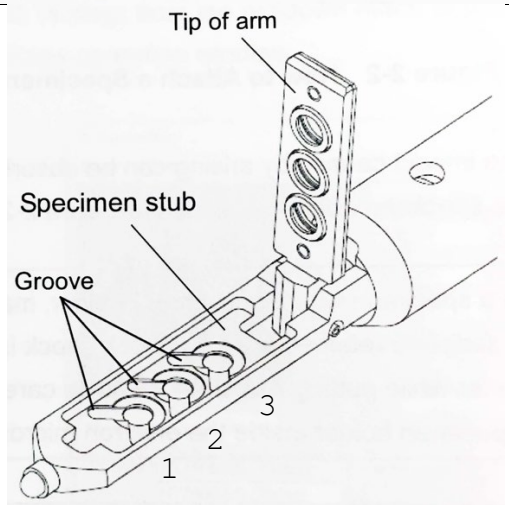
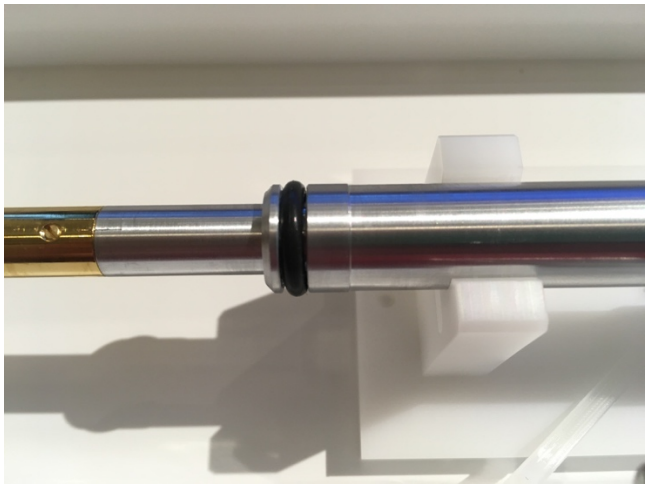
5. Make sure Filament is off 0.0V [HT/Filament] window
6. OPTIONAL



Remove holder from the goniometer

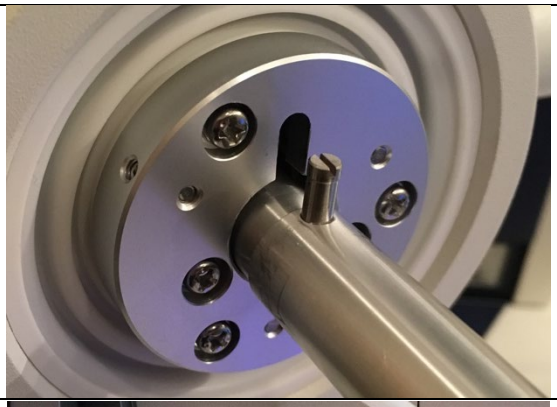
****Make sure stage is homed**** See page 4, item 1 for instructions 1. Pull holder straight out until it comes to a stop- 2. Turn CLOCKWISE 15° (it will stop)	
3. Pull the holder out again until it stops, 4. turn it COUNTER CLOCKWISE 45° **STOP**	
5. Turn the specimen chamber switch to AIR **WAIT** for the **red** light to come on	
6. Carefully break the vacuum seal 7. Remove the holder from the goniometer Do not touch anything past the O-ring towards the tip of the holder without clean gloves on	

Load sample grid(s) into standard holder

1. Place standard holder in acrylic stand 2. Place jig over end of rod *(do not touch the end of the rod with bare hands!)* 3. Using blunt forceps, lift the retaining arm.	
4. Using fine forceps, place your grid(s) over one of the holes on the specimen stub, making sure it is within the recessed edge. a. Position **1** is at the tip b. Position **2** is the middle c. Position **3** is closest to the handle 5. Close the retaining arm.	
6. Check the O-ring for obvious lint, dirt, or damage; gently remove any contaminates with lens paper if necessary.	

Load holder into goniometer

1. Check for position of small pin on the holder.
2. Line this up with the slot on the goniometer



3. Carefully insert holder into the goniometer until it comes to a stop

   Turn the specimen chamber switch to EVAC

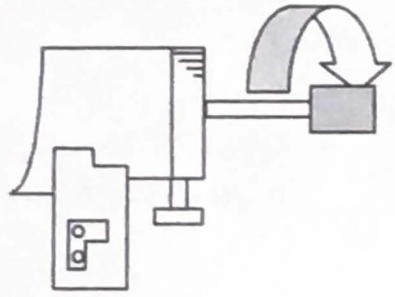
   WAIT

   for the **GREEN** light to come on

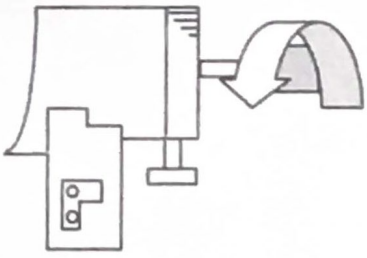
4. When the pre-pump cycle is complete, the **Green** light will glow, and the TEM will beep.
   a. If you wait too long, you will need to repump the airlock. (10 seconds)

Now it is safe to fully insert the holder into the column of the TEM.

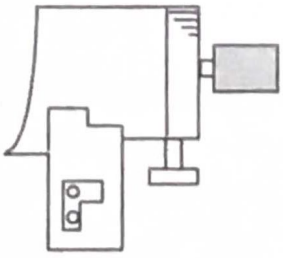
5. turn the holder 45° clockwise



6. then turn it counter-clockwise 15°



7. Wait for the column vacuum to come back down to < $5X10^{-5}$ Pa (with a tungsten filament)

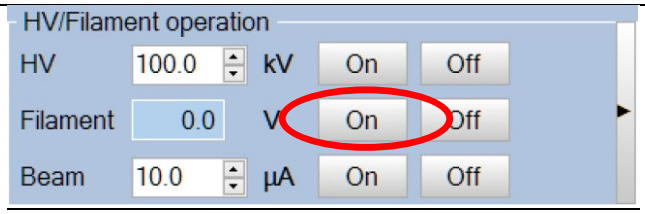


Find and center the beam use SCREEN camera

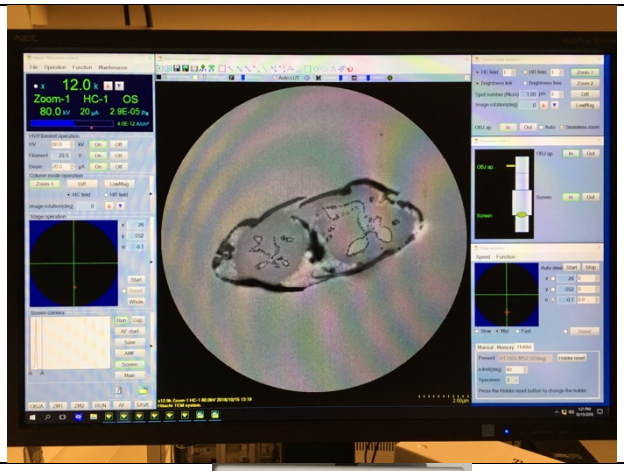
1. Go to the [HV/Filam.] window
2. turn on [Filament] to generate beam

<u>options for blanking (stopping) the beam</u>

- bias blanking: turn off Beam
- sample exchange: turn off filament (Optional)
- end of session: turn off HV



3. Using the [Screen] camera, you should see the circle mask around the image in the middle of the left monitor



4. If you do not see a transmitted electron image
   a. MAKE SURE YOU ARE LOGGED INTO THE CLEAN SYSTEM
   b. Reduce ***MAGNIFICATION. Lens reset***
   c. Use the ***STAGE CONTROL*** trackball to move the stage/sample
   d. Change ***BRIGHTNESS*** to spread/condense the beam

5. Condense and center the beam
   a. Set the ***MAGNIFICATION*** to 5KX or higher
   b. use the ***BRIGHTNESS*** knob and turn so that the beam becomes smaller
   c. Continue turning until the beam condenses into a spot
   d. Press the ***BH*** button in the ***ALIGNMENT*** box on the hard panel
   e. Use the ***X*** and ***Y*** knobs to center the beam
   f. Spread the beam ***BRIGHTNESS***

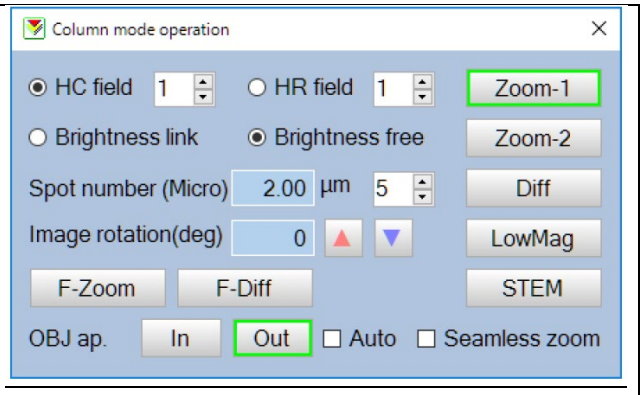
You will use a focused, or condensed spot of illumination frequently. This is also referred to as "Crossover"

User daily alignment

Center condenser aperture use SCREEN camera

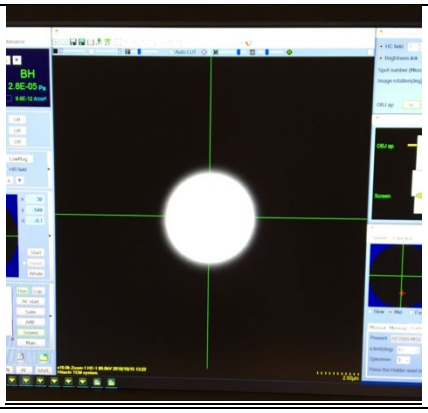
1. Select [spot number] appropriate for your work.
   a. Low magnification, high contrast [HC field] start with [spot number] 5
   b. High magnification, better resolution [HR] start with [spot number] 1

Column mode operation	Spot 1	Spot 2	Spot 3	Spot 4	Spot 5
HC (high contrast)	0.60 µm	0.70 µm	1.00 µm	1.50 µm	2.00 µm
HR (high resolution)	0.60 µm	0.65 µm	0.70 µm	0.80 µm	1.00 µm



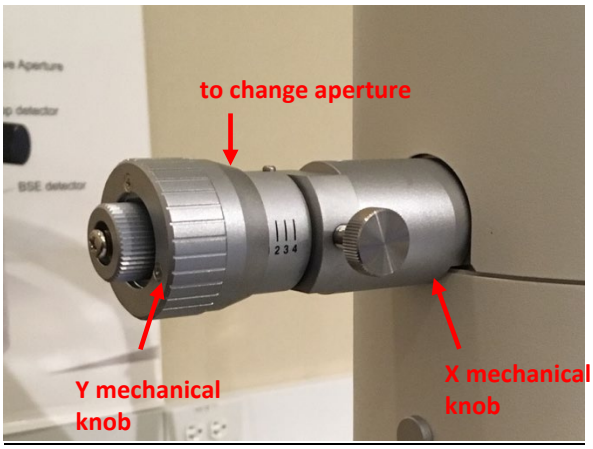
2. bring the beam to a spot ***BRIGHTNESS***

3. Center spot on phosphor screen using ***X, Y*** knobs while ***BH*** is lit



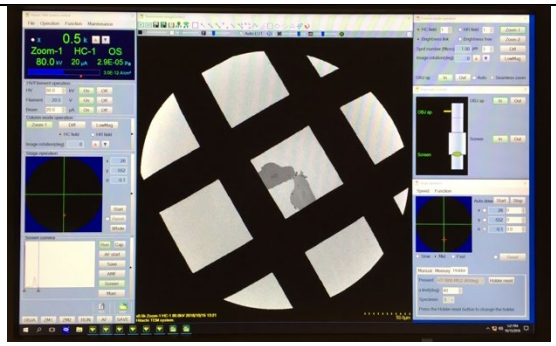
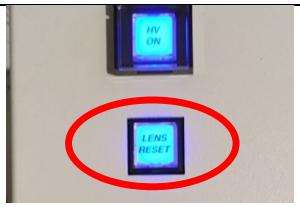
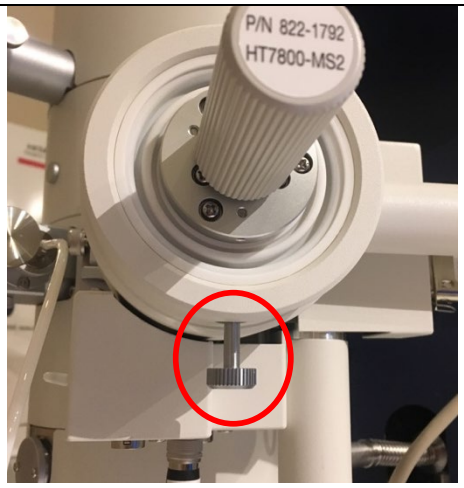
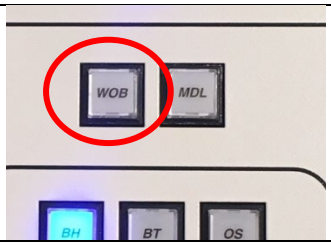
4. Spread beam ***BRIGHTNESS***, Clockwise( ) until part of the illumination just reaches the edge of the circle mask
   a. If all of the illumination reaches the edge at about the same time, it is already aligned; go to 5a.
   b. Use condenser **X, Y *mechanical knobs*** to center illumination if it does not reach the edge at the same time
   c. If the beam is off significantly, only bring illumination halfway to center

5. Condense the beam, ***BRIGHTNESS***, counter clockwise ( ) and spread to edge of the circle mask
   a. If all of the illumination reaches the edge at about the same time, it is already aligned.
   b. Use condenser ***X, Y mechanical knobs*** to center illumination if it does not reach the edge at the same time
   c. If the beam is off significantly, only bring illumination halfway to center



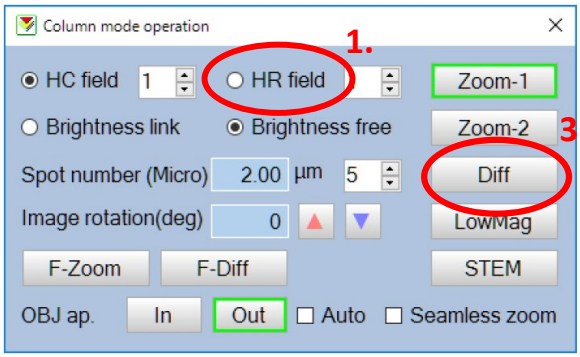
6. Go to the highest mag needed, then repeat steps 3-5 until beam spreads evening on both sides of crossover

Sample height adjustment use SCREEN camera

1. Find an object in your sample (not a grid bar) a. Use ***STAGE CONTROL*** trackball to move sample b. Adjust ***MAGNIFICATION*** as needed c. Adjust ***BRIGHTNESS*** if needed	
2. press ***LENS RESET***	
3. press ***WOB*** so it is lit a. the sample should be moving as view from the [SCREEN] camera b. if the sample is not moving, the Z-height is already set	
4. adjust the Z-height until the image stops moving a. locate the ***knob*** on the ***goniometer*** just below where the ***sample rod*** is inserted b. while looking at the image on the monitor, turn this ***knob*** until the image stops moving	
5. press ***WOB*** so it is dim	
6. Increase magnification to the highest value you will need for your sample	
7. Repeat steps 3-5.	

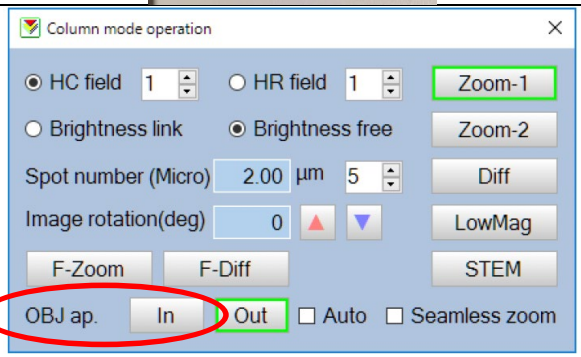
Inserting and aligning objective aperture use SCREEN camera

1. Switch to [HR field] in [column mode operation] pop-out window
2. Spread the beam ***BRIGHTNESS*** beyond the edge of the round mask on the screen camera
3. Change the column mode operation to [Diff]

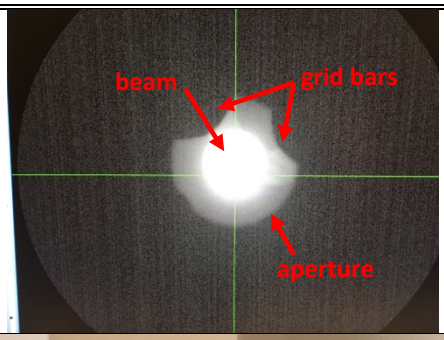


4. press ***LENS RESET***

5. insert objective aperture
   a. [Column mode operation] pop-out window
   b. Click [IN] box next to [OBJ ap.]



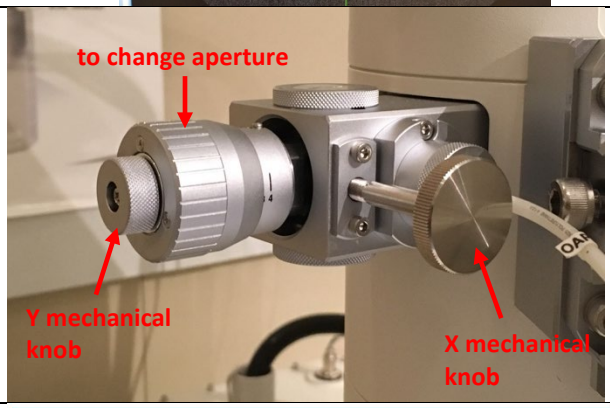
6. change the camera length using the ***MAGNIFICATION*** knob to 2M
7. spread the beam ***BRIGHTNESS*** knob counter clockwise ↺



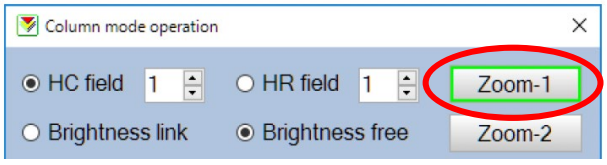
8. Using the ***mechanical knobs*** on the ***objective aperture drive***, move the image of the aperture to evenly surround the brightest point of the beam
   a. If needed, turn the ***large knob*** clockwise ↻ to allow a smaller aperture to move into place.

Obj. apt. position:	No. 1	No. 2	No.3	No. 4
Apt. diameter	150 µm	90 µm	25 µm	15 µm

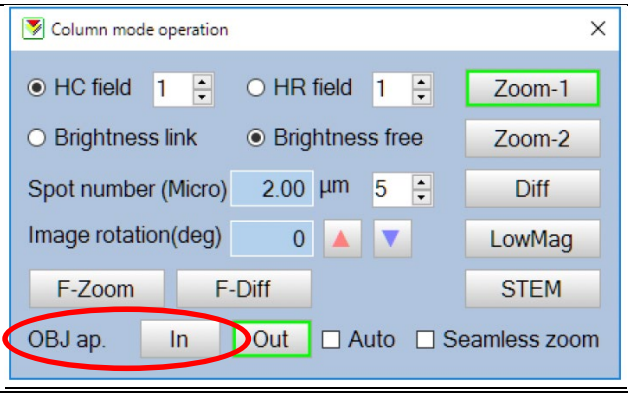
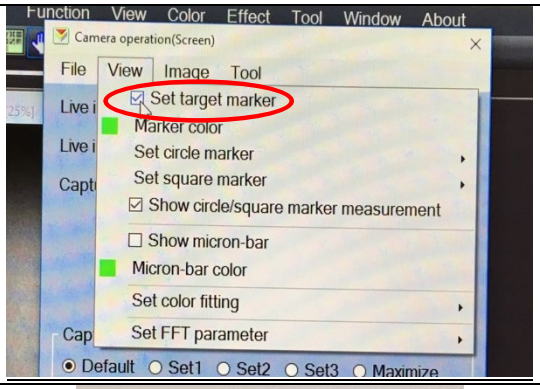
   b. Turn and pull the ***large knob*** counter clockwise to move to a larger aperture



9. Return [column mode operation] to [Zoom-1 HC Mode if used]
10. Condense the beam by turning the ***BRIGHTNESS*** knob clockwise ↻ to about the size of the round mask

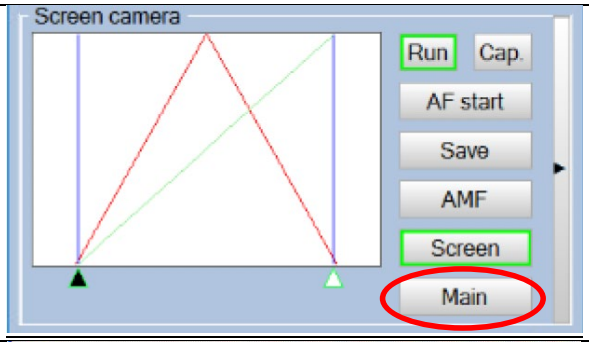
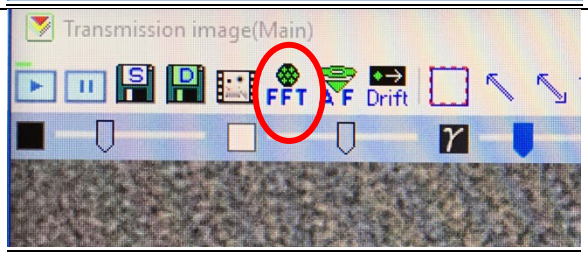
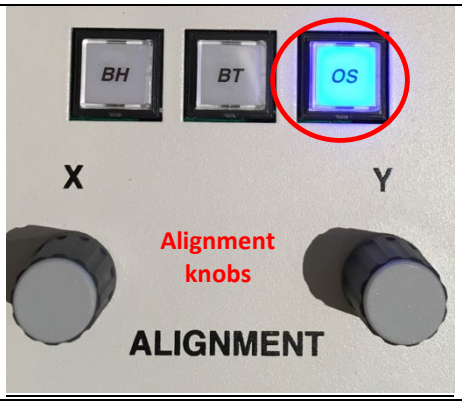


Voltage center use SCREEN camera

2. Insert the objective aperture a. [Column mode operation] pop-out window b. Click [IN] box next to [OBJ ap.]	
3. Focus on a round, high-contrast feature at around 30KX	
4. move this feature to the center of the round screen using the ***STAGE CONTROL*** trackball a. to view cross-lines marking the center i. go to the [Camera operation (screen)] pop-out window ii. click on the [view] drop down menu iii. select [set target marker] b. you can also select to view or hide circle and square markers, and show measurements	
5. press the ***MDL*** button on the hard panel a. adjust the ***X*** and ***Y alignment*** knobs so the image gets bigger and smaller, but does not shift b. only the very center of the screen can be corrected/aligned	
6. press the ***MDL*** button again to turn it off 7. After this is complete, you may notice the beam is not centered. If so Center using ***BH***, then repeat step 5. Repeat this until the beam stays centered when ***MDL*** is completed.	

Objective lens stigmation (using FFT) use MAIN camera

1. Focus on a uniform/background area of your sample at a magnification higher than your needs	

2. Switch to [main] camera in [Screen camera] window	
3. Generate a live FFT image a. Find the [FFT] icon at the top center of the left monitor (you will need to adjust the Black and white scale on FFT to see features. Pressing R is helpful b. Click on it c. A new window will appear; it may be on the right-hand monitor (you will need to adjust the Black and white scale on FFT to see features. Pressing R is helpful)	
4. Activate the objective lens stigmation coils ***OS***	
5. Use ***ALIGNMENT*** knobs ***X, Y*** to generate a round pattern in the FFT image	
6. Frequently check focus; objective astigmatism is most noticeable when over-focusing and under-focusing.	
7. Turn off ***OS***	

Recording images

In-between Samples

1. Turn off filament
2. Remove objective aperture
3. Reset holder position to zero
4. Remove holder
5. Return holder
6. Wait for Vacuum to recover 5X10-5 Pa
7. Turn on Filament

Shut Down Procedure

If someone is using the HT7800 in the next 4 hours:

1. Turn off Filament
2. Remove objective aperture
3. remove your last sample(s)
4. return the holder to the column
5. Leave HT on and software open
6. Log out of the clean system

If you are the last user of the day:

1. Turn off Filament
2. Remove objective aperture
3. remove your last sample(s)
4. return the holder to the column
5. Leave HT on and software open
6. Log out of the clean system