

# JEM 2100 Manual

## Operation and Basic Alignment Instructions

### 1. Verify the vacuum:

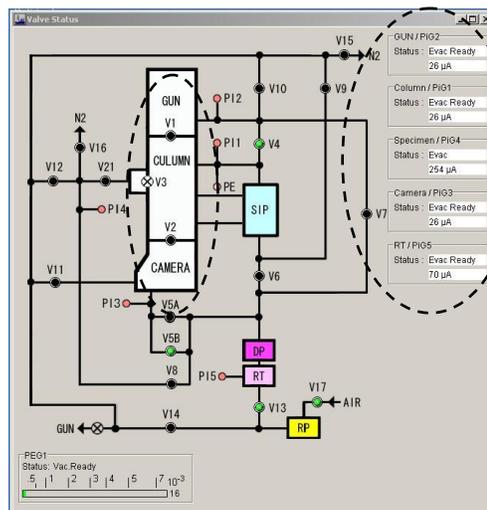
- ✓ Check the vacuum levels (power supply closet)



- Column (blue scale):  $< 2.5 \times 10^{-5}$  Pa

If vacuum is not good enough, contact somebody from the LME staff.

- ✓ Verify the vacuum in JEOL PC / TEMCON / Valve Status window



### VACUUM SYSTEM

- GUN/CAMERA valves
  - V1 and V2 = OFF (black)
- Pirani gauge status
  - PIG-2  $< 30$   $\mu$ A
  - PIG-1  $< 30$   $\mu$ A
  - PIG-4  $\cong 254$   $\mu$ A
  - PIG-3  $< 30$   $\mu$ A
  - PIG-5  $\cong 100$   $\mu$ A

### 2. Complete Cold Finger Dewar with Liquid Nitrogen:

- ✓ Fill with LN<sub>2</sub>, wait for a few minutes and complete the Dewar after it stops boiling.



- The microscope's view port must be covered before this procedure. The window can crack under thermal shock.
- Wear appropriated protective eyeglasses and gloves when manipulate LN<sub>2</sub> to avoid accidents.





**ATTENTION!!! Complete the dewar with LN<sub>2</sub> at each 6 working hours. In working days, in the morning (7 o'clock AM) and in the afternoon (1 o'clock PM).**

### 3. Check the camera:

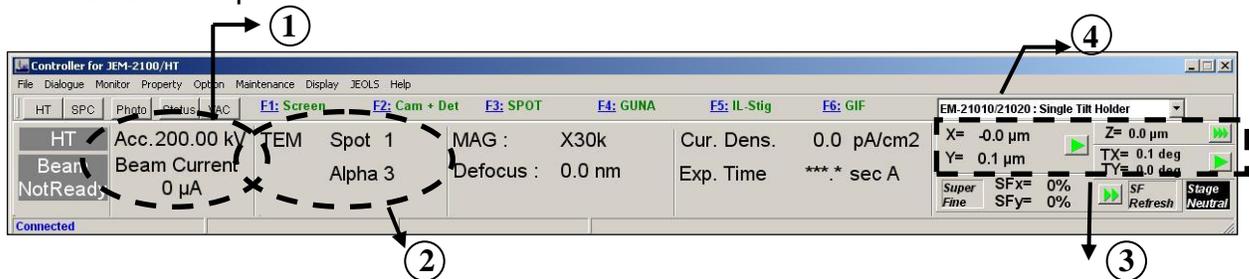
✓ **Check if the shutter box (TVIPS camera) is in CCD mode** (right side of the TEM Column). **Put a picture**

✓ **Check if CCD ES 500W is ON** (left side of the TEM Column).



### 4. Verify the TEMCON / Controller window (JEOL PC):

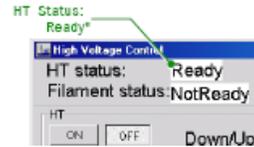
- If the program is not open click double-click on the TEM Controller icon in the PC monitor screen.
- Check the points bellow:



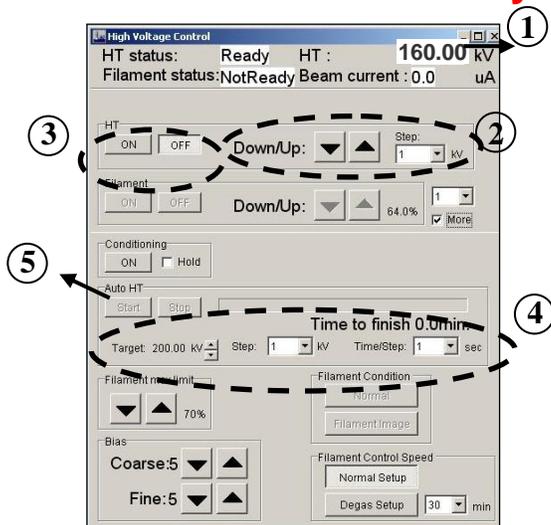
- 1) High Voltage (HT) : Acc. = 160kV (Start) or 200kV (Middle of the Day);
- 2) Spot Size = 1, Alpha = 3;
- 3) X = Y = 0, Z = 0, TX = 0° (TY doesn't matter);
- 4) Select the sample-holder (SH) type that you use:  
Single Tilt: EM-21010 = Single Tilt Holder;  
Double Tilt: EM-31630 = Specimen Tilt Holder (DTC);  
Single Tilt Rotation: EM-31650 = Rotation Holder (STR);  
Double Tilt Beryllium: EM-31640 = Specimen Tilt Holder Be (DTB);

## 5. Turning on the Accelerating Voltage

- Pa
- ✓ Make sure that the reading of the column pressure gauge is  $< 2.5 \times 10^{-5}$  Pa
  - ✓ Make sure that the HT Status = Ready (HT allowed) on the right upper corner of the window High Voltage

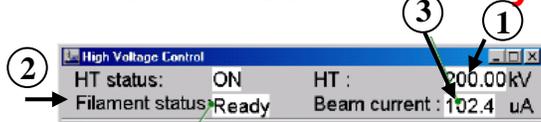


### For 1<sup>st</sup> user of the day:



- 1) Verify that Acceleration voltage has been set to Acc = 160 kV
- 2) **If not**, choose the Voltage Step = 10 kV in the right side of HT menu and using **Down** button decrease the **HT** to **160 kV**;
- 3) Click on **HT ON** icon. Current on **Beam Current** indicator must increase gradually. When **Beam Current** = **81 μA**, Voltage reached the correct value **HT = 160 kV**.
- 4) Increase voltage from 160 to 200 kV (**Target = 200kV**): In **Auto HT** Step = 0.1 kV and Time = 3 sec (Time to Finish = 20 min). (**PS: If the microscope has not been used for several days use 40 minutes – Time/Step = 6 s**).
- 5) Click on **START** to increase HT to 200 kV. While the **Accelerating Voltage** window is open, you cannot access any other windows

### For 2<sup>st</sup> user of the day:



- 1) Verify that Acceleration voltage has been set to Acc = 200 kV
- 2) Verify that the HT icon is **on**.
- 3) Current on **Beam Current** indicator must be **102 μA**. **If not**, repeat the step for 1<sup>st</sup> user.

## 6. Introducing the sample holder (SH):



**Always use glove when to manipulate the sample holder or your sample. The gloves are not for your protection. You are the main source of contaminations.**

- ✓ Put your sample on the adequate SH.



**Remember!!! Don't use the scalp to manipulate your sample. It can seriously contaminate it. Use the pick-up vacuum system on the working table.**

✓ For single tilt holder, loosen two screws TWO turns, rotate the cover plate away and mount specimen with film side UP. For double tilt holder, loosen two screws TWO turns, rotate the two plate-clamping fingers and mount sample facing DOWN. NEVER OVERTIGHTEN THE SCREWS!!!

✓ After to put firmly 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.



**Be Careful!!! Don't introduce the SH on the microscope until be sure that it is completely free of dust or lint. One can induce vacuum leak!!!**

✓ Introduce the SH in the microscope. Follow the instructions contained at the end of this manual, or in the copy that is **Always** over the TEM console. (**Be careful and always look at the Vacuum indicator**).



**Remember!!! After step (1) wait several minutes till Specimen/PIG4 indicator is just below 32  $\mu$ A (ideally 30  $\mu$ A). After this vacuum level be attained introduce completely the SH (steps 2 to 5).**

**Suring this time, write the informations in the Microscope Notebook:**

✓ User Name,  
Column Pressure  
Sample Holder,  
Sample name,

✓ **Check again the vacuum levels (item 1)**



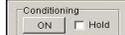
**If you will perform EDS analysis, insert the EDS detector at this moment to avoid an afterward gun misalignment.**

## 7. Heating the filament:

- ✓ Verify that Filament Status: READY (Filament Enabled).

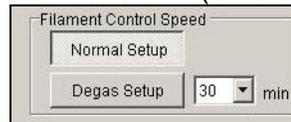


### **NEVER PRESS CONDITIONING BUTTON ON HT CONTROL WINDOW:**



This button implements HT conditioning and is used only when a JEOL service engineer asks you to operate

- ✓ In Right Lower Corner of High Voltage Control Window choose in Filament Control Speed the Heating Mode DEGAS SETUP - 30 min (1<sup>st</sup> user of the day) and NORMAL SETUP (2<sup>st</sup> user of the day).



<b>Normal Setup:</b>	Applies the current to the filament at the speed set in the factory.
<b>Degas Setup:</b>	Click on this button when degassing the filament. Specify the ascending time in the combo box.



**ALWAYS CHECK IF SOFTWARES NORAN SYSTEM SIX IS CLOSE BEFORE HEATING THE FILAMENT!!!**

- ✓ To Heat the Filament click ON button under HT in the Left Middle of High Voltage Control Window.

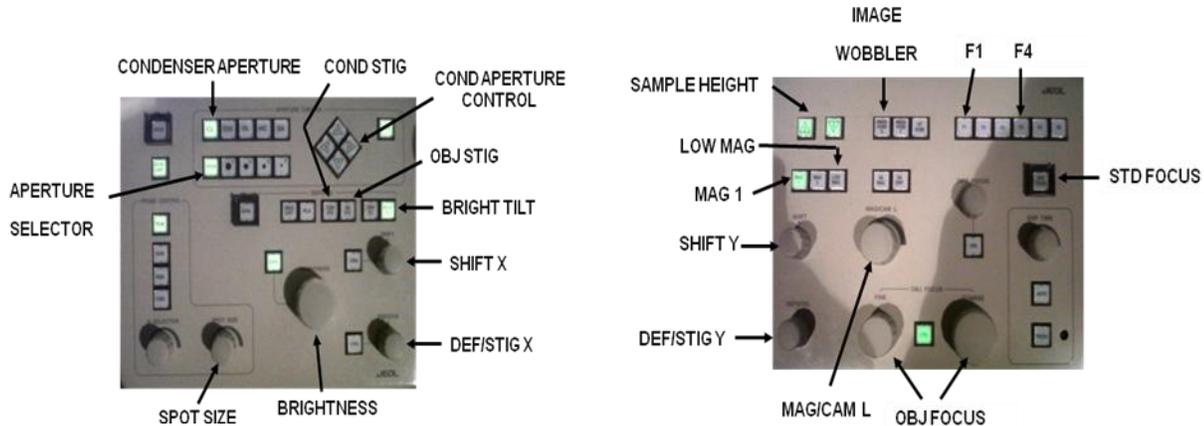


- ✓ A small window opens: Filament → Setting the Filament: going from 0 to ~57%. Wait until this window close and Beam Current = 112  $\mu$ A. While the Filament window is open, you cannot access any other Windows.

- ✓ When the filament shutdown is complete, the Filament window closes and you have got an electron beam in the phosphorous screen of the microscope

## TEM MODE ALIGNMENT

### Control Panels



Most of the commands on the control panels (right and left) related to Standard Operation, Apertures and Movements of the Stages can be accessed in **Operation** menu.

### **8. Finding the electron beam:**

- ✓ If the beam is not visible on the phosphor screen, set to a lower magnification (10 kx) with the MAG/CAM L knob, and move the sample using the track ball until the beam be visible. Put the sample in an open region.
- ✓ Set magnification to 50 kx with the MAG/CAM L knob for the initial adjustments.

### **9. Centering the Condenser Aperture:**

- ✓ To insert the Condenser Aperture press CL button and choose the Aperture #2 to be inserted (If other Aperture # is Light On, means that you are changing CL aperture. If no aperture is inserted, OPEN Lights On). The second biggest aperture is the standard one.

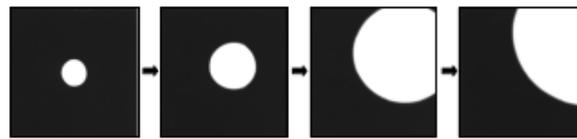
**a)** Remember to always compensate the condenser lens hysteresis (**BRIGHTNESS** knob, counterclockwise-crossover-clockwise).

b) 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**.

c) Defocus (clockwise) the beam till approximately the same screen diameter. If the beam is not centered use the aperture position adjust to center it (left panel).



**Important: Remember to check the condenser aperture alignment from time to time. It is mechanical and it will misalign more often than you imagine.**



(a) Aperture is not correctly inserted.



(b) Aperture is correctly inserted.

## **10. Sample Height (Eucentric Center)**

- Press Standard Focus STD FOCUS button in the Upper Right Control Panel to optimize the focus of Objective Lens (OL).
- Use IMAGE WOBLER X or Y (right panel). The image will start to oscillate. You need to stop it from moving by adjusting Z control buttons (right panel) at the right position.
- Disable IMAGE WOBLER X or Y.

## **11. 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 (F4 button on the Keyboard – Right Control Panel). Turn **DEF X** and Y knobs until maximize **CUR. DENS.** (Current Density =  $\mu\text{A}/\text{cm}^2$ ) measurement of the Mic. Computer screen.

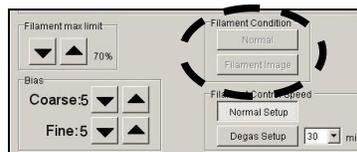
**Disable F4 gun buttons by clicking on it again!!!**

## **12. Optimizing the Gun Shift (Gun ↔ Condenser Lens Coupling):**

- a) Repeat 9a to 9d.**
- b) Set SPOT SIZE to 5.** Focus the beam with BRIGHTNESS knob, and center it by using the SHIFT knobs.
- c) Set SPOT SIZE to 1** and refocus the beam with BRIGHTNESS knob.
- d) Press F4 button** (Gun command) and center the beam with the SHIFT knobs. **Disable F4 button.**
- e) Repeat the procedures a to c** several times till the focused beam don't move from the center when the spot size is modified from 5 to 1. **Disable F4.**

## **13. Condenser Lens Astigmatism Correction:**

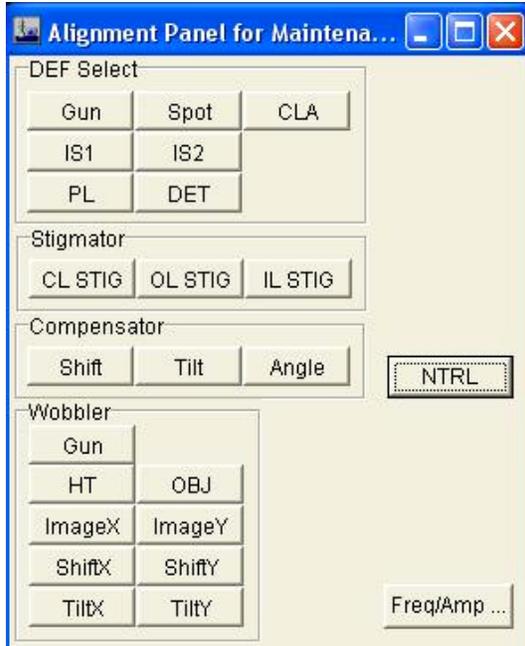
- a) Repeat 9a to 9d.**
- b) Set magnification to 250 kx.**
- c) Focus the beam with the BRIGHTNESS knob** (at fine) to smallest size as possible.
- d) In HIGH VOTAGE CONTROL window,** in the menu FILAMENT CONDITION click on FILAMENT IMAGE in order to see the filament image desaturating it.



- e) Press COND STIG** and improve the image quality with DEF X and Y knobs to obtain the smallest and brightest spot.
- f) With BRIGHTNESS knob** check if there is astigmatism (i.e. the image is distorted in perpendicular directions if the BRIGHTNESS knob is turned from under-focus to over-focus).
- g) Repeat e** till a good correction. **Disable COND STIG.**

## **14. Beam Tilt Correction (Condenser Lens ↔ Objective Lens Coupling):**

- a) Press **STD FOCUS**.
- b) Repeat 9a to 9d.
- c) Focus the beam with the **BRIGHTNESS** knob (at fine) to smallest size as possible.



- d) In tool bar click Maintenance → Alignment.
- e) In Alignment Panel click **TILT X** on Wobbler box.
- f) Click **TILT** on Compensator box.
- g) Use **DEF X** knob to stop the **longitudinal** movement of the central bright spot.
- h) Click **ANGLE** on Compensator box.
- i) Use **DEF X** knob to stop the **transversal** movement of the central bright spot.
- j) Click **TILT Y** on Wobbler box.
- k) Click **TILT** on Compensator box.
- l) Use **DEF Y** knob to stop the **longitudinal** movement of the central bright spot.
- m) Click **ANGLE** on Compensator box.
- n) Use **DEF Y** knob to stop the **transversal** movement of the central bright spot.
- o) Disable **TILT Y**.

## **15. Voltage Center (Coma-Free alignment):**

- a) Put the sample at the correct height (in focus)
- b) Press **WOBBLER HT**. The image will oscillate.
- c) Press **BRIT TILT** and use **DEF X** and **Y** to minimize the oscillation. If properly adjusted, the image will pulsate symmetrically.
- d) Disable **WOBBLER HT** and **BRIT TILT** buttons.

✓ If you prefer, you can use the **ESW 500** camera to adjustment the voltage center. To use the camera, 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. In the



**Remember to never image the diffraction pattern or the focused beam directly on the camera.**

## **18. Objective Lens Astigmatism Correction:**

- ✓ 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 camera to do the final touch.

### **Camera TVIPS**

#### **1. Open the EMMENU program**



- ✓ Click on icon in the PC monitor screen.

#### **2. Save the images**

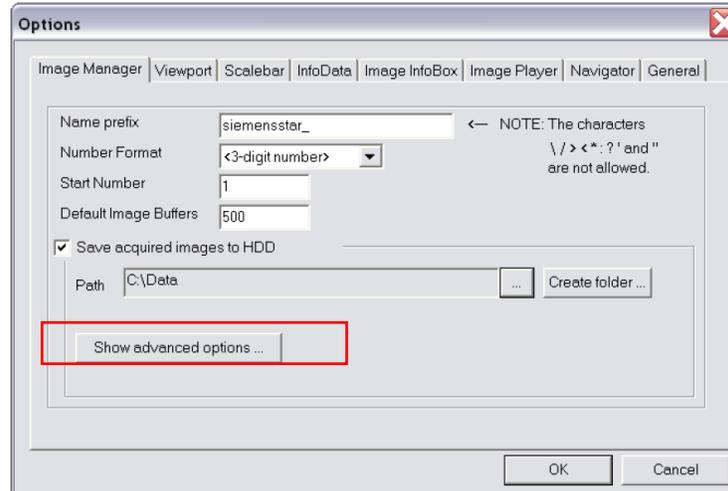
- ✓ Tools → options → image manager:

*Prefix name:* The image names are created out of the prefix first (this is a fix string) followed by a number.

*Number format:* The number can be a simple number (1, 2, 3, ...), a 2-digit number (01, 02, 03, ...), a 3-digit number (001, 002, 003, ...) or a 4-digit number (0001, 0002, ...). With each acquisition the image number is increased. The start number can be specified in this dialog too.

*Default Image Buffers:* Each folder in the image manager has a number of empty image buffers at creation time. The value of "Default Image Buffers" specifies exactly this number.

**During the training do not save acquired images to HDD:** the acquired images are stored as 16 bit TIFF files in the specified folder. Unselect the button with the ellipsis (...) to select the folder. It is also possible to create a new folder by clicking on the "Create folder ..." button



Be sure that the images are not being saved on the hard drive (HD)

### 3. Objective Lens Astigmatism Correction

✓ To use the camera, the illumination is spread until it covers an area slightly larger than the whole phosphor screen (turn the **BRIGHTNESS** knob clockwise) until the CUR DENS reaches values around **10 pA/cm<sup>2</sup>**.



Focusing the beam directly on the detector may cause permanent damages to the detector's scintillator.

✓ Press **F1** (right panel) to up the fluorescence screen.

✓ Click on the camera  icon (2k x 2k RS), exposure time (200 s). With these buttons it is possible to switch between camera formats with different exposure time settings.

✓ Press the "Continuous exposure" button  and acquire consecutive images;

✓ To visualize the FFT image, click in ;

An amorphous material will image as below. If properly adjust it will image .  
The astigmatism creates the asymmetry of the FFT. Once it is corrected, you will see concentric rings whose number will decrease as you approach to the right focus. Remember that this method works better if you are already close to the right focus condition.

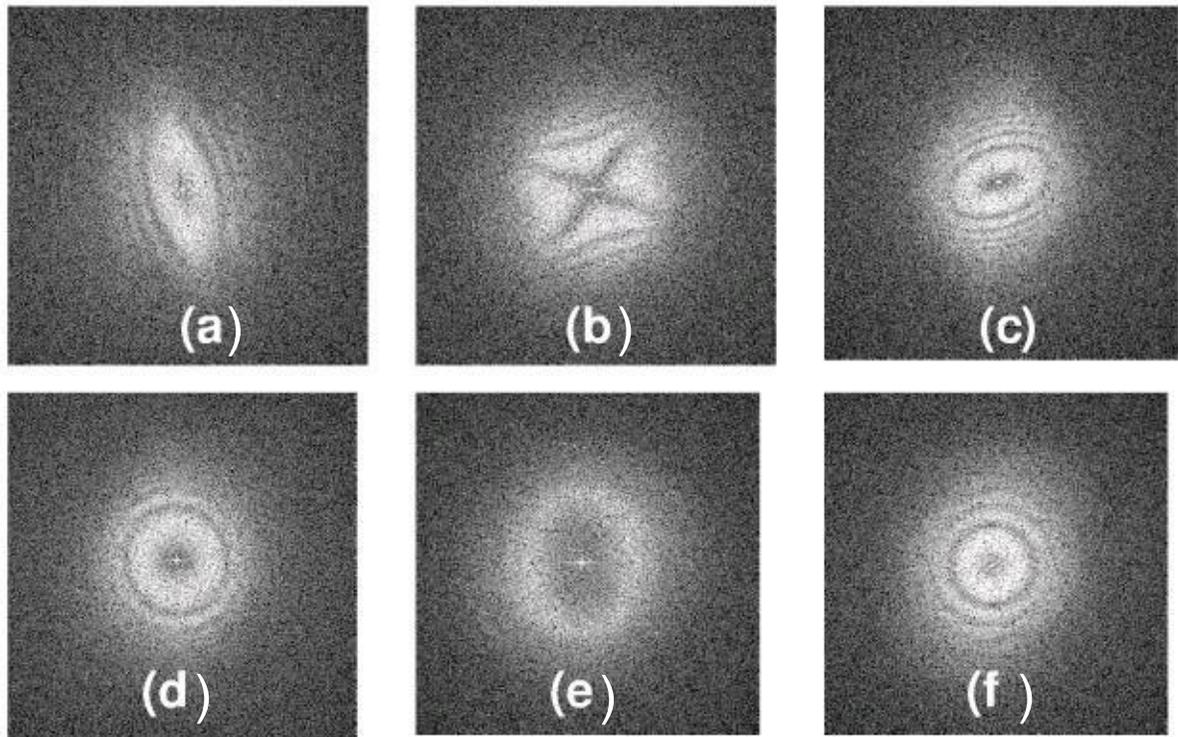


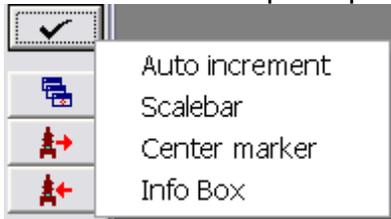
Figure 1: Image with strong astigmatism: (a) under-focus, (b) focus, and (c) over-focus. After almost fully correcting the astigmatism: (a) under-focus, (b) focus, and (c) over-focus

✓ Press  icon to stop the continuous exposure process before change the camera for acquire image.

#### 4. Acquiring Images

✓ Click on camera1  icon (4k), exposure time (600s).

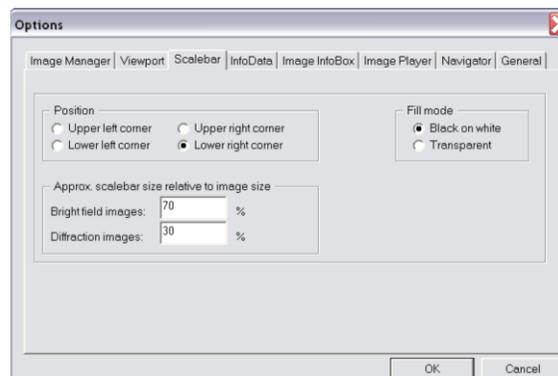
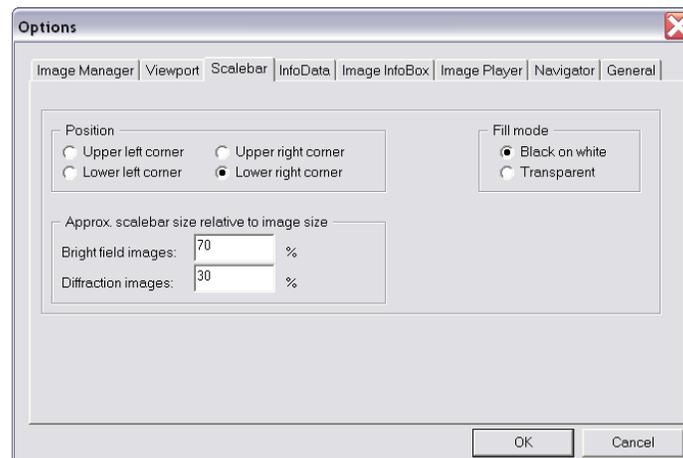
✓ Press the "Viewport options" button and it will open a popup menu.



- *Checked Auto increment:* each click on the exposure button will store the image in the next available image buffer in the image manager. If it is not checked, the image in the current image buffer will be overwritten without a warning.

- *Checked "Scale bar",* a scale bar is shown above the image data.

The position and appearance of the scale bar can be modified in EMMENU 4's option dialog. There are 4 different predefined positions to display the scale bar: upper left, upper right, lower left and lower right corner.



- ✓ The color bar displays the distribution of the virtual memory.   
Dark red: used virtual memory. Light green and dark green areas indicate the available memory. Light green: greatest contiguous part of free memory. If the available memory is too low, the dark red area becomes red. The yellow area indicates that the contiguous memory is very low. 

## Finishing your session (middle of the day)

### ✓ Turn off the filament

- Just turn off the filament. You can leave the TEM HT at **200kV**.
- Click on **FILAMENT OFF** on **HIGH VOLTAGE CONTROL** window. BEAM CURRENT must decrease gradually from **112  $\mu\text{A}$**  down to **102  $\mu\text{A}$**  (means that just HT is ON = 200 kV). Wait!!!

- ✓ TEM phosphor screen must be down (no beam going to the cameras).

- ✓ Remove the objective and the SAD apertures if you did use them.

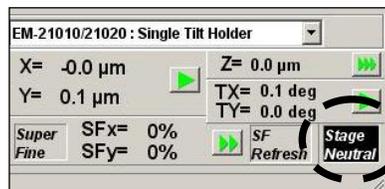
- ✓ CCD Camera (Gatan MSC794): Leave it on, but the **CAMERA** key must be down (position **OUT**).

### ✓ Removing Sample Holder (SH)



**Important: Before removing the SH, neutralize!**

- To neutralize: JEOLPC: TEMCON – **Double click** on black button **StageNeutral** (on right upper corner of software window) to neutralize SH position. Make sure sample shifts and tilts are **zero**.



- 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. **Read it 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.**

Before leaving **CHECK PLEAAASSE!!!!!!!!!!!!!!!!!!!!!!!!!!!!**

- **Acceleration Voltage (HT) is 200 kV**
- **Filament is OFF, Filament Control Speed on Normal Setup**
- **High Voltage (HT) is ON (Beam Current = 102  $\mu$ A)!!!**
- 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**.
- All the Apertures: **CL, EDS, OL, HC** and **SA** must be **OPEN (Removed)**
- **LCD Microscope Computer screen OFF.**
- **VACUUM IS OK.**

**Use the same setting for interrupt your session or  
exchange sample !!!**

## **Finishing your working session (end of the day)**

### ✓ **Turning Off the Filament:**

- Click on **FILAMENT OFF** on **HIGH VOLTAGE CONTROL** window.
- **BEAM CURRENT** must decrease gradually from **112  $\mu$ A** down to **102  $\mu$ A** (means that just HT is ON = 200 kV). Wait!!!

### ✓ **Turning Off the High Voltage:**

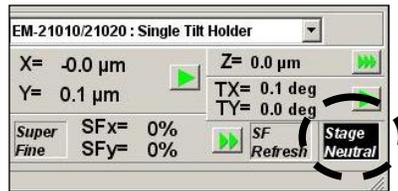
- **Verify the filament is effectively OFF**
- Click on **HT OFF** button on **HIGH VOLTAGE CONTROL** window. **BEAM CURRENT** must decrease gradually from **102  $\mu$ A** down to **ZERO** (HT = 0 kV) **BEAM CURRENT** must be **0  $\mu$ A**.
- Set the next user initial acceleration voltage to **160 kV**.

## ✓ Removing Sample Holder (SH)



**Important: Before removing the SH, neutralize!**

- To neutralize: JEOLPC: TEMCON – **Double click** on black button **StageNeutral** (on right upper corner of software window) to neutralize SH position. Make sure sample shifts and tilts are **zero**.



- 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. **Read it 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 the following information in the Microscope Notebook:  
**Filament: ????????????** **Power:**  
**ΔFil: ??????????** (Number of Hours of Filament Used)

Before leaving **CHECK PLEAAASSE!!!!!!!!!!!!!!!!!!!!!!!!!!!!**

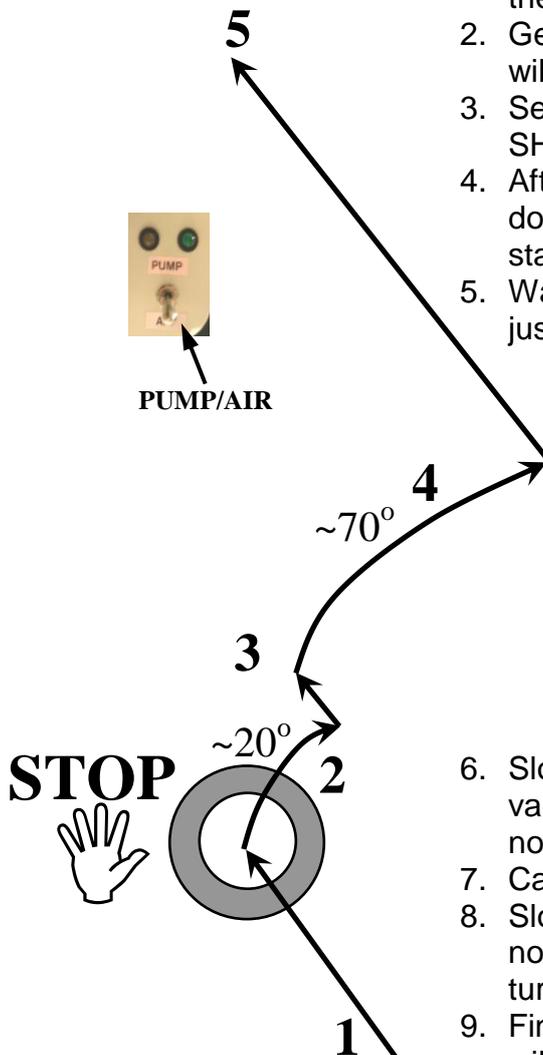
- **Acceleration Voltage (HT)** must be set to **160 kV**
- **Filament** is **OFF**, **Filament Control Speed** on **Degas Setup**
- **High Voltage (HT)** is **OFF (Beam Current = 0 μA)**
- TEM phosphor screen is down.
- TEM viewing port is covered.
- TEM telescope eyepiece is capped.
- Optical microscope is covered (table).
- **MAG 1** is on, and magnification set to **50k**.
- **PANEL LIGHT** is off.
- All the Apertures: **CL, EDS, OL, HC** and **SA** must be **OPEN (Removed)**
- **LCD** Microscope Computer screen **OFF**.
- TEM computer screen is off (contrast and brightness to the minimum).
- **VACUUM IS OK.**

## Inserting the Sample Holder into TEM Column

1. To insert the SH align the guide pin with the notch on the goniometer, located at 9 o'clock. Insert the SH.
2. Gently 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 several minutes till Specimen/PIG4 indicator is just below 32  $\mu$ A (ideally 30  $\mu$ A).



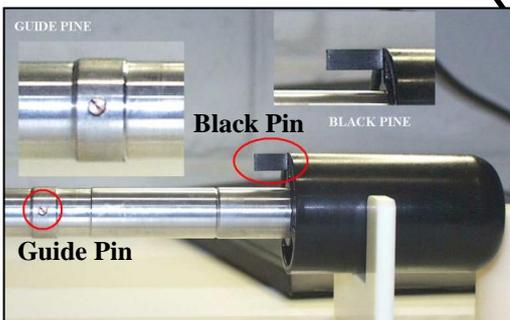
PUMP/AIR



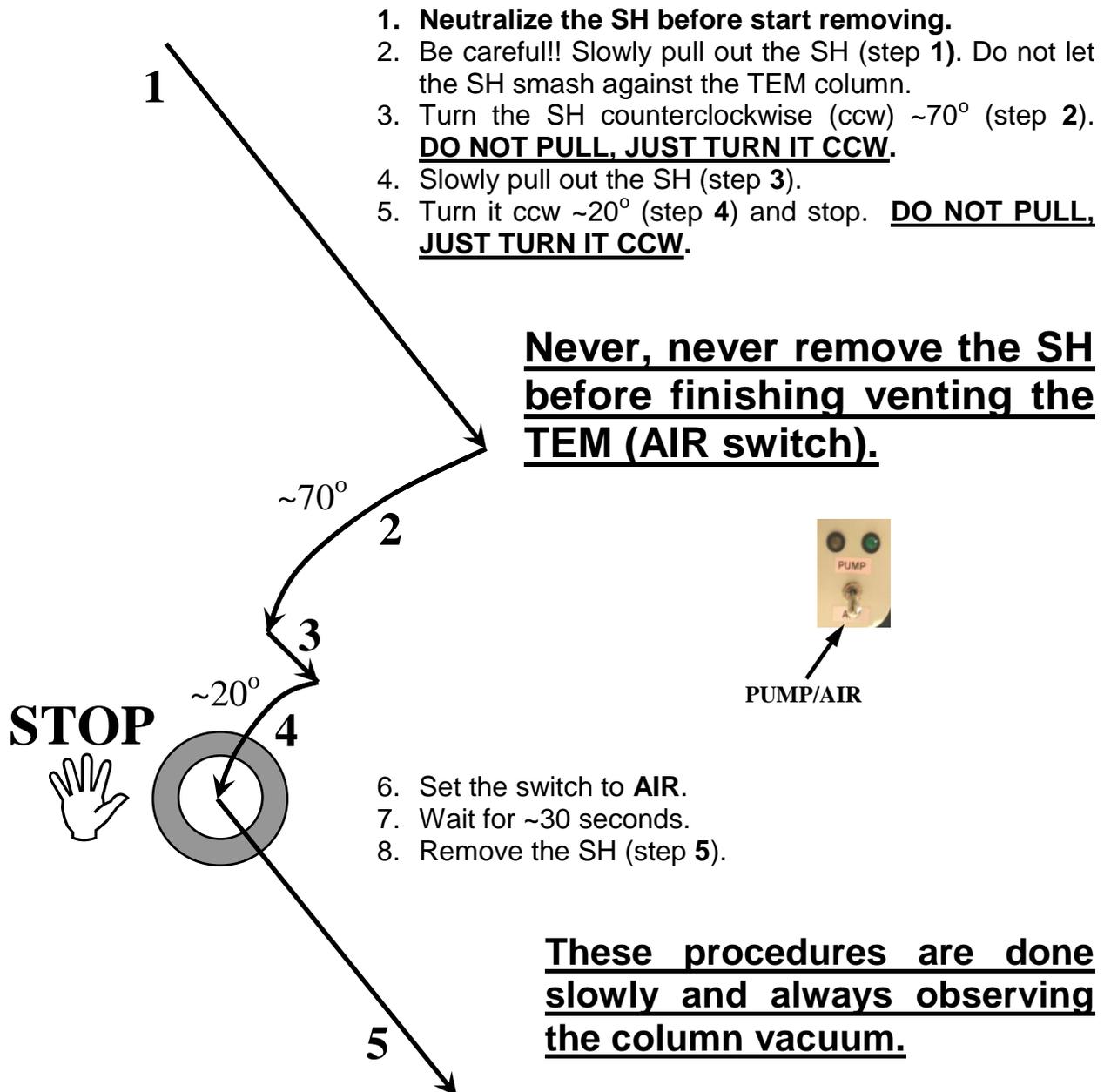
**Never, never turn the SH clockwise before finishing the PUMP step.**

6. Slowly turn the SH clockwise  $\sim 20^\circ$  (step 2). The vacuum will pull the SH after completing this step. Do not let it go and smash into the TEM.
7. Carefully hold the SH and insert it (step 3).
8. Slowly, turn the SH clockwise  $\sim 70^\circ$  (step 4). Again, do not let it go and smash into the TEM after finishing turning the SH.
9. 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  $\sim 3$ mm.

**All the steps have be done slowly and always observing the column vacuum. If after inserting the SH the vacuum does not recover after few minutes, look for the staff.**



## 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)  $\sim 70^\circ$  (step 2). **DO NOT PULL, JUST TURN IT CCW.**
4. Slowly pull out the SH (step 3).
5. Turn it ccw  $\sim 20^\circ$  (step 4) and stop. **DO NOT PULL, JUST TURN IT CCW.**

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

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