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I. POLICIES AND INTRODUCTION

Reservations

JEM-2100F reservations are made using the NUCore online reservation system. Start your reservation before you begin using the instrument. When your session is complete, be sure to end your reservation in NUCore. If you need extra time on the microscope, we recommend 'extending' your original reservation, rather than making an additional reservation.

There is a hardware control system on the JEM-2100F, so the system will not function unless you are logged in to NUcore. Tampering with or disabling the hardware control may result in revocation of your EPIC privileges.

Saving Your Data

During your session, you may store image data to your own folder on the local drive. But please transfer your data to the NUANCE TEM File Server. The TEM server is accessible through a computer in the lab. You can transfer your data from the computer outside the microscope room to a USB, etc.

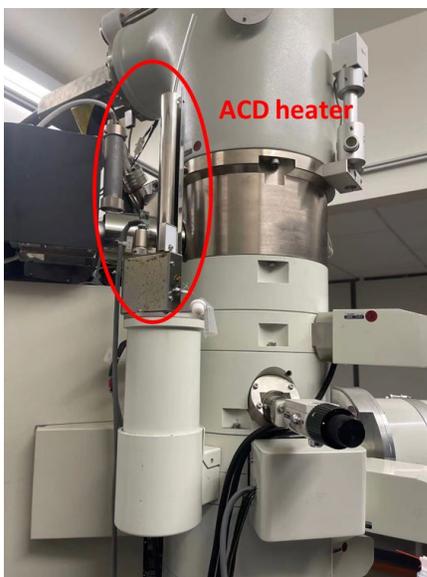
JEM-2100F Rules

1. Please wear gloves when handling any component and samples that will go into the TEM column.
2. Do not install any softwares onto the JEOL PC, Gatan PC, EDS PC and Protochip PC without the manager's agreement.
3. Do not insert any flash drives into any microscope computers.
4. The microscope is usually operated at 200 kV.

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5. Do not use any third-party holders without the manager's permission.

II. START-UP

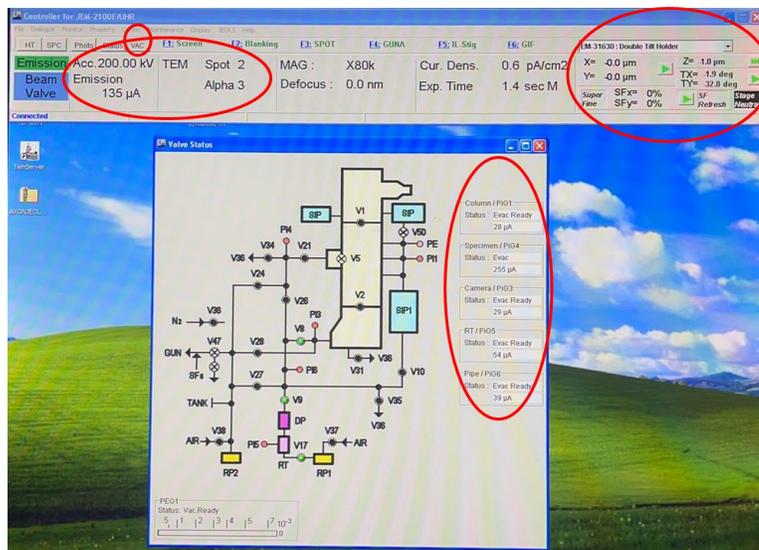
1. Log in through NUcore. (<https://nucore.northwestern.edu/facilities/nuance>). Note that the monitors of Gatan PC will not be on without turning on the relay control in your NUcore system.
2. Take out ACD heater if it is inserted. Fill in liquid N2 using funnel, wait 1-3 minutes till boiling then top off LN2 again. **Note that full tank LN2 will last 8 hours.**



3. Check conditions of JEOL 2100F:
 - a. Left-bottom rack: Vacuum meter $<3 \times 10^{-5}$ Pa with blue scale.



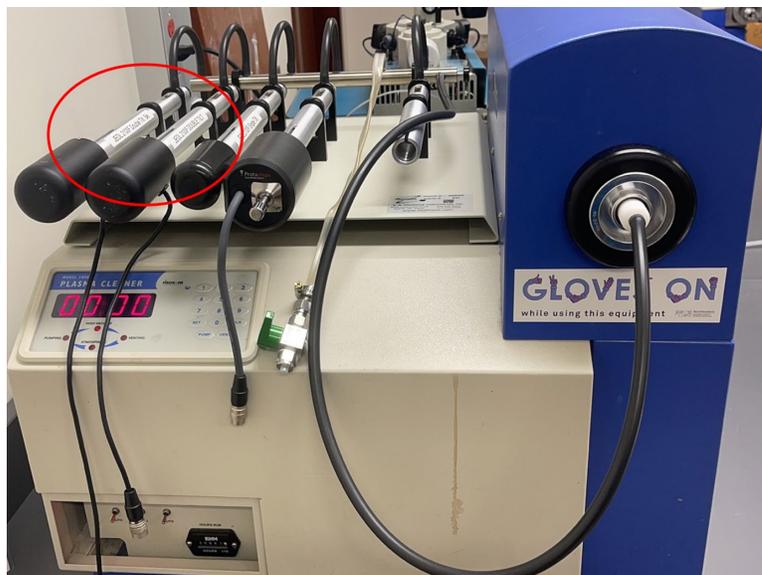
- b. JEOL PC (right): Vacuum System (TEMCON – upper left corner – VAC), V1=OFF, V2=OFF, PIG1<35, PIG4~255, PIG3<45, PIG5<65, PIG6<40.



- c. JEOL PC: Acc.= 200 kV, Emission \sim 140 μ A, TEM Spot=1-3, Alpha=3, X=Y=Z=0, TX=0 (TY doesn't matter)

III. LOADING SAMPLES

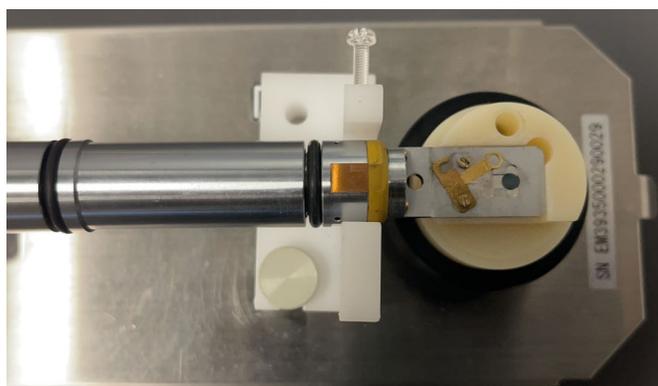
1. Refer the operation manual of Fischione 1020 pump station to take out the needed single/double holder.



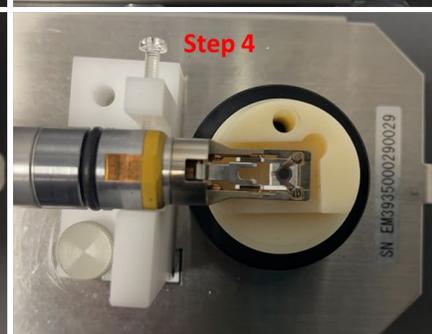
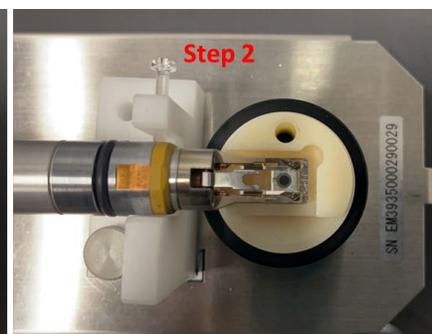
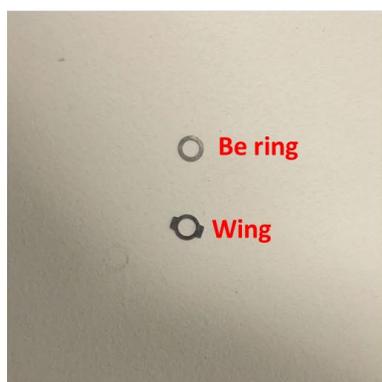
2. Put holder on sample loading station, with appropriate holder support lying underneath hold tip.



3. For single tilt holder, loosen two screws TWO turns, rotate the cover plate away and mount specimen with film side UP.

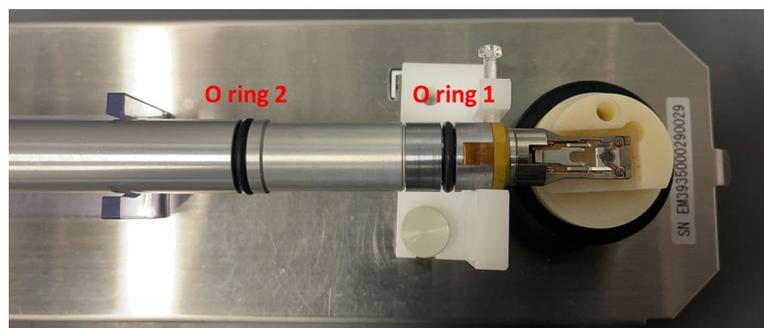


For double tilt holder, loosen two screws TWO turns, rotate the two plate-clamping fingers and mount sample facing DOWN.



NEVER OVERTIGHTEN THE SCREWS!!!

4. Check O-ring and clean with duster if necessary.



5. Align holder guide pin with the guide groove on the goniometer, push till it stops (DO NOT TURN!).

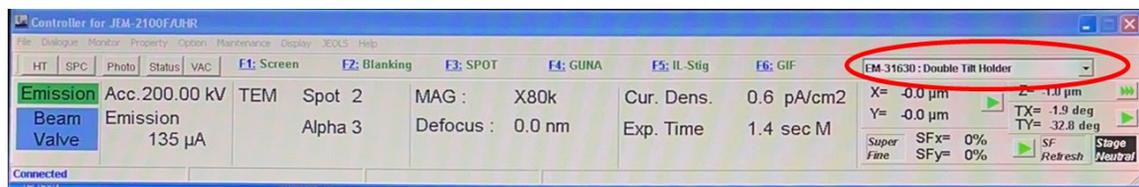
6. Keep pushing the holder in position, pull PUMP/AIR switch on goniometer and turn it up to PUMP. For double tilt holder, put the cable across the goniometer cover to prevent rotation of sample holder due to cable weight.

7. Wait (DO NOT TURN!) until the **green** LED on goniometer turns on.

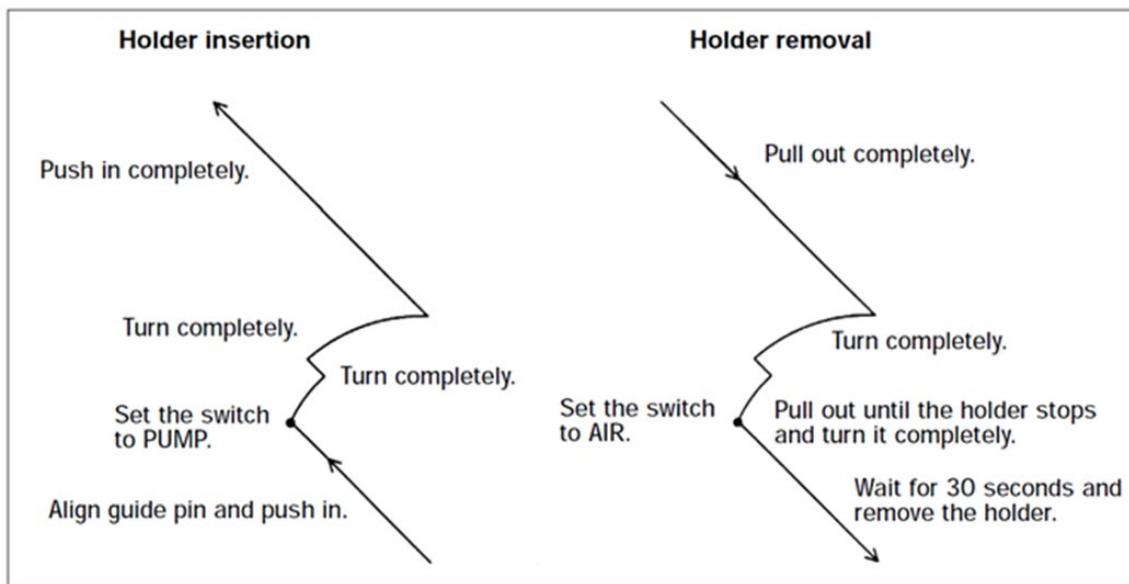
8. Turn holder clockwise and insert specimen holder into the goniometer (follow the chart on TEM column). Try to insert smoothly and gently. Never use force side ways!

9. Wait till column pressure is below 1.3×10^{-5} Pa (lower-left panel) and PIG4 is below 35.

10. JEOL PC: TEMCON – upper right corner – select “EM-21010/21020” for single tilt holder, 31630 for double tilt holder, or 31640 for low background Be tilting holder.



***Please refer the following schematic for holder insertion and removal. When you push/pull the holder, do not rotate the holder. When you rotate the holder, do not push/pull it.**



IV. TEM ALIGNMENT AND IMAGING

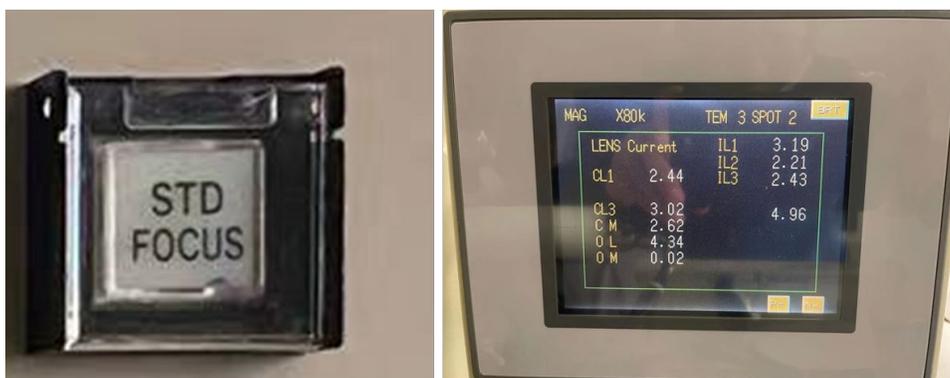
1. Press Beam button (left panel) to open the gun valve.



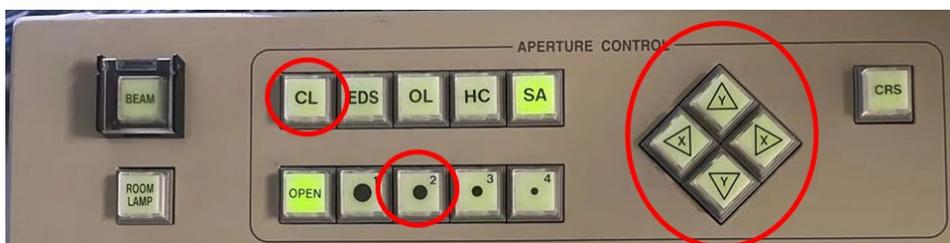
2. Make sure the operation mode is TEM MAG mode. Decrease mag using **MAG/CAM L** button and move sample around if cannot see beam. You can even change to **LOW MAG mode** to find the sample.



3. Press **STD focus** (right panel) and make sure OL current is 4.31 (CRT at right side of column).



4. Alignment of condenser aperture. Note that there are many **CRS** buttons on left/right panels. When **CRS** button is activated, the changing step will become larger.



- a. Insert the suitable condenser aperture **CL** #1-2 (left panel, Aperture Controller).
- b. Adjust **BRIGHTNESS** knob to get the smallest beam, use **SHIFT-X** & **SHIFT-Y** knobs to center the beam;



- c. Spread beam to a large disk, use aperture controller **ARROW** buttons to center the beam again;
- d. Repeat steps (b) and (c) until beam center does not change (beam concentrically shrink/expand).
- 5. Find sample. Smallest beam, lower mag (~20kx) adjust Z-height button(right panel) to minimize clouds around beam.

Note: Move sample SLOWLY when trying to find sample in low-mag mode! You may turn on PIEZO when you want to move sample under very high magnification. Remember to turn it off after use!



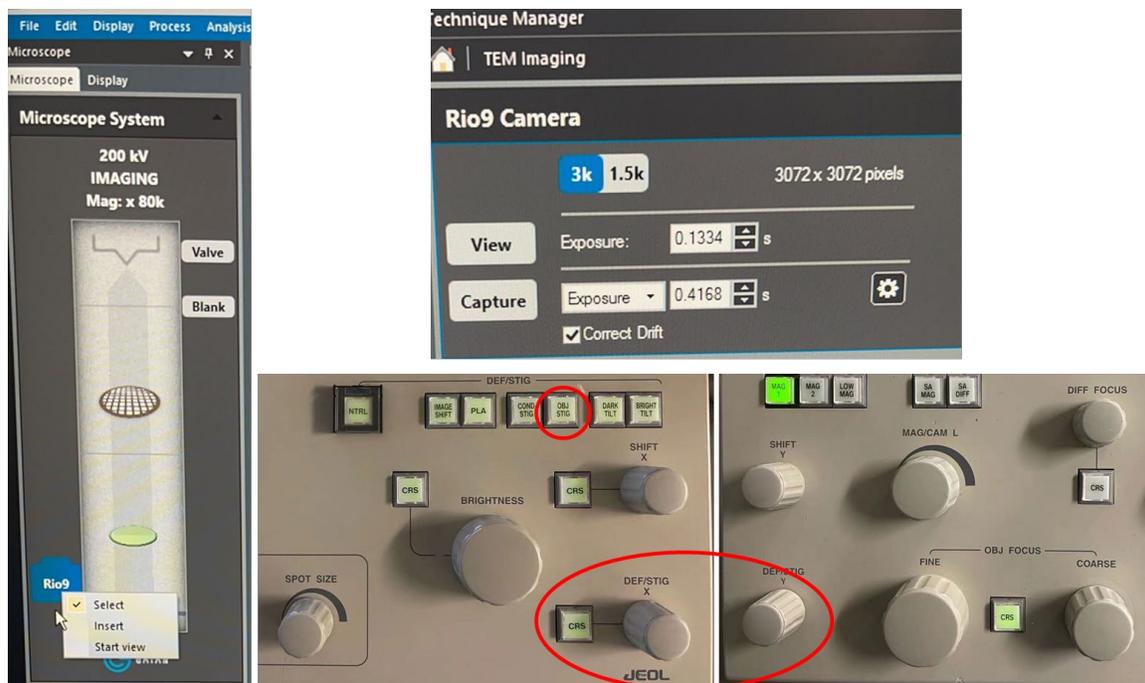
- 6. Correct condenser stigmatism: press **COND STIG** and adjust **DEF-X** & **DEF-Y** knobs to make beam round.



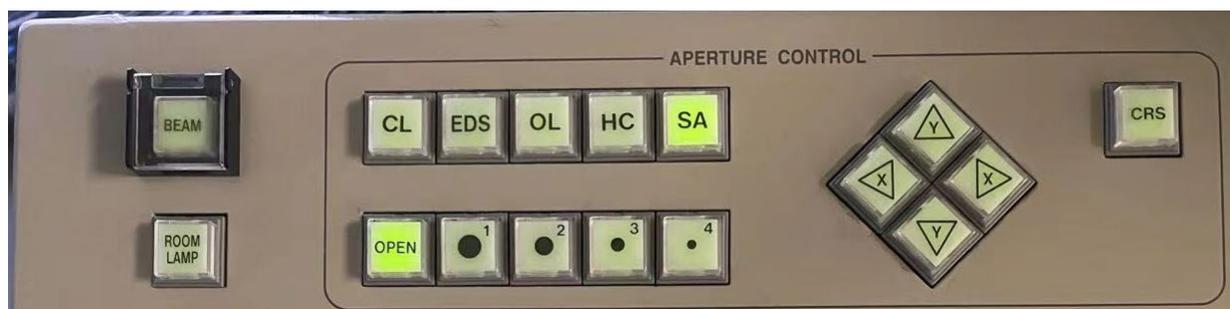
7. Check/Align Gun Tilt: at 40kx, press **F3** “Anode Wobbler”, press **F4** “GUN Align”, adjust **DEFLECTOR-X** & **DEFLECTOR-Y** knobs to get a uniformly expanded beam.
8. Check/Align Gun Shift: at 40kx, adjust **SPOT SIZE** knob to Spot 1, press **F4** “GUN Align” and adjust **SHIFT-X** & **SHIFT-Y** to center the beam; then **SPOT SIZE** knob to Spot 5, depress **F4** “GUN Align” and adjust **SHIFT-X** & **SHIFT-Y** knobs to center the beam; repeat until beam does not move.
9. Eucentric height: double press **STD FOCUS** (right panel) to standard defocus, press **IMAGE-X** or **IMAGE-Y** WOBBLER (right panel) and adjust **Z-UP/DOWN** buttons (stage controller) to minimize vibration. **This step is used to focus your sample.**
10. Align Beam Tilt (Voltage Center): press **HT WOBBLER** (right panel), press **Bright tilt** (left panel) and adjust **DEF-X** & **DEF-Y** knobs to minimize lateral translation (image only moves in and out).



11. Objective stigmatism: acquire an image of amorphous region (>500 kx) in GMS3 (Gatan PC, middle) using RIO camera. You need to insert the camera first. Click Process >> Live >> FFT, press **OBJ STIG** and adjust **DEF-X** & **DEF-Y** knobs to make the FFT pattern round.



12. Objective/HC aperture: press **OL/HC** to insert high-contrast aperture to enhance contrast if needed.



13. Image the sample: find a region of interest, adjust **OBJ FOCUS** and/or **Z-UP/DOWN** to focus sample, select proper exposure time and image size, record image.

14. Save images: File >> Save/Save As, in .dm3 or .dm4 format, temporarily save files in \Documents\Users\ folder, and move to NUANCE Data Server (Z:\ Create Folder by NetID) and delete local files immediately after your session.

V. SELECTED AREA DIFFRACTION (SAD)

1. Press **SA MAG** (right panel), center region of interest to the center of screen. Spread beam by **BRIGHTNESS** knob. You can use smaller condenser aperture CL1 #3 or #4 to reduce the beam intensity.



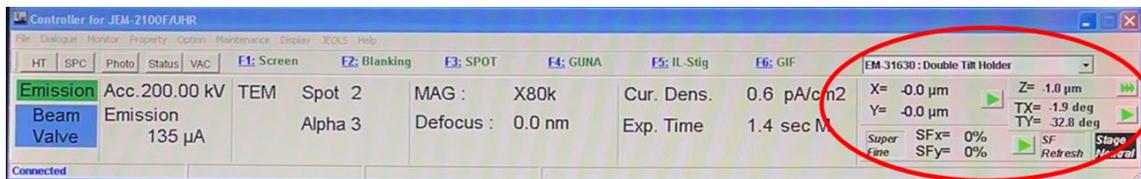
2. Insert selected area aperture: press **SA** and desired size number (left panel). You need to align the selected aperture well with the beam by clicking the UP/DOWN/LEFT/RIGHT arrows.
3. Press **SA DIFF** button (right panel) and adjust **DIFF FOCUS** knob to get the smallest transmitted spot.
4. Adjust **MAG/CAM L** to change camera length (from 10 cm to 2 m).
5. If the center transmitted spot is too strong, you can insert the beam stopper manually to block the transmitted spots. If needed, press **PL ALIGN** button (left panel) and adjust **DEF-X & DEF-Y** knobs to center the transmitted spot to the beam stopper.
6. Press **F1** on right panel to lift up the screen. Use shortest exposure time (0.0399s) to take SAD on RIO camera. **Over-exposure may damage camera!**

VI. CHANGE SPECIMEN & SHUTDOWN

1. Remove any aperture that is inserted during your session.
2. Turn off **BEAM** (left panel).



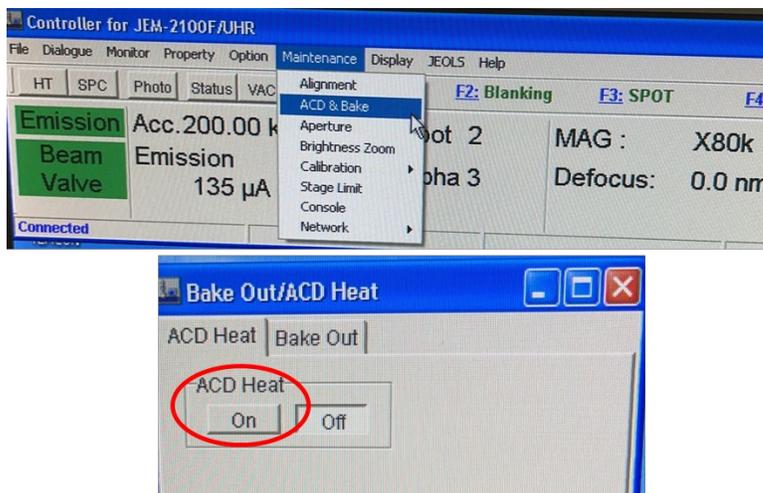
3. Double click **Stage Neutral** button to neutralize sample position. Make sure **sample position (X/Y/Z) and tilts (TX/TY) are all ZERO**, then remove specimen holder from the microscope. **Removing the holder is a reverse predure of the inserting process.**



4. Load new specimen if you want, or store the holder in Pumping Station.
5. If you are the LAST USER of the day (nobody after you in next 12 hours), follow this procedure:
 - a. Plug in ACD Heater.



- b. Pull out the ACD&Bake page (TEMCON---Maintenance---ACD&Bake) and enable ACD heat.



6. Logoff your session in NUcore system.