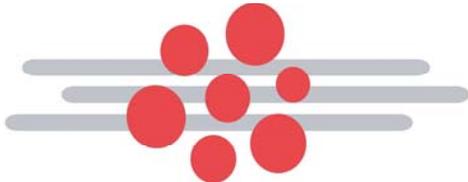


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|  <p style="text-align: center;">Center for Functional Nanomaterials Brookhaven National Laboratory</p> | NUMBER |
| | REVISION 0.1 |
| Subject: Helios SEM/FIB Basic Instructions | DATE 09/12/2014 |
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This SOP does not replace the detailed training sessions you should receive before being allowed to use the machine independently.

It is your responsibility to avoid any dangerous situations and to understand and follow these instructions. If we observe any negligence from your part, you will not be allowed to use the system anymore.

Under any doubt of any kind contact Fernando Camino (x7606) or Aaron Stein (x3527). If neither of us is around, **STOP YOUR WORK** until we show up. The cost of parts, repairs or tool downtime is certainly worth a day of your project.

STARTING

- 1) Log on your session in the Facility Online Manager (FOM) software (icon on the desktop of the DATA computer on the mobile cart). After a correct login, the Helios computer monitor should automatically turn on. If it does not, **STOP YOUR WORK AND CONTACT A STAFF MEMBER. Never modify any physical connections or push any system buttons. Doing so is unsafe for you and the instrument. Any transgression of this rule will automatically ban you from using the instrument.**
- 2) Move the Helios computer mouse to wake up the Helios computer monitor (in case in went into sleep mode). You should encounter the Helios user interface logged as “supervisor”. In case someone accidentally logged off, please log in using the credentials displayed on a label on the Helios monitor computer. If the user interface is not active, hit the "FeiSystemControl" icon on the desktop of Helios computer to open the microscope server window, hit "Start UI" to start user interface (UI); then log in using the credentials for the supervisor account.

3) Loading your sample:

- Hit "Vent" in beam page of UI. It takes about 5 min to be able to open the chamber. Make sure sample holder is far from SEM final lens. For this you need to see a "live" CCD camera image of the chamber.
- Open chamber door slowly, while observing chamber image to make sure no accidents occur.
- Figures 1 and 2 show the stub holder mounted on the Helios stage. It has a piece with three holes for stubs. Only use the hole shown in Figure 1. This hole has a corresponding setscrew to hold the stub (see Figure 2).

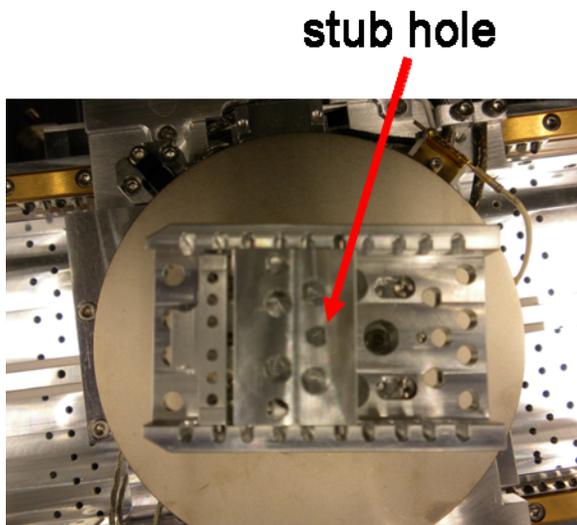


Figure 1. Top view of stub holder.

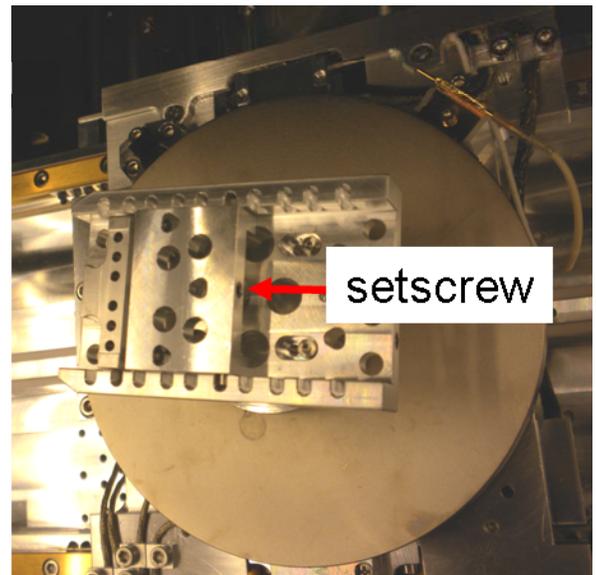


Figure 2. Angle view of stub holder.

- To facilitate adjusting the setscrew (use very little force), you may need to rotate the stage so that the setscrew is to your right when you stand in front of the chamber. You may also need to bring the stage closer to you to make it easier to place the stub and tight the setscrew. You move the stage only from the User Interface (see figure 3). If you have any questions about this step, do not hesitate to call Fernando or Aaron.

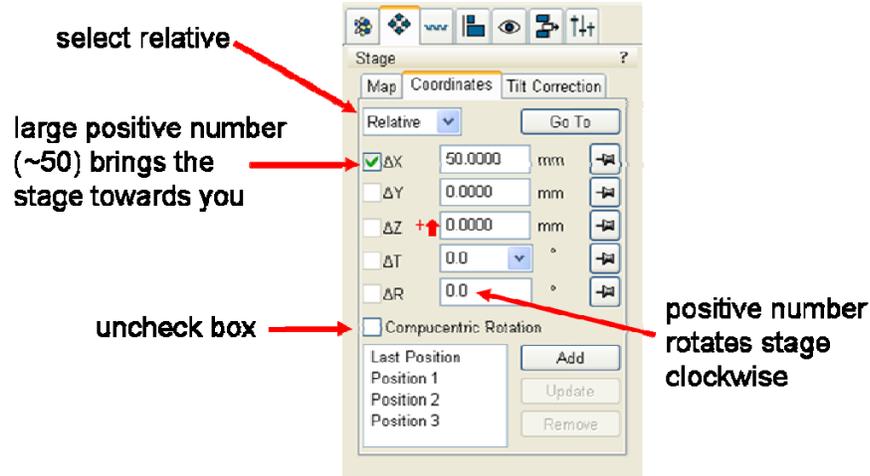


Figure 3. User interface settings for moving the stage in order to facilitate access to stub hole setscrew.

- To rotate the setscrew to secure or to remove your stub, use the hex wrench placed inside a plastic box next to the Helios tool. Do not use any other wrench!
- The holder has another larger hex screw (opposite to the stub setscrew, see figure 4). This needs to be always tight (you should not need to tighten it, it uses a different wrench). In case it is loose, please call Fernando or Aaron.

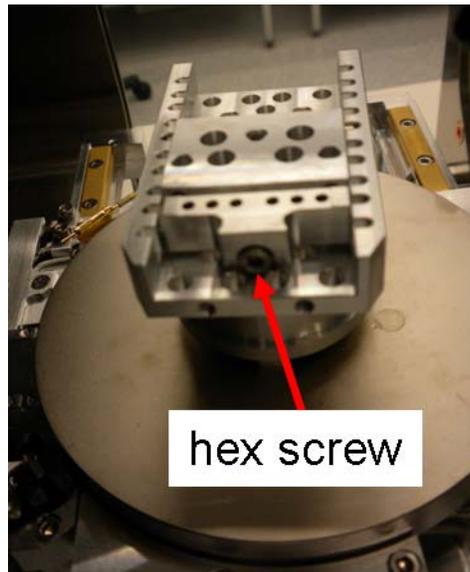


Figure 4. Hex screw that holds stub holder in place. You do not need to tighten this screw.

- There is no height adjustment with this new holder. **Samples should be less than 3-mm thick. If you have thicker samples, first contact Fernando Camino or Aaron Stein. Always check the height using the sample height gauge (inside plastic box next to Helios).** The highest point of your sample should be at the same height or below the top surface of the gauge (ignore max. or min. lines in the height tool). **If your sample is above, STOP your work and call Fernando or Aaron.**

- Be very careful when you close the chamber door. Always look at the CCD camera quadrant to detect any possibility of crashing the objective lens.
- Hit "Pump" in UI. Push the door close for the first few seconds until mechanical pump stops making loud sound. Wait until chamber color in bottom SEM icon turns green. It takes about 5 min for this.

NOTE.- Only certain users are allowed to replace the general use holder with another one that suits their projects. Once finished, these users should put back the general use holder, taking care to line up the alignment marks as shown in Fig. 4-1. Also, the side tightening piece should show a black dot on the top surface (see Fig. 4-1). If the position of any of these marks is not correct please contact Fernando. Correct alignment is needed for correct compucentric rotation operation and to assure that the (0, 0) coordinate lies approximately at the center of the stub.

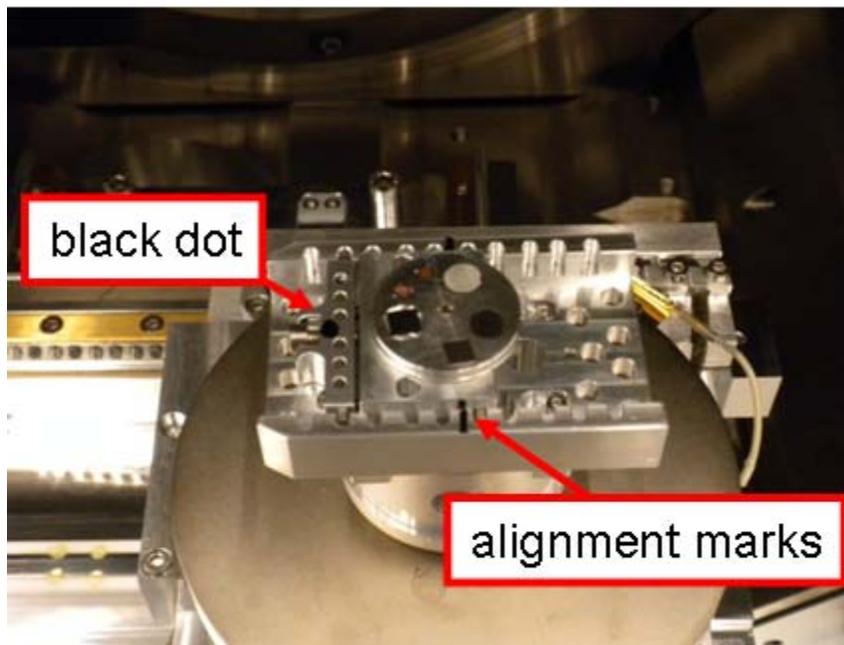


Figure 4-1. Correct position of alignment and reference marks on general use holder.

- 4) If only using SEM, then hit "Beam On" button for e-beam only. If also using FIB, hit "Beam On" for FIB too.
- 5) Linking Z stage coordinate to the Free Working Distance (Link Z to FWD): This is a most critical part of the tool operation. Before any stage movement in the Z-direction, you should tell the system to update the Z stage coordinate to the actual distance between the final lens and your sample, which is called free working distance (FWD). This updating operation is called "Link Z to FWD".

- In QUAD 1 (e-beam) window, locate a good feature in your sample and focus it. You move the stage by pressing down the mouse thumbwheel (you'll see a yellow dot on QUAD 1) and by moving the mouse. Setting $x=y=0$ using absolute stage coordinates will place the center of the stub approximately at the center of the SEM field of view. Focus on your sample. You should be able to recognize a mark or feature on your sample to be sure it is the surface of your sample. If you have a pattern this will do it. If not, make scratches at two corners, at least, to be able to correlate the distance between the marks and the size of your sample (you should also know the approx. dimensions of your sample). If you are in doubt, **STOP your work and contact Fernando or Aaron.**
 - For regular samples (less than 1-mm thick), after focusing on your sample, the working distance (WD) should fall around 9 mm. Thicker samples can have WD less than 9 mm, but never less than 6 mm. **If you get a WD outside the 6-10 mm range, most likely you are not focusing on your sample. THIS IS A POTENTIALLY DANGEROUS SITUATION. DO NOT PROCEED. Contact Fernando or Aaron at this point.**
 - Once the feature is focused, link the stage Z coordinate to the working distance by hitting the "link Z to FWD" icon on top icon bar. This action will pass the FWD value to the Z stage coordinate.
- 6) Move the stage to your desired FWD, making sure you keep the Z stage coordinate updated (linked) after any Z stage movement.
 - 7) If doing FIB work, understand the procedure to find the eucentric point. If in doubt, **STOP your work and contact Fernando or Aaron.**
 - 8) Never attempt to tilt the sample if you have not first correctly determined the eucentric point.

FINISHING:

- 1) Hit "Vent". Remove sample and pump chamber back down as explained in step 3 of the previous section. **YOU NEED TO WAIT UNTIL THE MAIN CHAMBER REACHES A WORKING VACUUM** (chamber icon turns green). In rare occasions, the pump process times out (chamber icon remains "gray" and the "PUMP" button becomes enabled). If this happens, please pump the chamber a second time and if this still doesn't pump the main chamber, put a "system down" sign on the Helios desk and inform Fernando.
- 2) THERE IS NO NEED TO LOG OFF FROM THE USER INTERFACE.
- 3) Log off from FOM in the DATA computer (monitor of Helios computer should turn off) and make sure you have completed the tasks listed in the "Logoff reminder items".
- 4) Make sure the Helios room is left clean and in order.

APPENDIX I. HELIOS SYSTEM DATA STORAGE GUIDELINES

The objective of these guidelines is to minimize cyber security threats to the Helios system.

A) EXTREMELY IMPORTANT

- No external data storage device is allowed to be connected to the Support and Helios computers. This includes USB memories, CDs, DVDs, etc. External storage devices and internet usage is only allowed in the DATA computer (computer on mobile cart).

B) HOW TO SAVE AND TRANSFER DATA FILES

- 1) From the Helios or Support computers, you can save files into your own folder in the DATA computer (C:\Helios Data\Your_Folder) by opening the shortcut “Helios Data” found in the desktop of the Helios or Support computers.
- 2) You can copy your data from the DATA computer (click on the desktop icon “Helios Data”) into any storage device. You can also use the internet to transfer your files.

C) DATA HOUSEKEEPING

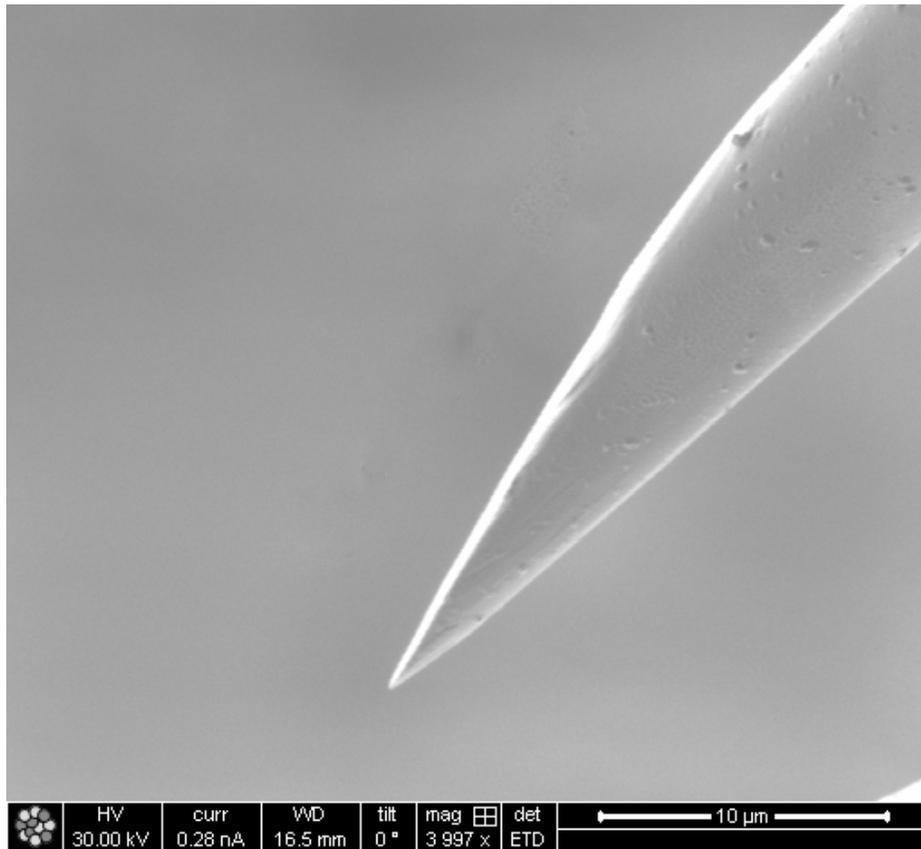
- 1) Once you have successfully transferred your files, you are supposed to erase them from the DATA computer to free up space.
- 2) At the beginning of a new year, if storage space is becoming an issue, we will remind Users to clean their directories. Starting the first week of February, **ALL DATA OF PREVIOUS YEAR WILL BE DELETED!**

D) IN CASE OF A PROBLEM

Either due to network problems or cyber security issues, sometimes there might be a lack of communication between computers in the Helios system. If you cannot save images in the DATA computer, save your data in a folder with your name on the desktop of the Helios or Support computers and let Fernando know of the problem. Once the issue is resolved, Fernando will copy these folders to your respective folders in the DATA computer. **Do not use any USB ports to get your data out from Support or Helios computers.**

APPENDIX II. GUIDELINES FOR MICROMANIPULATOR (OMNIPROBE) USERS

- 1) You must leave the tip sharpened for the next user. The quality of the tip should be comparable to the one shown in the image below (courtesy of Kim Kisslinger).
- 2) Take an ion beam image of the tip close to the eucentric height using a magnification of 4000x (see image below).
- 3) Save the tip image in the folder "Omniprobe" located in the data folder of the DATA computer. Use the format: YYYY-MM-DD-lastname (e.g., 2011-11-15-Camino.tif).
- 4) **DO NOT FORGET** to bring the tip out to the edge of the circular aperture before fully retracting the tip using the pneumatic valve. Serious problems can occur if you do not follow this step. You all have been trained in this procedure, however, if you have any doubts, please do not hesitate to contact me.
- 5) Sometimes, it would be impossible to sharpen the tip to a good quality, for example, after a crash. In these instances: a) take an tip image and save it as described above. b) Leave a comment in the LabView log in software letting users know about the bad tip. c) Let Fernando know about this so that the tip is replaced.



APPENDIX III. SESSION CANCELATION POLICY

- 1) If you cancel a session within 4 days of the reserved time, you need to send an email to Fernando Camino explaining the reason for the cancellation. If no email is sent or if the explanation is unfounded, this cancellation will count as an “unjustified cancellation”.
- 2) Only one unjustified cancellation is allowed per cycle. At the second unjustified cancellation, tool access will be denied for the remaining of the current cycle.