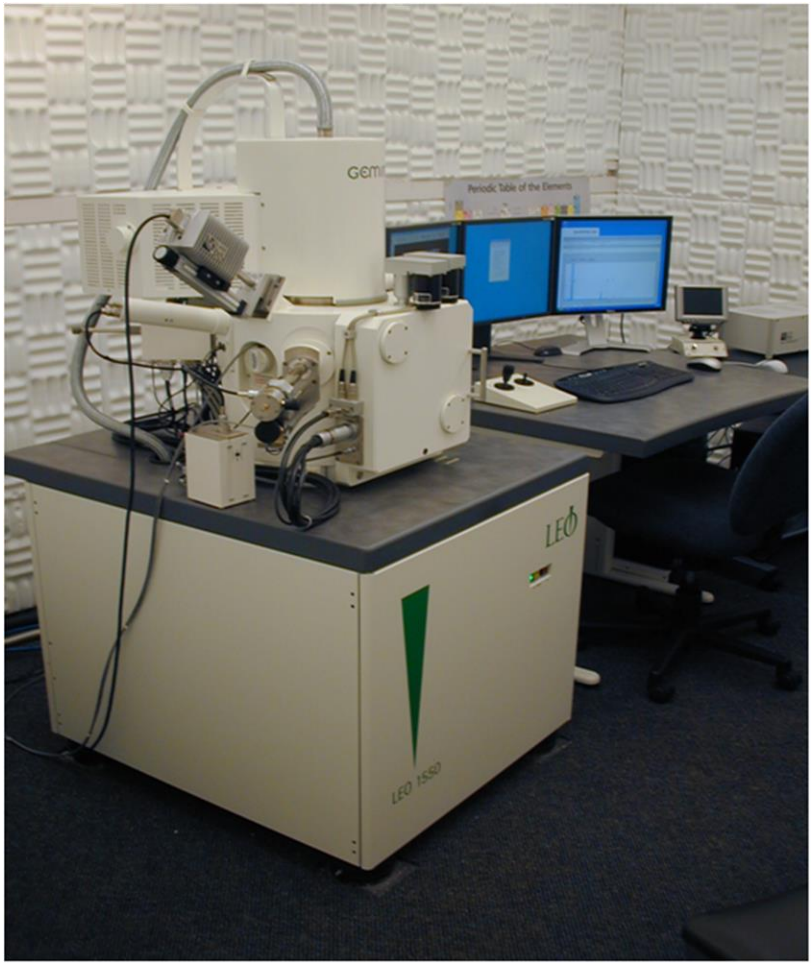
Keck SEM

part of the
CCMR EM Facility

Operating Manual

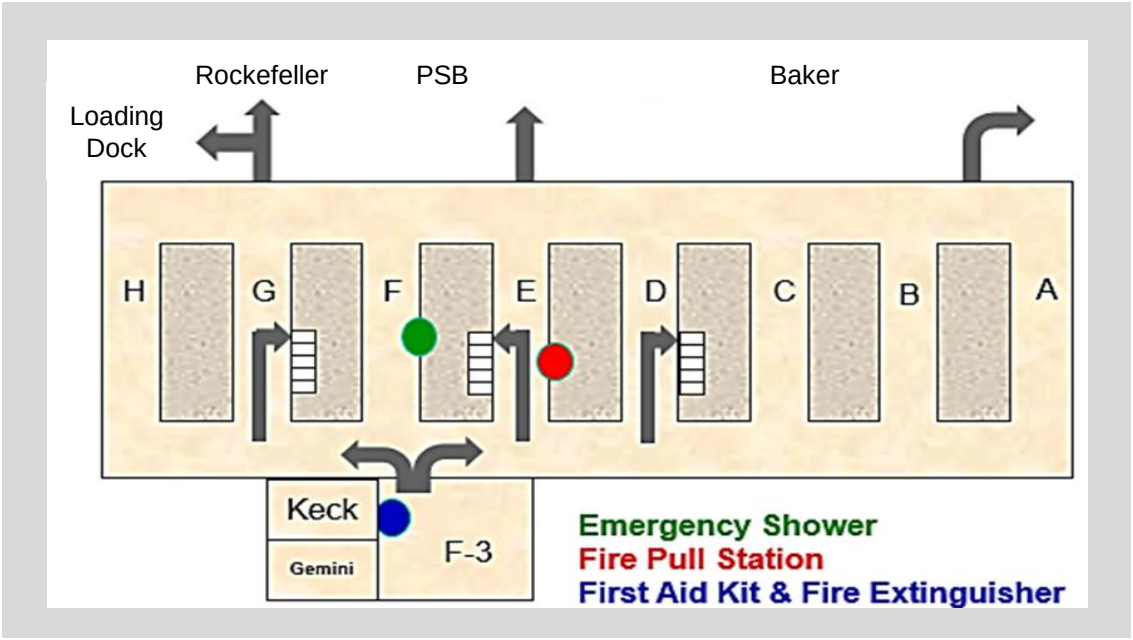


Laboratory Safety

- Users are required to have passed Cornell's on-line laboratory safety training courses specific to this lab before they can use the Keck SEM.
- Users must fill out a CCMR Shared Facility Access Form.
- Do not bring any chemicals or hazardous samples into the lab.
- Take specimens, stubs, and raw material with you when you leave.
- Isopropanol and Aero-Duster are supplied for your use. Wear safety glasses and read the appropriate MSDS before using them.

Users failing to follow safety rules will be denied access to the lab.

In case of fire leave immediately and close the door behind you. Do not use the elevators.



Mick's Golden Rule

If ANYTHING does not even SEEM right, leave everything as it is and call Mick (607-592-5217). If Mick cannot be reached by phone, then use text or email.

Laboratory Policies

Do not attempt to repair or remove ANY part of the microscope.

Do not add any software or hardware to the computer.

Flash Drives (Memory Sticks) are forbidden.
Data MUST be transferred via Cornell DropBox.

Do not change the room temperature or bring food or drink into F-3.

Wear gloves when touching the shuttles and specimens.

All samples loaded at the same time must be dry and the same height.

If you sign up and then cannot use your time, cancel your time via CCMR-FOM or contact Mick at least one weekday before your scheduled time.

Failure to show up without canceling at least 24 hours in advance, unless due to illness or other emergency, will result in use charges.

If you are more than 30 minutes late, then the microscope becomes available to other users.

If you feel sick, please reschedule your session. You will not be charged if you stay home due to illness.

Data Storage

Data storage and safety is not guaranteed – users are responsible for their data and must copy their data in a timely fashion.

Data will only be kept on the hard drive for one year. Periodically the hard drive will be erased, leaving only one years' data there.

How to acknowledge CCMR in publications

When research is published using data from this microscope, the facility and the grant number must be acknowledged:

"This work made use of the electron microscopy facility of the Cornell Center for Materials Research (CCMR) with support from the National Science Foundation Materials Research Science and Engineering Centers (MRSEC) program (DMR-1719875)."

Laboratory Access

User Status	Signup	Room Entry	Access
Novice (Sessions 1 and 2)	SEM training request form	Manager	Mon – Fri 8am – 5pm

Email ccmr-sem@cornell.edu

Subject Line : Keck Access

Trained (Sessions 3 and 4)	CCMR-FOM	Card Swipe	Mon – Fri 8am – 5pm

Email ccmr-sem@cornell.edu

Subject Line : Keck Access 24/7

Experienced (session 5 and on)	CCMR-FOM	Card Swipe	24/7

Problematic Samples

Some samples pose special problems to the SEM and must be avoided or prepared very carefully.

Moist or wet samples

Moist or wet samples are not allowed at all in the SEM.

Magnetic samples

Magnetic samples are not allowed in the SEM without consultation with Mick.

Powder samples

All powder samples must be vacuumed and blown off.

All powder samples must be coated with gold/palladium or carbon.

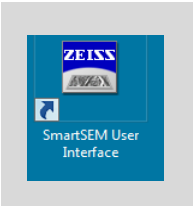
All powder samples shall not go closer than 4mm to the pole piece.

About this manual

This is a ***limited use manual*** intended to help new users get started on the Keck SEM. It provides information on basic imaging, column setup, and saving and annotating images. It is not intended to be exhaustive, and there are many principles about scanning electron microscopy and features of this microscope that are not covered by this manual. This manual will be updated and expanded as needed.

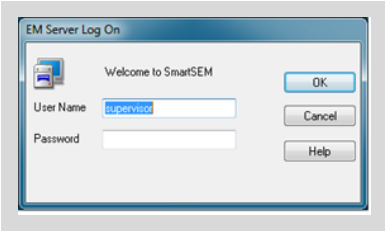
Starting the software

Log into CCMR-FOM



Log into Smart SEM

Enter the username and password:



Username: supervisor

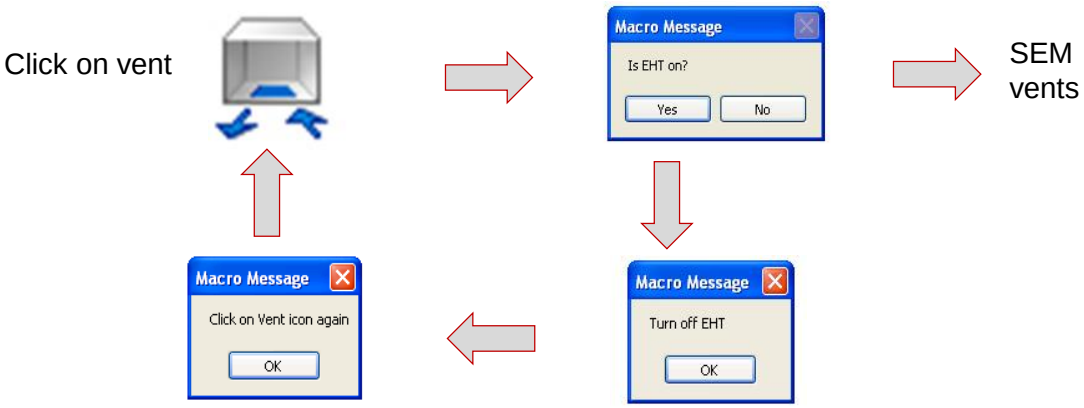
Password: kecksem

Check: Vacuum -> ✔

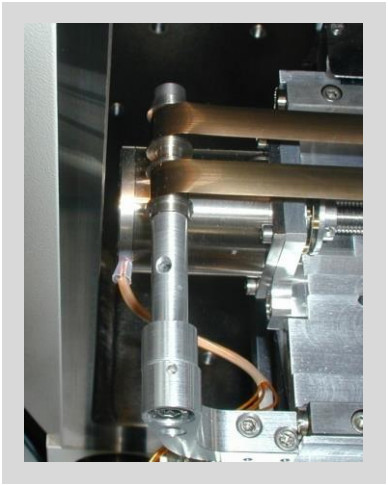
Gun -> ✔

EHT -> X

Loading samples



After venting, ensure that there is a gap between the motor and the chamber wall and the support bar and the chamber wall



Loading samples

Load the stage so that the flat part of the sample holder is flush with the flat part of the stage.

Squeeze the chamber door shut with your left hand and click on the ‘Pump’ icon.

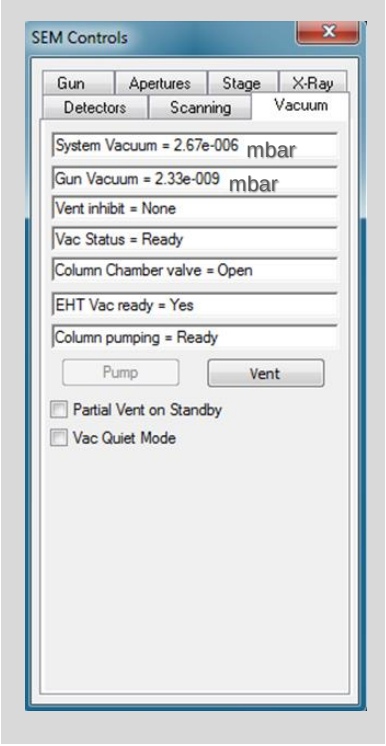
Checking the vacuum

Confirm that the vacuum levels of the gun and system are as follows:

Gun: Less than $6X10^{-9}$ mbar

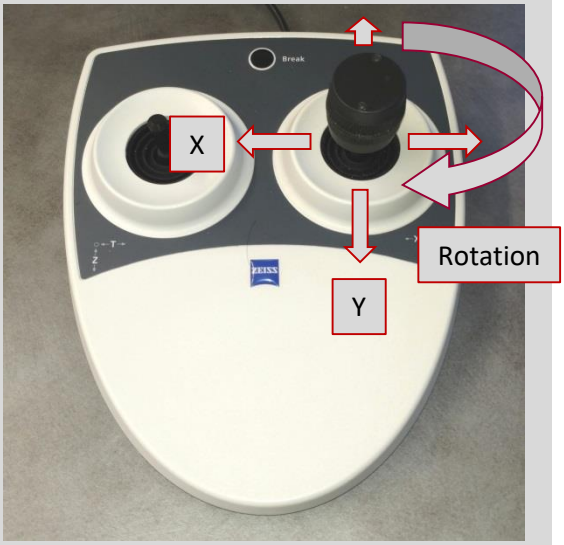
System: Less than $2x10^{-5}$ mbar

If the vacuum does not reach these levels, contact Mick

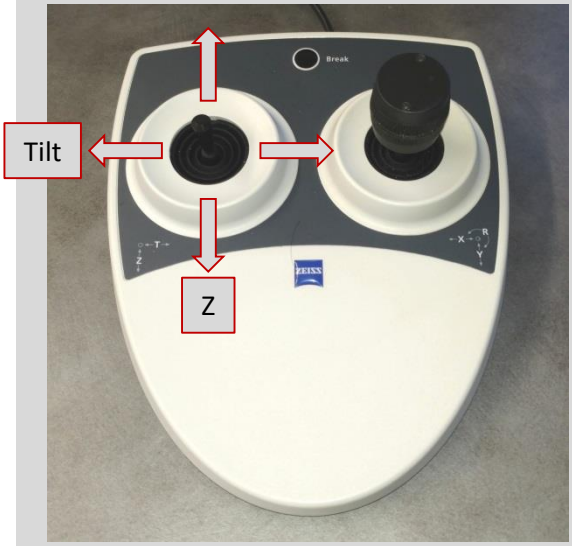


Positioning your sample

To move the stage in the X-Y direction or rotation, use the larger of the two joysticks as shown.



To move the stage in the Z-direction or to tilt, use the smaller joystick as shown.



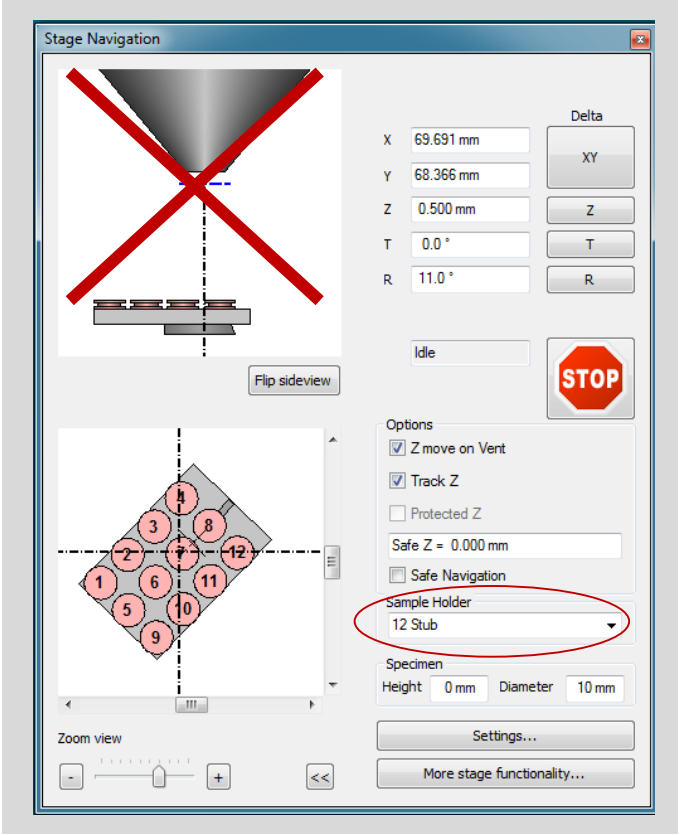
Positioning your sample

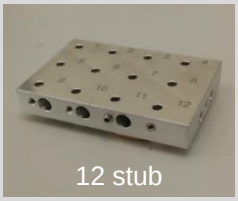
If it is not already on, click the ‘TV’ icon to turn on the external camera.

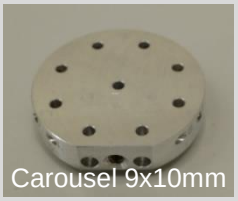
Select your sample holder from the drop-down menu.

Then select the sample you want to examine by double clicking on the number.

Do not trust the top cartoon in the Stage Navigation tab showing the gap between the sample and pole piece – your sample height will likely be different!





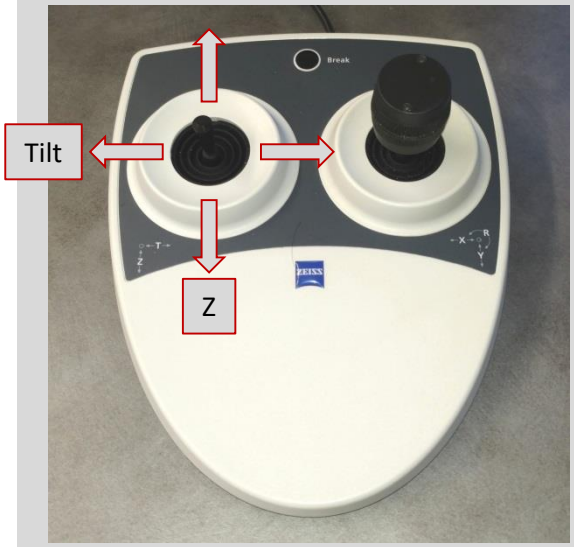


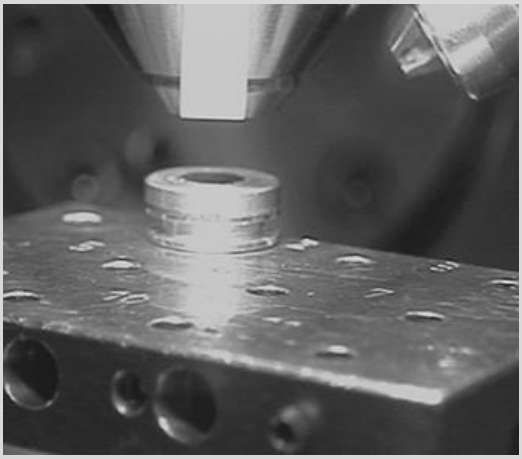
Positioning your sample

Using the Z-joystick, move the sample to about 5 mm from the end of the pole piece as shown below (5 mm corresponds to roughly the width of your index finger).

Set the working distance to be 5 mm (double click on the WD in the image bar).

Once you have focused on the surface of your sample, the focal working distance (WD) value in the datazone bar will update to an accurate value. Then you can further adjust your Z-height to bring the WD to 5 mm.

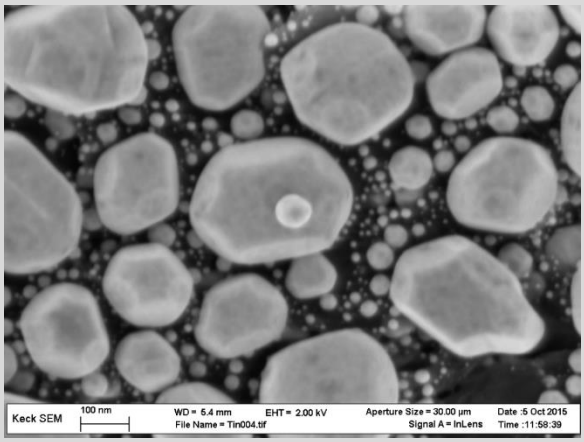




Choosing a voltage

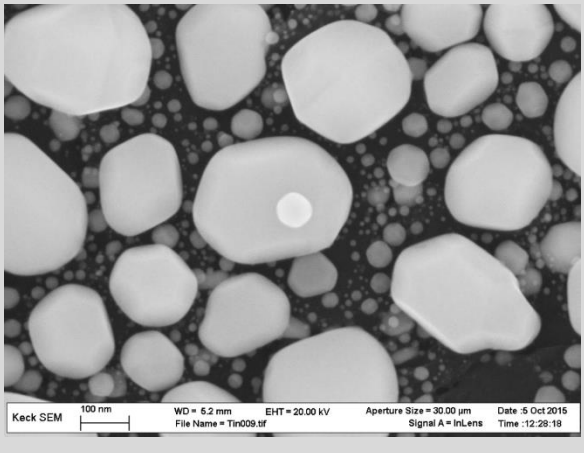
Lower voltages tend to produce more surface detail. However lower voltage electrons also tend to make edges a little softer.

Note the differences in the images of gold islands at right, one taken at 2keV, and the other at 20 keV.



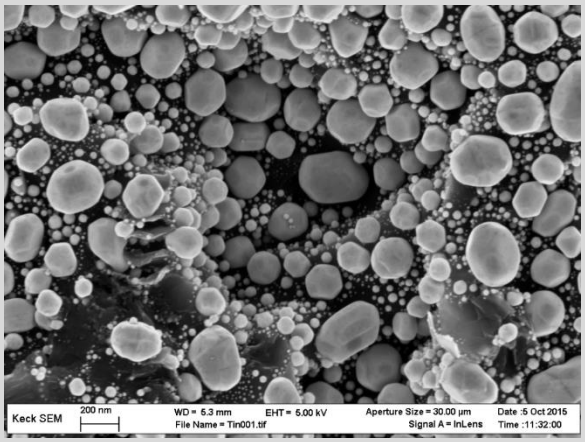
2 keV

20 keV

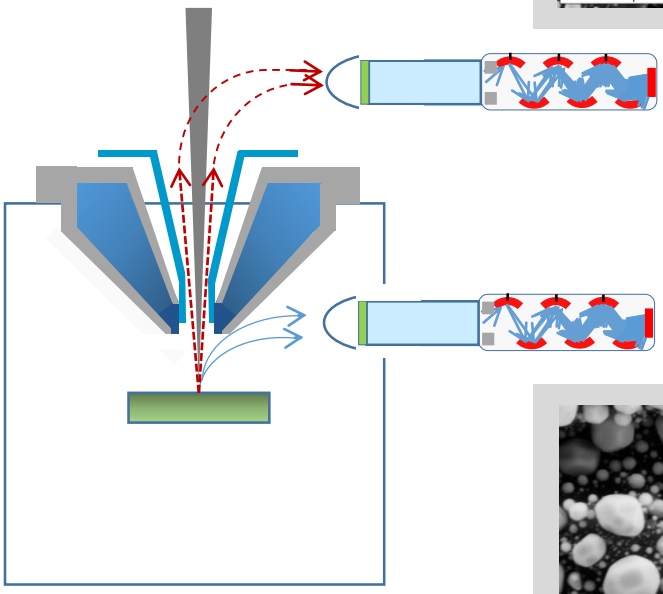


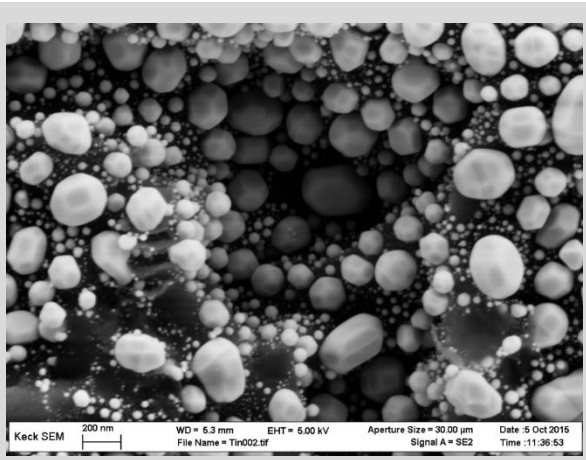
Choosing a detector

The In-Lens detector usually produces the best resolution but can flatten images.



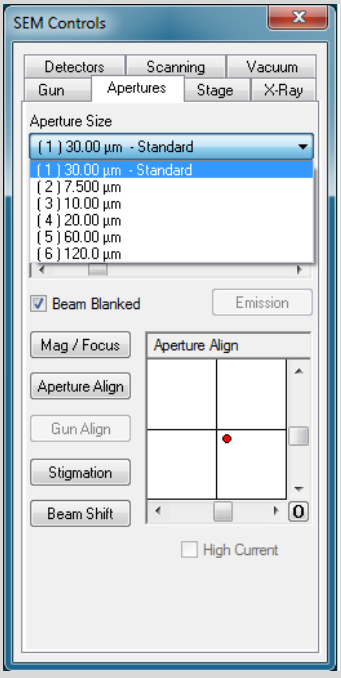
The SE2 renders a more faithful topographic image but sometimes with less resolution.





Choosing an aperture

Click on the Aperture tab and select an aperture.



<u>General aperture guidelines:</u>

10um

Best for insulating samples, beam sensitive samples, or high depth of field:

20um

30um

Best for high resolution imaging and general purpose imaging

60um

120um

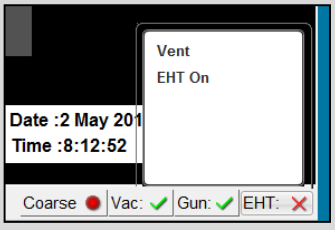
240um (listed as 7.5um)

Best for high current applications such as x-ray analysis:

Imaging

Click on the **H Volt** icon and type in a value in keV (typically a good starting value is in the 1-3 keV range if you are using the In-Lens detector).

Click on the red X next to EHT and click EHT ON

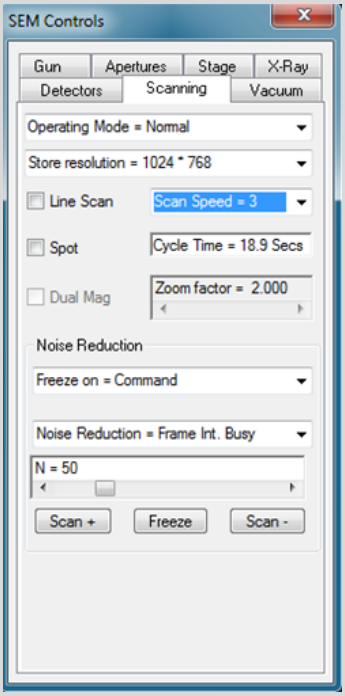


On the Scanning tab, Set the Scan Speed = 3

Set the Noise Reduction = Pixel Average.

Set Signal A = In Lens

You should now see an image on the screen

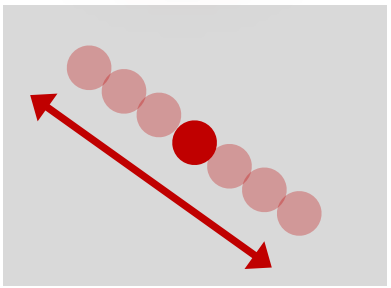


Imaging

When you first turn on the beam the image may be poor, or you may not see anything at all. Some reasons are:

1) The brightness and contrast are not set properly.

2) The focus is off.



3) The aperture is not centered (the object moves back and forth while you go through focus).

4) The stigmatism is not corrected (the object stretches back at 90 degree angles as you go through focus).

5) The sample is not under the beam.

Adjustments - magnification and focus

Click on the MagFoc icon in the toolbar

Click and hold the left mouse button, then move the mouse left and right to adjust the magnification.

Click and hold the middle mouse button, then move the mouse left and right to adjust the focus.

Adjustments - brightness and contrast

To adjust the brightness and contrast of your image, click on the B/C icon in the toolbar.

To adjust the brightness: click and hold the left mouse button, then move the mouse left and right.

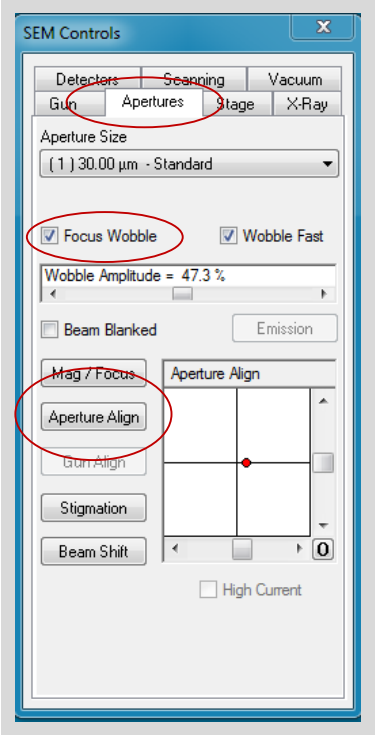
To adjust the contrast: click and hold the middle mouse button, then move the mouse left and right.

Adjustments - centering the aperture

To center the aperture:

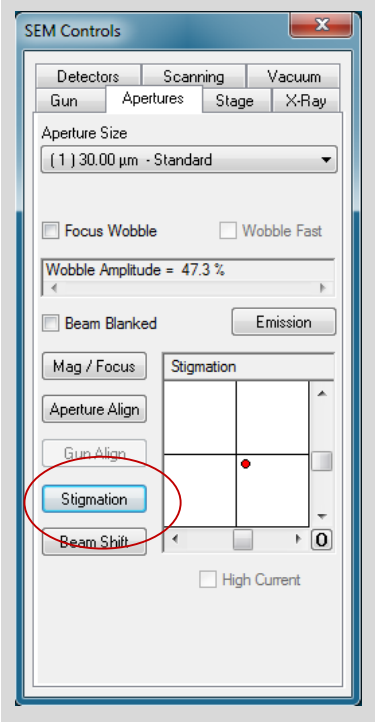
- Start with a reasonably focused image at a reasonably high magnification.
- Click on the Apertures tab.
- Click on Aperture Align.
- Click on Focus Wobble.

Then use the up/down and left/right arrows to adjust the aperture to minimize the image shift.



Adjustments – stigmatism

There are two ways to adjust the stigmation. The first way is similar to the aperture alignment.



- Start by re-focusing the image.
- Click on the reduced icon and center it on an object of interest.
- Increase the scan speed to 5 (optional).
- Click the Stigmation button.
- Then use the up/down and left/right arrows to make the image look crisp and clear.

Adjustments – stigmatism

This is the other way:

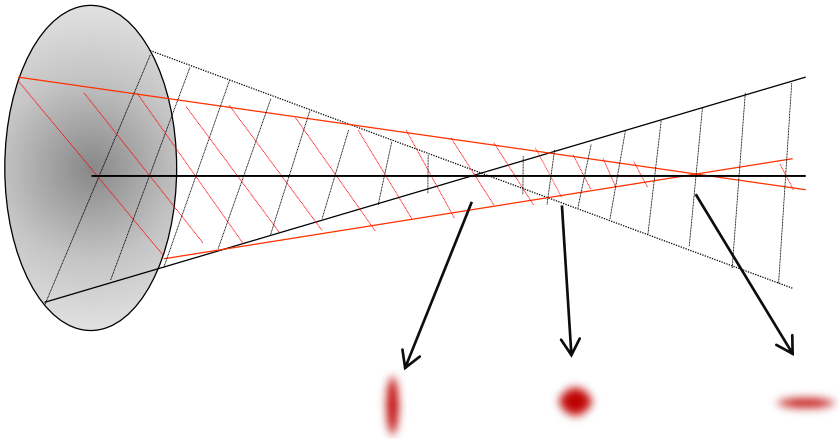
Click on the Stig icon in the toolbar

Click and hold the left mouse button, then move the mouse left and right to correct the stigmatism in the X-direction

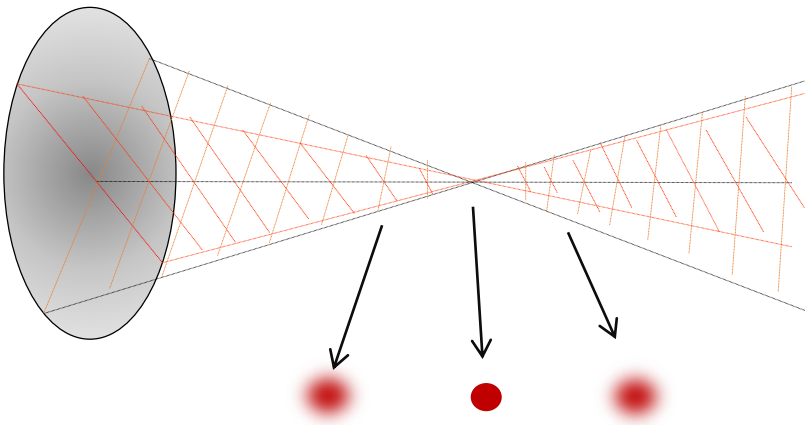
Click and hold the middle mouse button, then move the mouse up and down to correct the stigmatism in the Y-direction

Adjustments – stigmatism

Initially, as you go through focus your image probably looks like this:

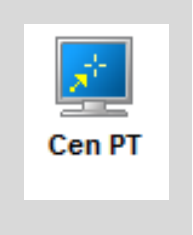


The goal is to adjust the X and Y stigmators so that the object will look like this as you go through focus:



Positioning your sample

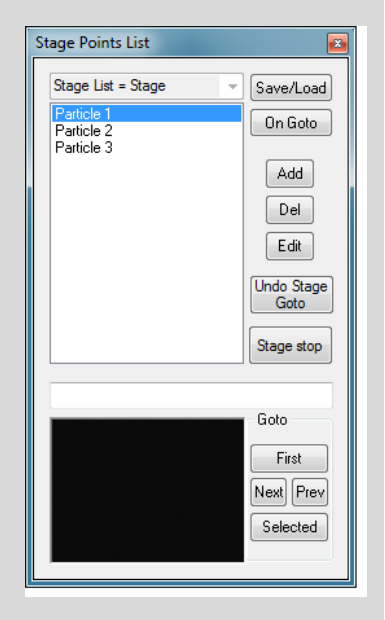
To center a feature in the screen, click on the icon labeled “Cen PT” or on CTRL + Tab. A green cross will appear. Clicking on a feature with the green cross will move it to the middle of the screen.



You can save positions so that you can come back to a previous position quickly.

On the Stage Points List click on **Add.**

Give the location a name.

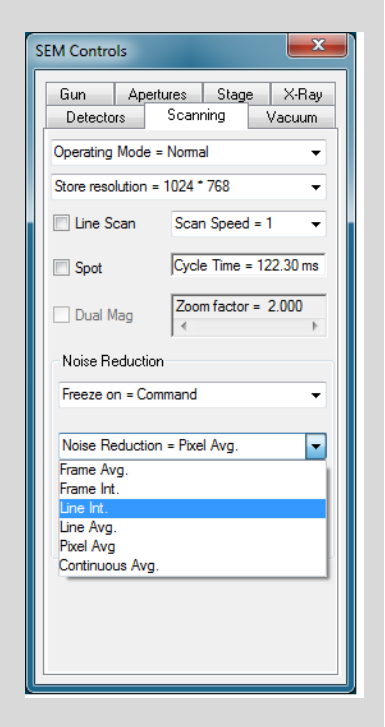


Imaging – taking the image

To take an image go to the scanning tab and in the Noise Reduction section choose either:

1) Line Integrate

2) Frame Integration



For Line Integration a typical setting is:

Scan Speed = 3

N = 100

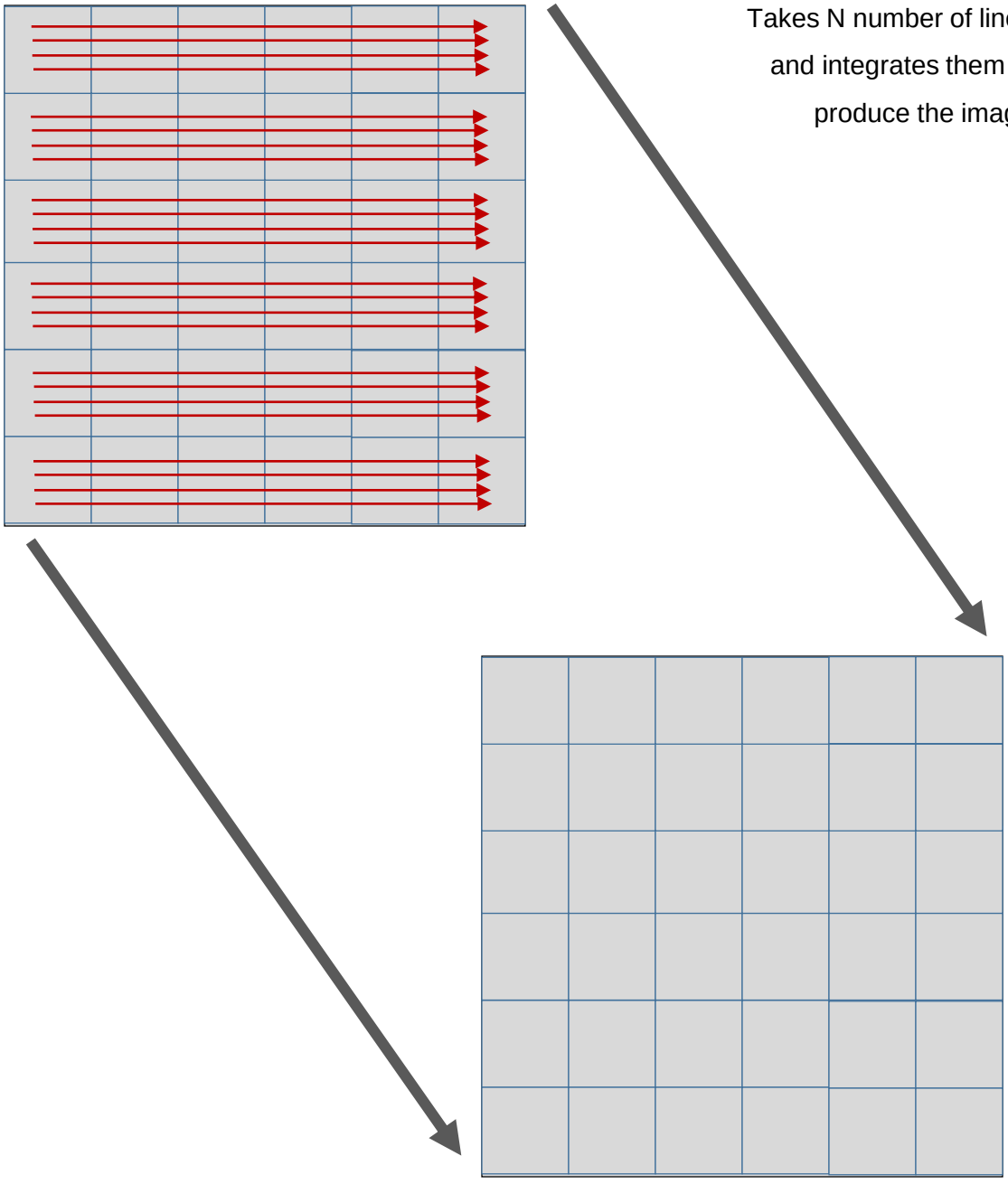
For Frame Integration a typical setting is:

Scan Speed = 1

N = 50

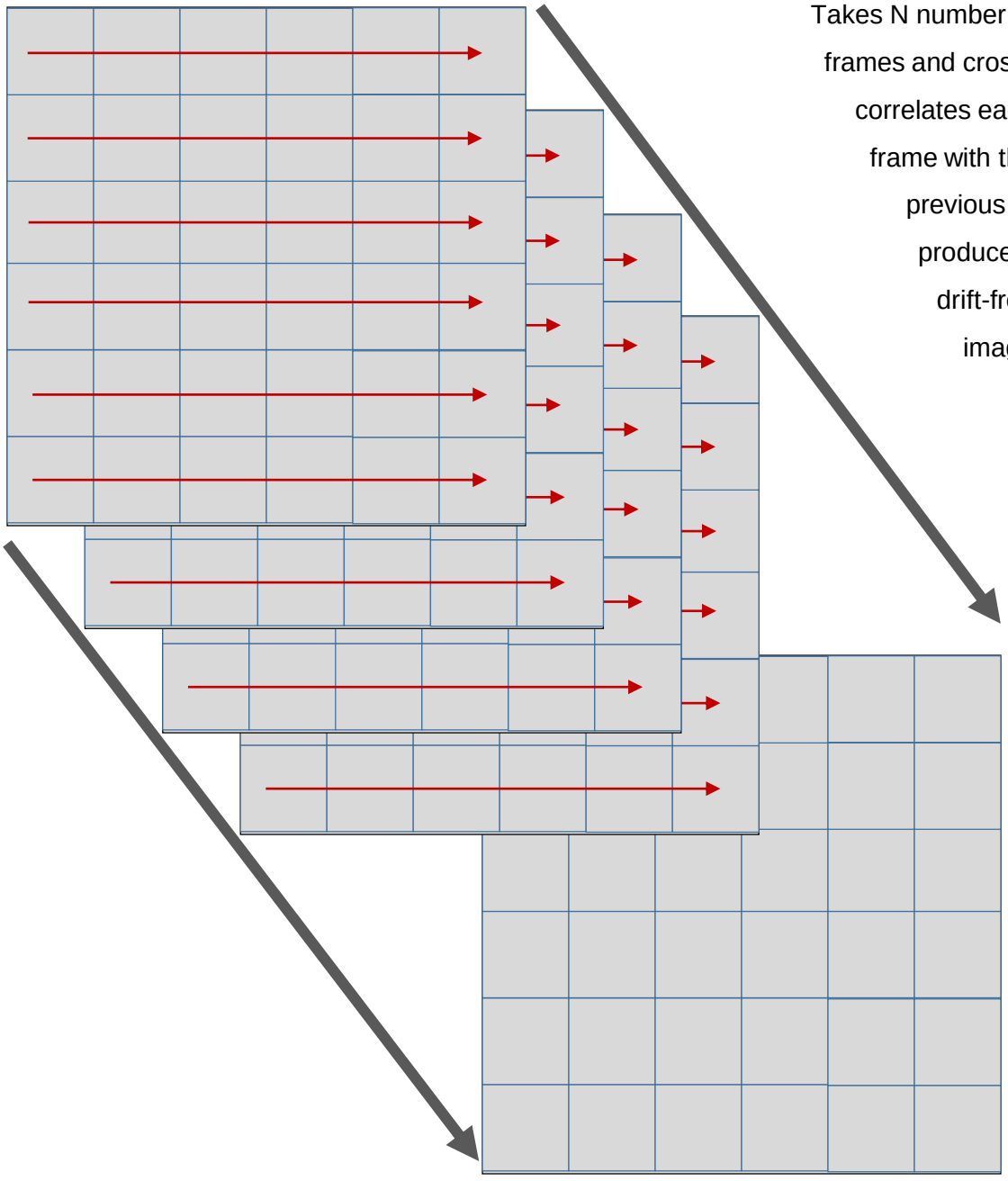
These are just suggested starting values; the best values for your sample could be quite different.

Imaging – line integration



Takes N number of lines and integrates them to produce the image

Imaging – frame integration

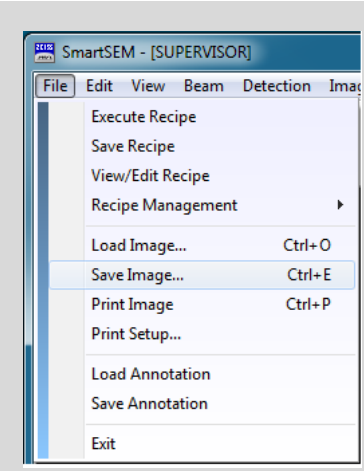


Takes N number of frames and cross-correlates each frame with the previous to produce a drift-free image

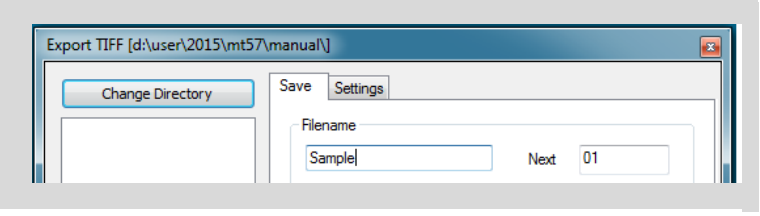
Saving images

To save the first image from a sample:

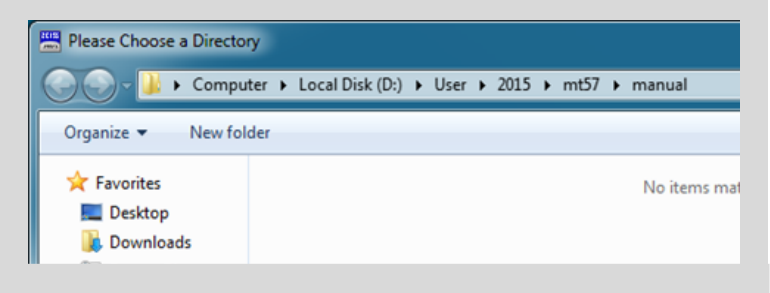
Click On File -> Save Image



Click on Change Directory

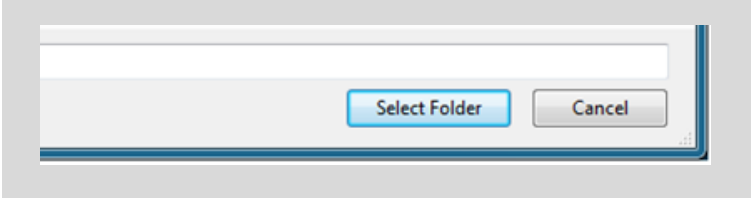


Click on the year and select your folder (or create a new one)

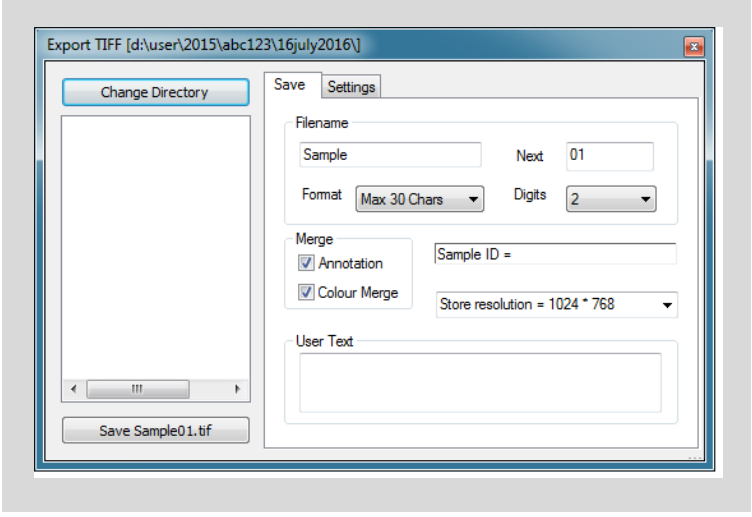


Saving images (con't)

After selecting your folder click on Select Folder at the bottom of the window



The following window will appear:

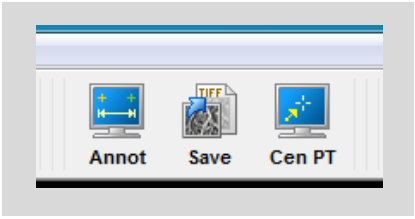


Click in the box below Filename and type in your new filename

Click on Digits and type "2" or "3"

Click in the box to the right of Next and type "01" or "001" as appropriate

Click on Save

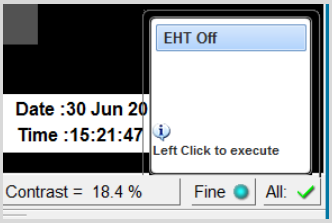


For subsequent images of the same sample simply click the 'Save' icon

Turning off the SEM

Note the icon to turn off the EHT says “**All**” now, not “**EHT**”.

Click on “**All**” and turn off the EHT



Wait for the EHT to ramp down.

Click on the Vent Icon

Answer the questions in the dialogue windows

The stage will automatically go to the correct position

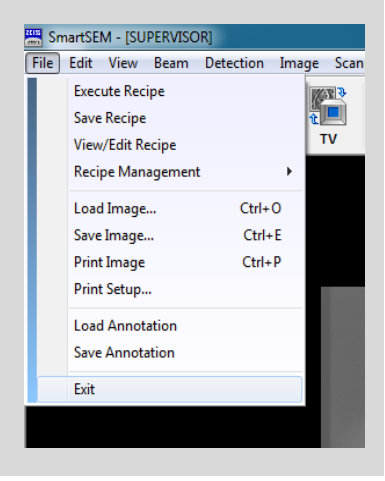
The column will vent

Turning off the SEM

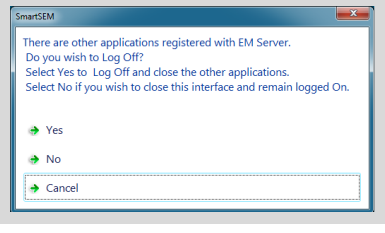
Critical – You MUST pump the chamber back down when you are finished – do not leave it up to air!

After removing your sample from the chamber, hold the chamber door shut and click on the 'Pump' icon.

When the noise of the roughing pump has died away, Exit SmartSEM.

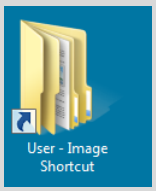


When this window appears, click Yes

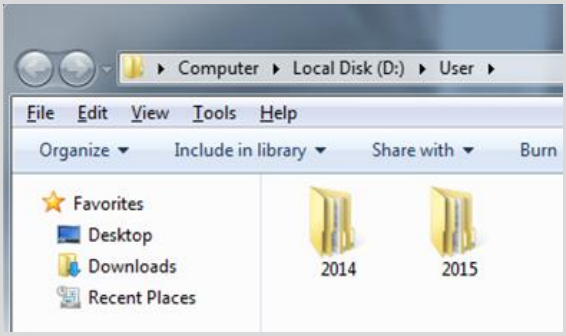


Transferring data

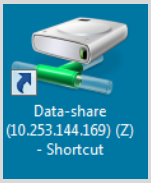
Click on the User-Image Shortcut and select the appropriate folder



Click on the User-Image Shortcut and select the appropriate folder

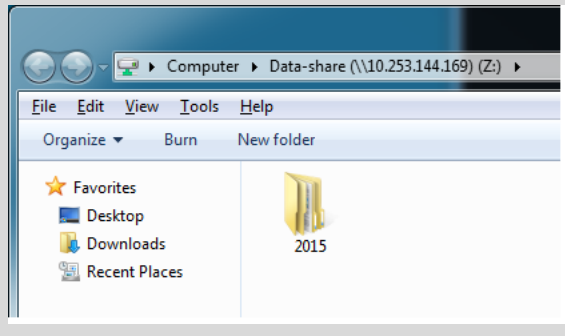


Click on the Data-share Shortcut and select the appropriate folder



Copy your data from the User-Image folder to the Data-share folder

Using Cornell Drop-box transfer your files



Logoff

Log off of the instrument in CCMR-FOM