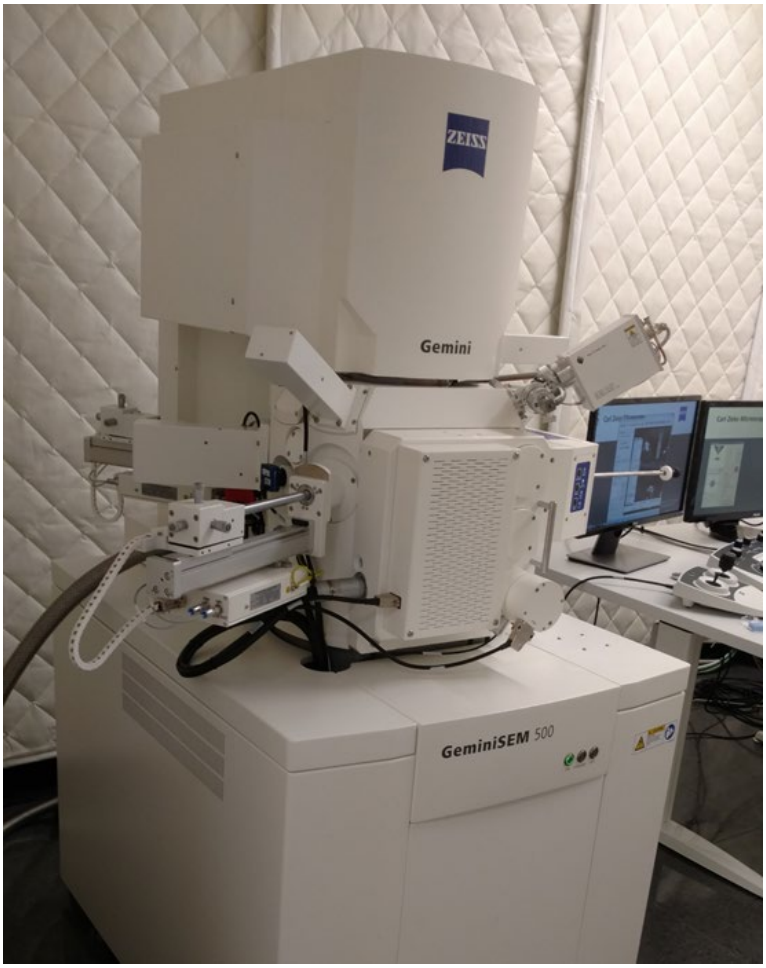
Gemini 500 SEM

part of the CCMR EM Facility

Operating Manual

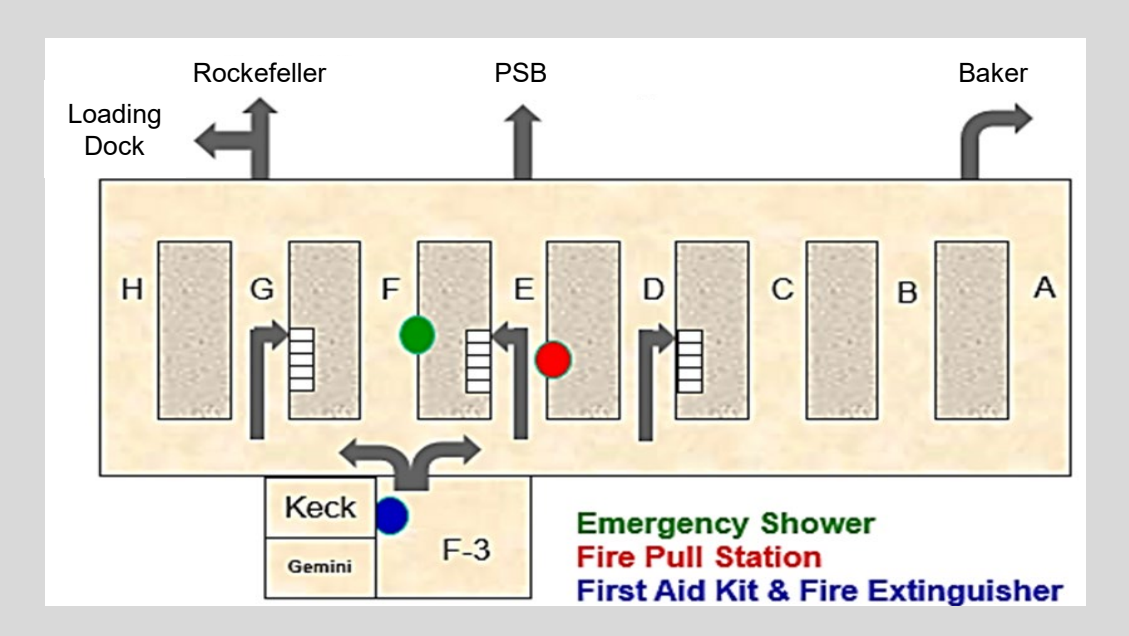


Laboratory Safety

- Users are required to have passed Cornell's on-line laboratory safety training courses particular to this lab before they can use the Gemini 500 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 before using them.
- Do not use your card to let others into the SEM lab.

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 must be dry and the same height.
- If you sign up and then cannot use your time, cancel your time via FOM 24 hours ahead of time or contact Mick at least one weekday before your scheduled time. Failure to cancel in time will result in use charges.
- Failure to show up without canceling, unless due to illness or other emergency, can 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.
- Periodically the hard drive will be erased.

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 & 2)	Email	Manager	Mon – Fri 8am – 5pm

Email Mick

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

Email Mick

Experienced (session 5 +)	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 Zeiss Gemini 500 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

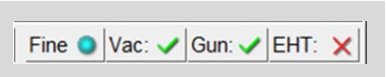
Log into SmartSEM ✔

✔

Check: Vacuum ->

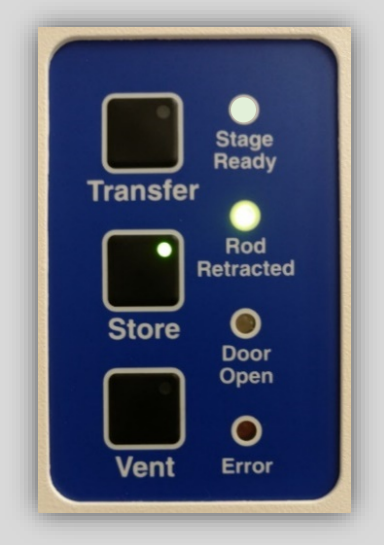
Gun ->

EHT -> X



Mount your samples onto a shuttle

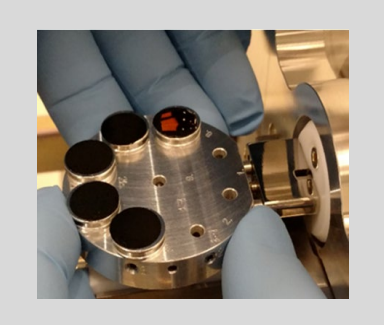
The Rod Retracted LED and the Store LED should be lit.



Push the VENT button on the load-lock.

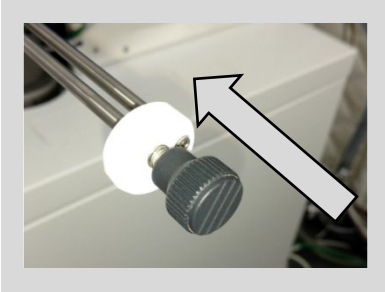
About 10-15 seconds later, gently pull the door open.

Slide the shuttle onto the Teflon loading plate. The screw hole should point towards the load arm.

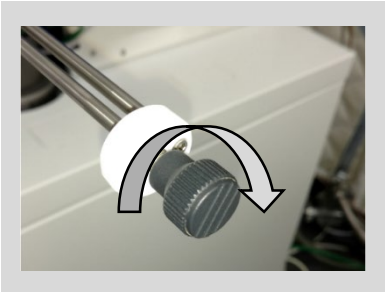


Loading your sample

Push rod gently to engage the shuttle

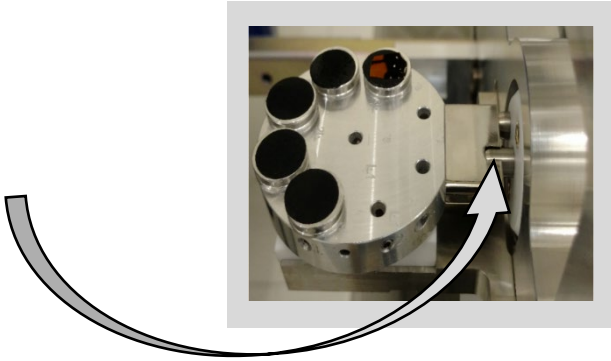


Rotate the rod clockwise until snug



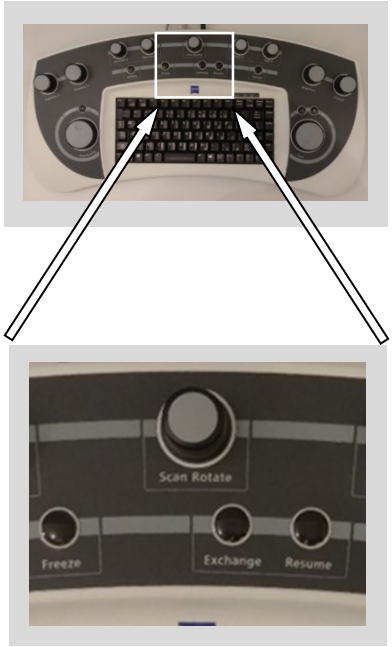
When it is snug the shuttle and the rectangular block will be touching as shown at right.

Note that a small cylinder will fit the slot.

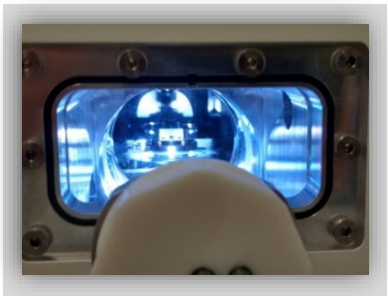


Loading your sample

Close Load Lock door and push the EXCHANGE button

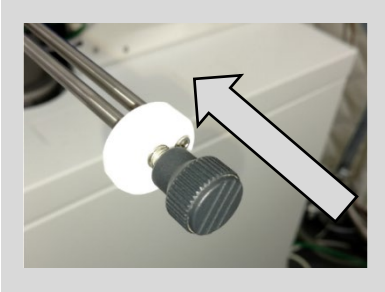


When the vacuum in the Load- Lock is low enough, the valve will automatically open and the light will turn on.

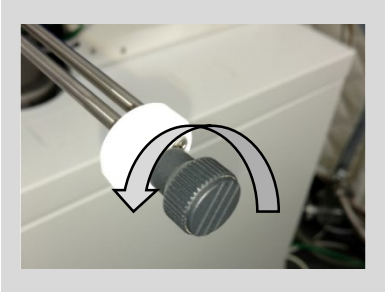


Loading your sample

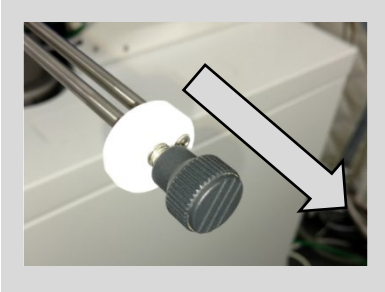
Push the rod into the chamber to load the Shuttle.



You will feel a hard stop, then rotate the knob on the load arm counter-clockwise at least 5 turns.



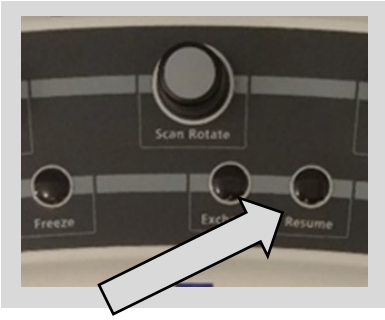
Pull the load arm out until you hear and feel a click.



Loading your sample

Important! After you pull the rod out the Rod Retracted light must be on or you have not pulled the arm all the way out.

Push the RESUME button. The light in the load lock will turn off and the valve will close.

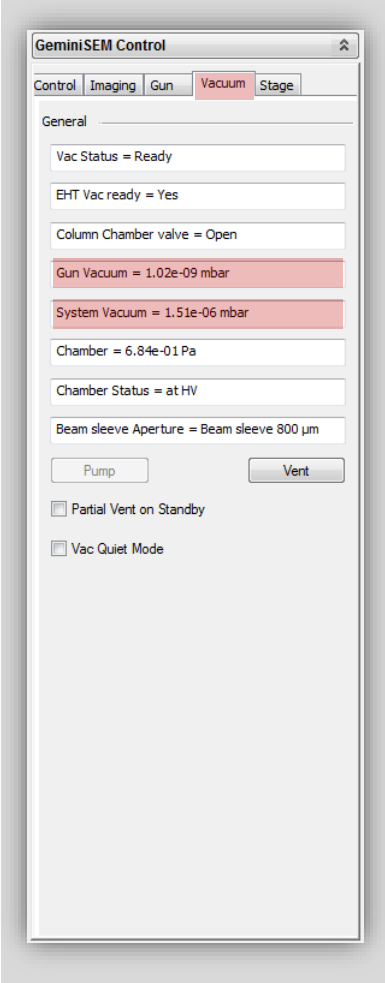


Checking the vacuum

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

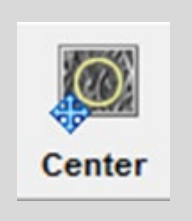
- Gun: Less than $2X10^{-9}$ mbar
- System: Less than $4x10^{-6}$ mbar

If the vacuum does not reach these levels contact Mick



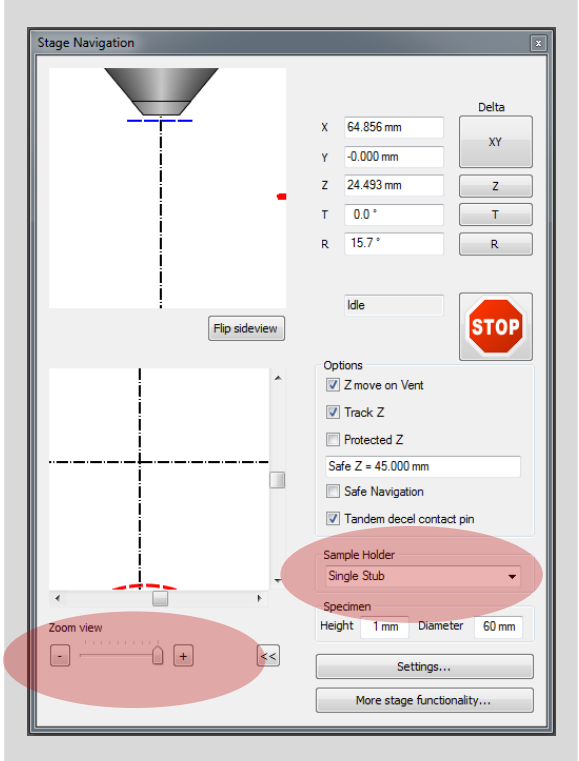
Positioning your sample

Click on the Center icon.
This will move your sample to the middle of X-Y travel



Use the Zoom View slider to magnify the image of your sample holder

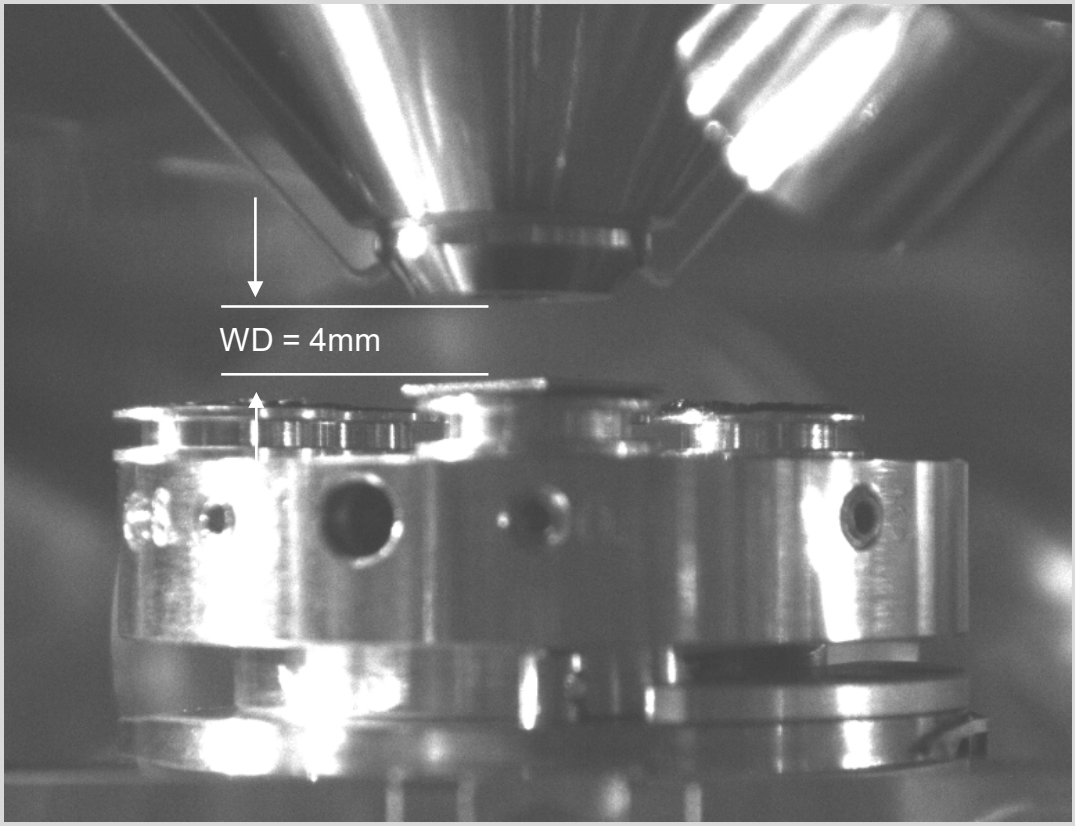
Select your sample holder from the drop-down menu:



Positioning your sample

Click on the TV1-TV2 button to go back and forth between the two different cameras.

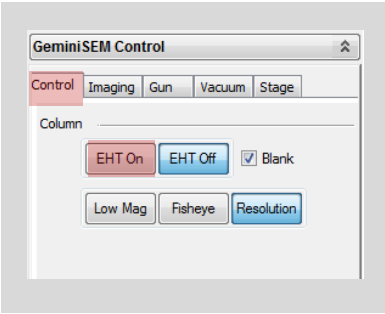
Use the Z-control to move the sample to about 4mm from the end of the pole piece. Set the WD to 4 mm.



Imaging

Click on the EHT icon and set the EHT to the desired value

Turn on the EHT

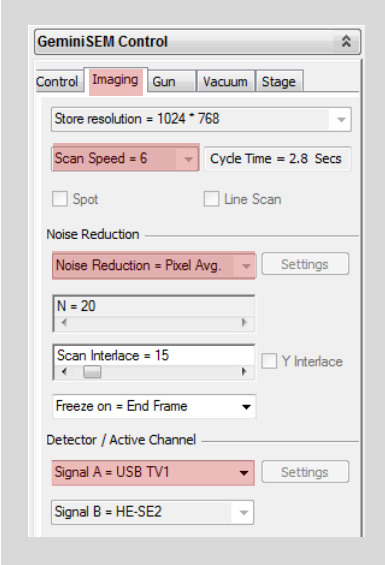


Set Signal A = InLens

Set the Scan Speed = 3

Set the Noise Reduction = Pixel Average.

You should now see an image on the screen.



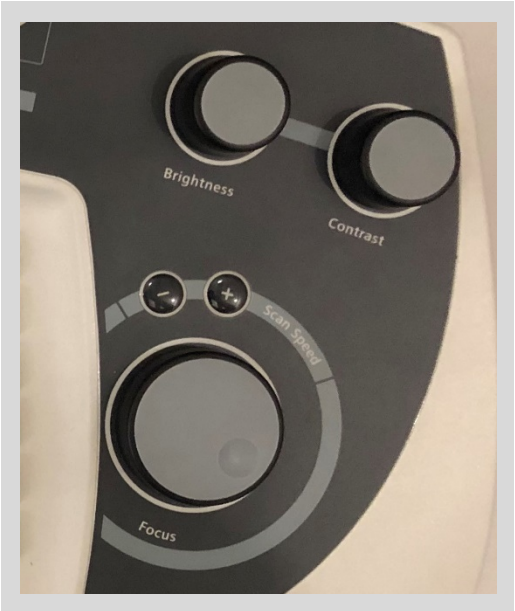
Imaging

When you first turn on the beam you may not see anything at all. Some reasons are:

1) You are not over your sample.

2) The brightness and/or contrast may not be set properly

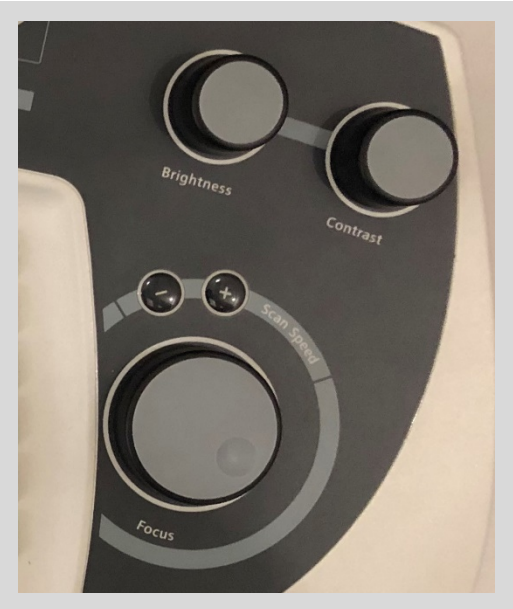
3) Your working distance (WD) may be too short (0.0 mm) or too long (Focus). Try setting WD to 4 or 5mm



4) Your magnification may be too high, set it as low as possible

Imaging

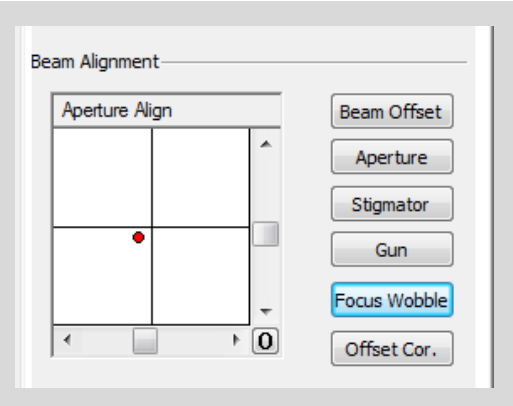
Once you can see an image of your sample, it is still very unlikely that it is optimized, so you must follow the procedure below.



Start with a reasonably focused image.

Rotate the focus knob back and forth (over focusing and under focusing the beam)

If objects in the field of view move back and forth, then the aperture is not centered.



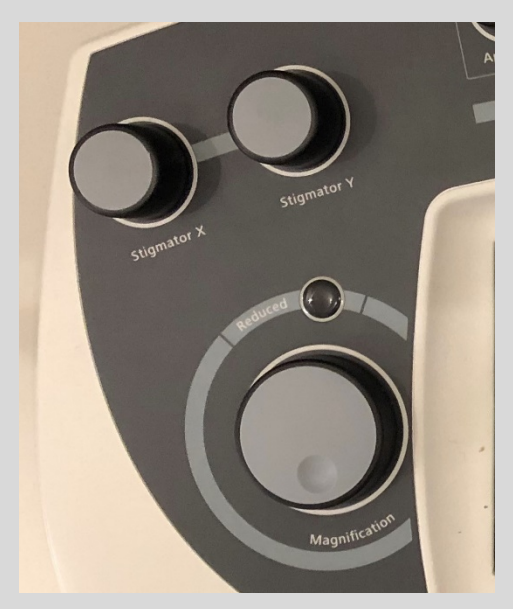
Centering the aperture:

1. Set the scan speed to 1 or 2
2. Click on Aperture
3. Click on Focus Wobble
4. Click on the up/down and left/right arrows to stop the motion

Imaging

2) If the object stretches as you go through focus, first one way, then the other, you have stigmatism which must be corrected

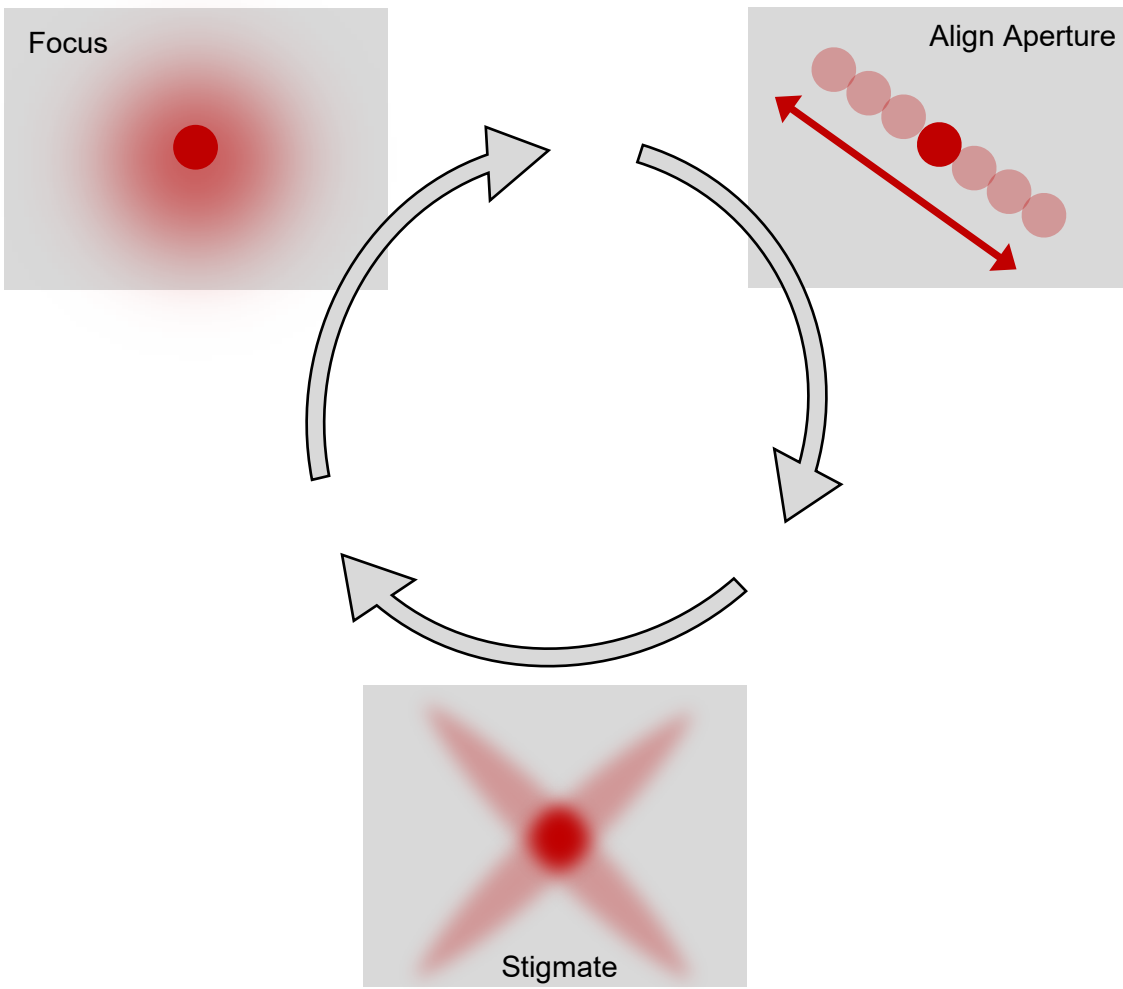
.



Imaging – complete process

This process needs to be checked and repeated:

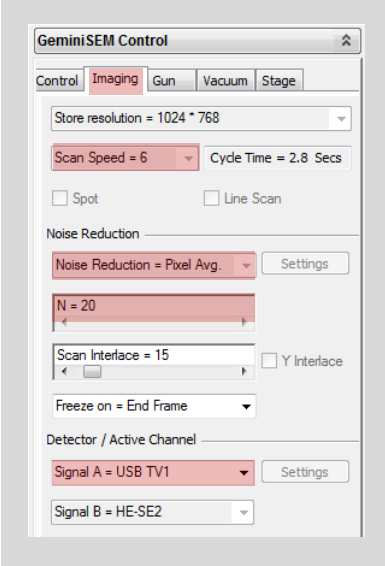
1) As you increase the magnification
2) After you move to a new area
3) Any time you want to make sure you are getting the best image possible.



Imaging – taking the image

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

1) Line Integrate
2) Drift Compensated Frame Integration



For Line Integration a typical setting is:

Scan Speed = 3

N = 100

For Drift Compensated Frame Integration a typical setting is:

Scan Speed = 1

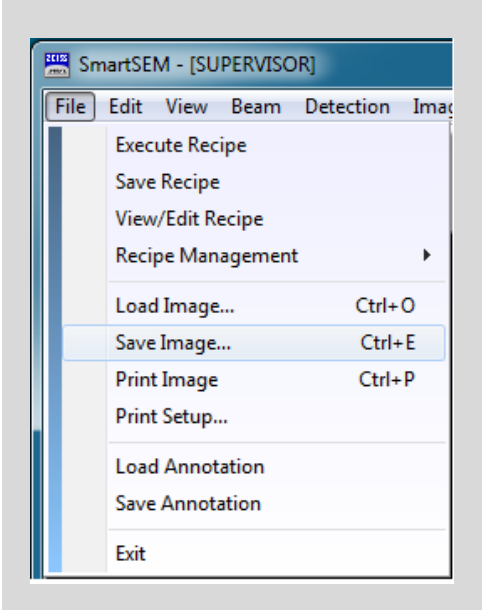
N = 50

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

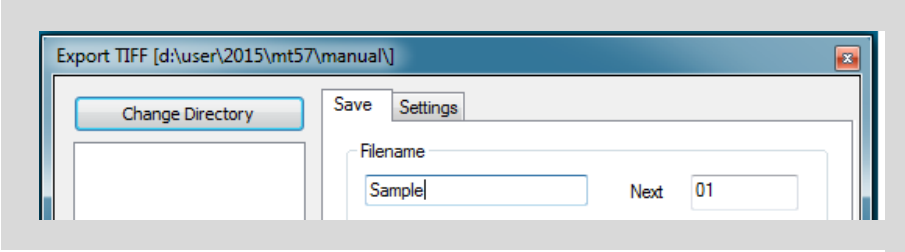
Saving images

To save the first image from a sample:

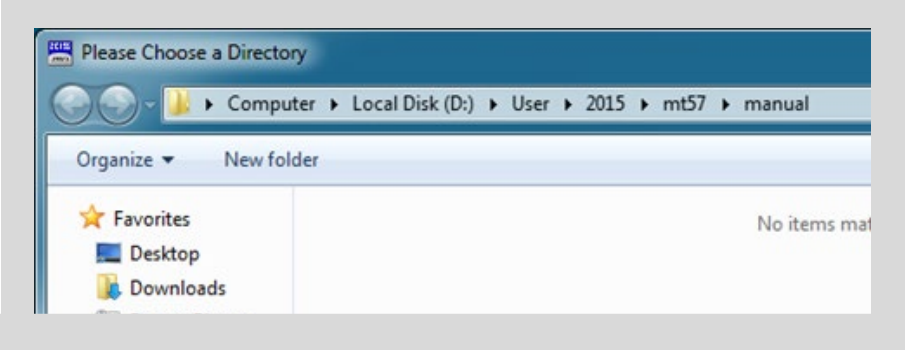
Click On File -> Save Image



Click on Change Directory

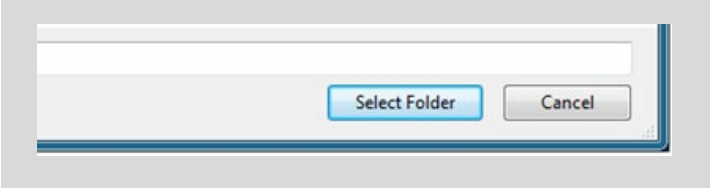


Click on the year and select your folder (or create a new one)



Saving images (con't)

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



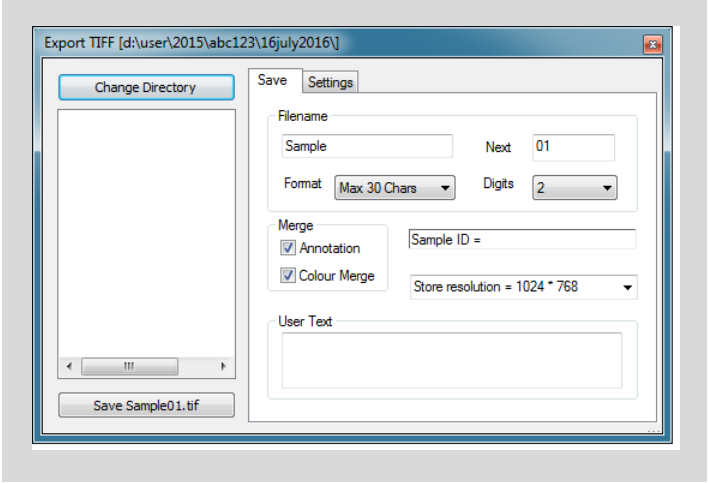
The following window will appear:

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

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

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

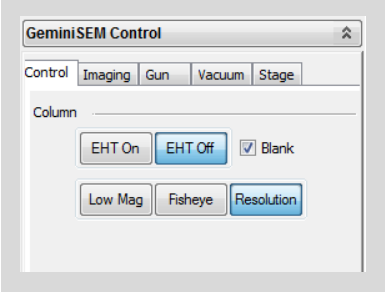
Click on Save



For subsequent images of the same sample simply click on Save

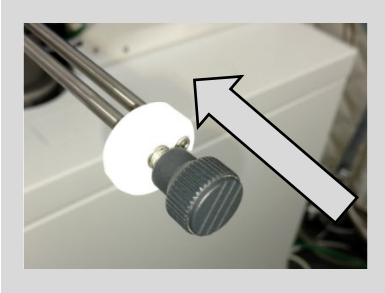
Turning off the SEM

Go to Gemini SEM Control -> Control -> EHT off



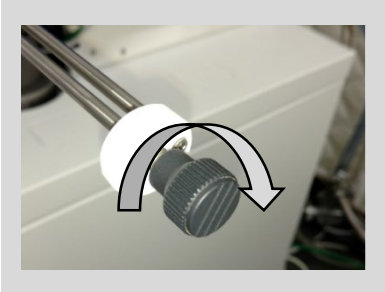
After the EHT is fully off click on 'Exchange' on the console (do not move X, Y, Z or rotation manually – the software will do this automatically).

The stage will move into position and the valve will open.



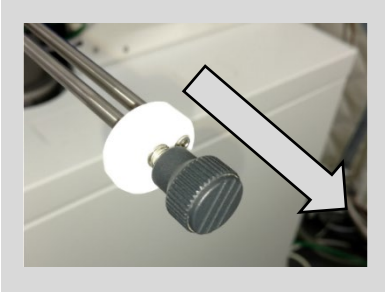
Gently push the arm in until it engages the shuttle.

Rotate the arm about five turns clockwise until snug.



Turning off the SEM

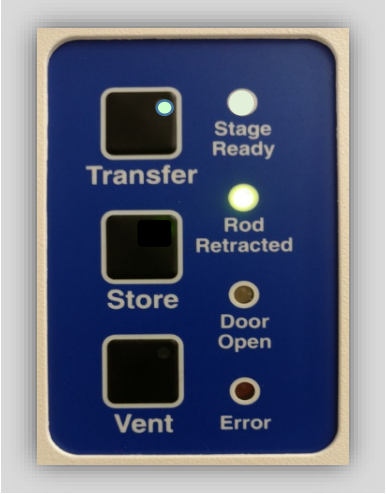
Pull the arm out until you hear a click.



IMPORTANT! The green "Rod Retracted" LED must be lit.

Click on Resume - the valve will close and the light will turn off.

Resume Complete will show up on the monitor – click OK



Push the Vent button on the load lock.

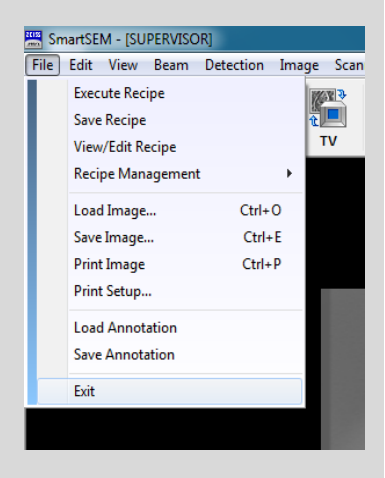
When the load lock is vented, open the door and remove the shuttle.

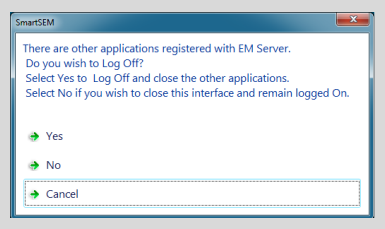
Close the load-lock and click on Store

Turning off the SEM

When your sample is removed and the loadlock is pumped back down, go to File ->Exit

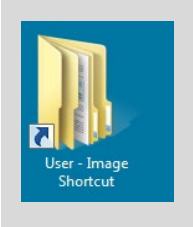
When this window appears, click Yes



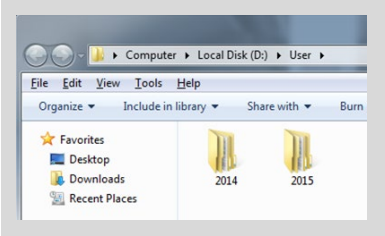


Transferring data

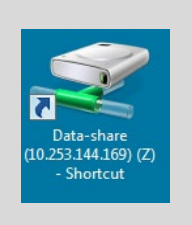
Click on the User-Image Shortcut and select the appropriate folder



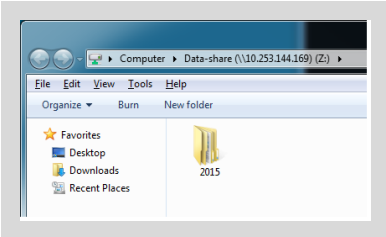
Click on the Data-share Shortcut and select the appropriate folder



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



Using Cornell Drop-box transfer your files



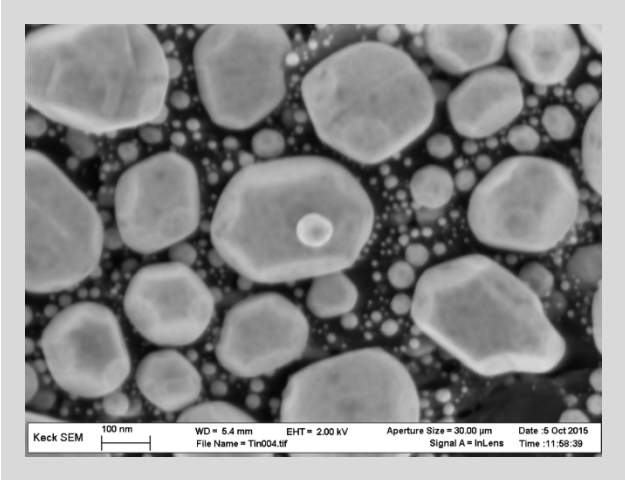
Logout

Log off from CCMR-FOM

Choosing a voltage

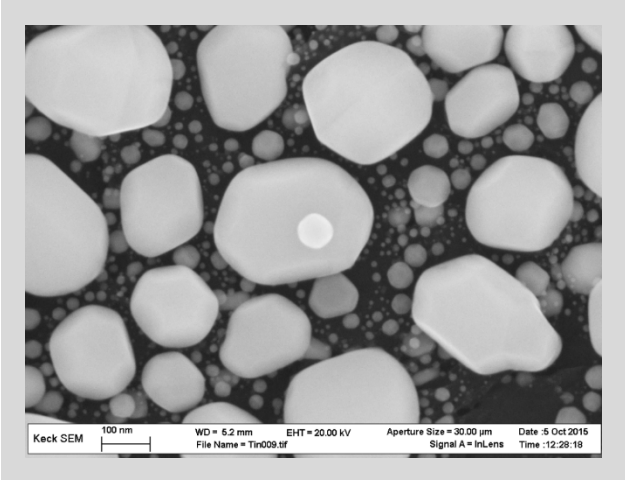
Lower voltages tend to produce more surface detail. However lower voltage electrons also tend to make edges a little softer.

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



2 keV

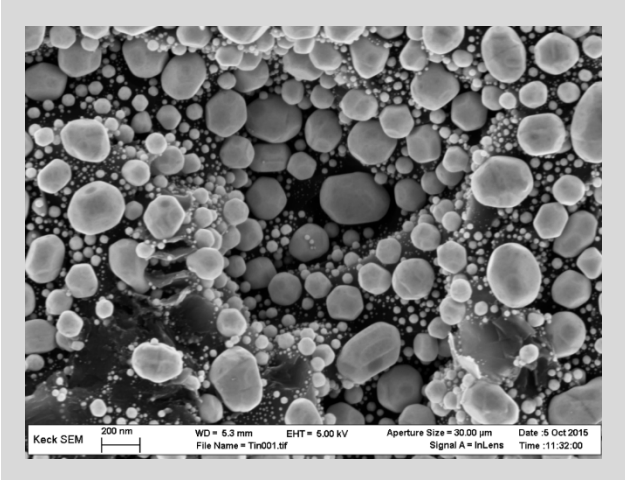
20 keV

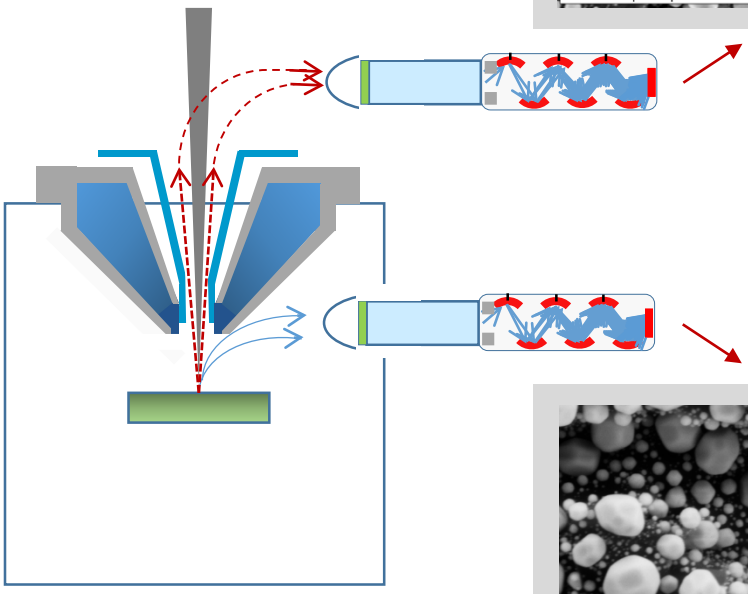


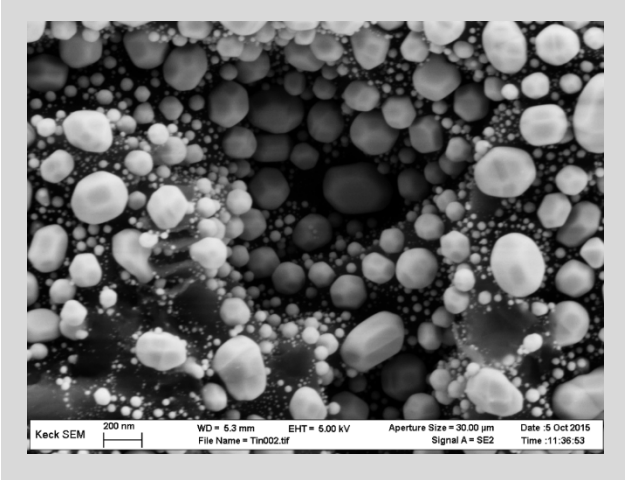
Choosing a detector

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

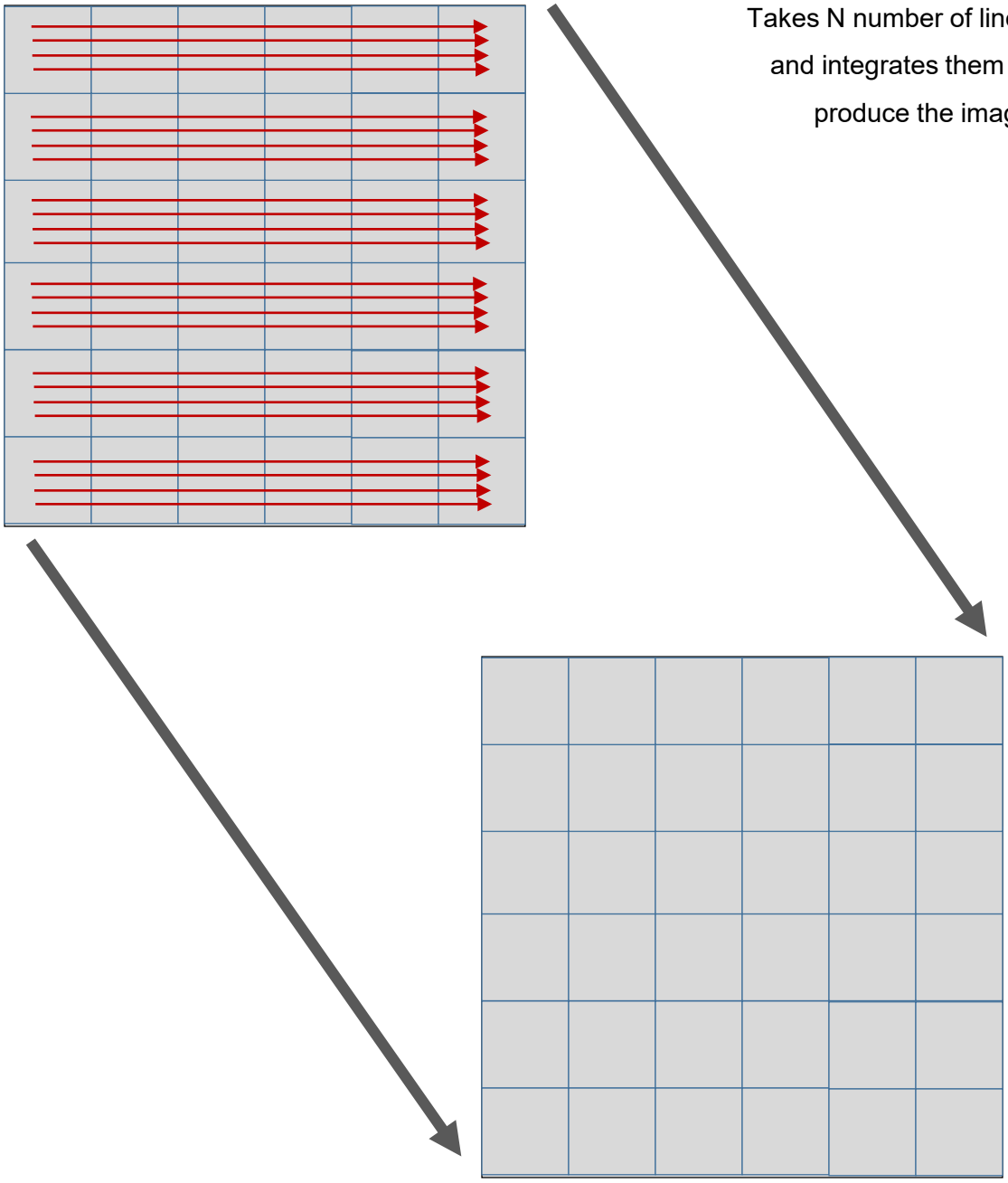
The HESE2 renders a more faithful topographic image but sometimes with less signal to noise.





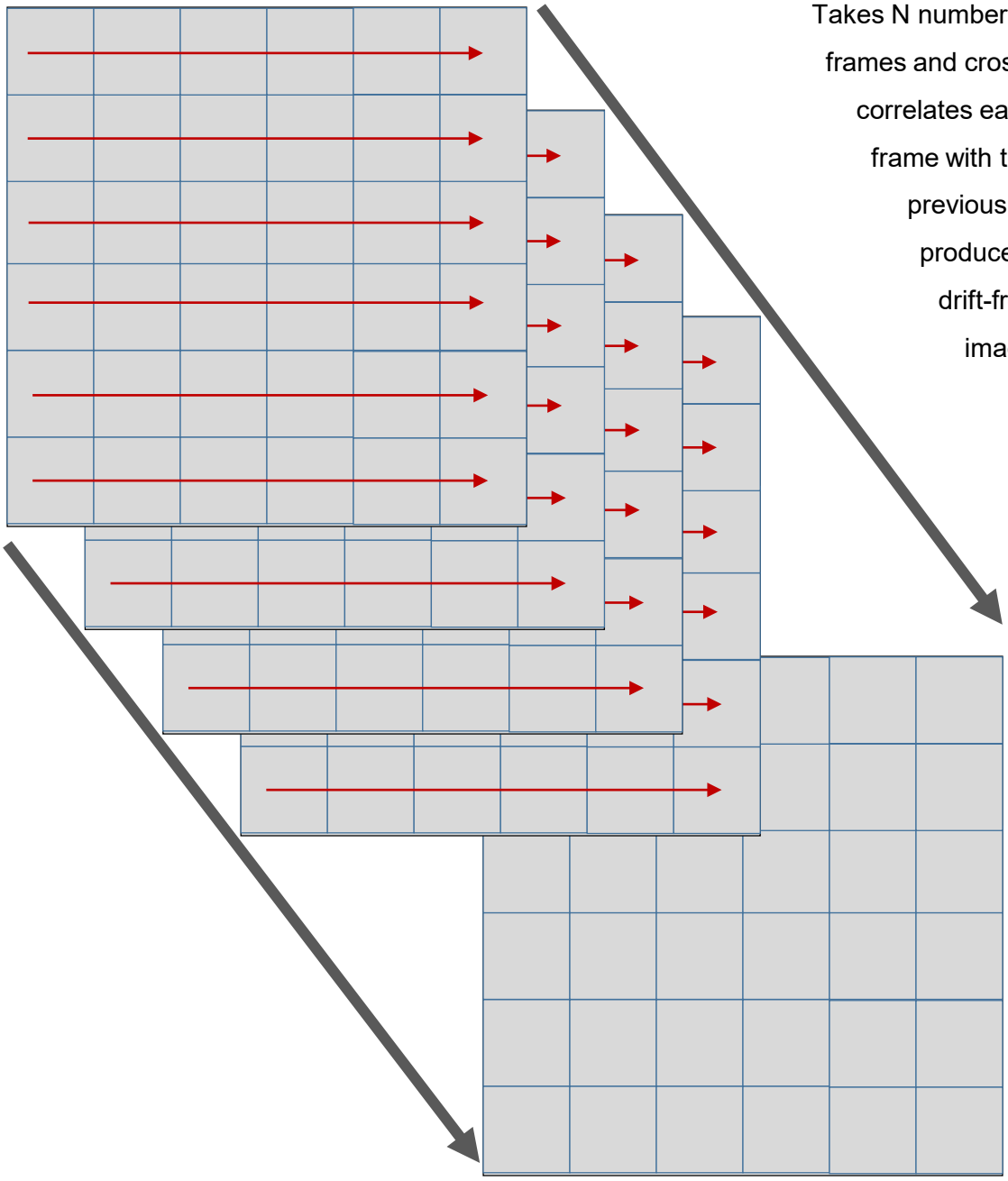


Imaging – line integration



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

Imaging – drift compensated frame integration

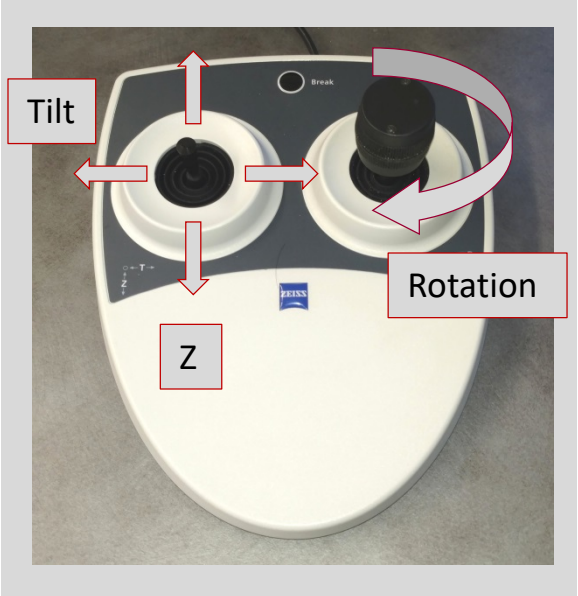


Takes N number of frames and cross-correlates each frame with the previous to produce a drift-free image

Positioning your sample

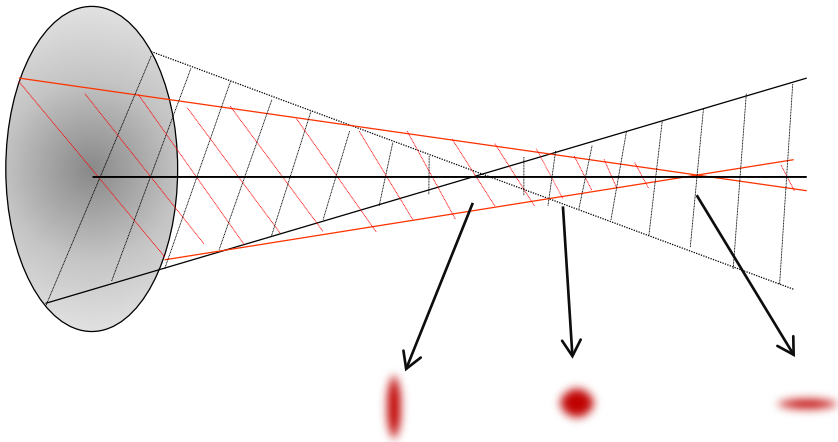
To move the stage in the X-Y direction, use the larger of the two joysticks as shown

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

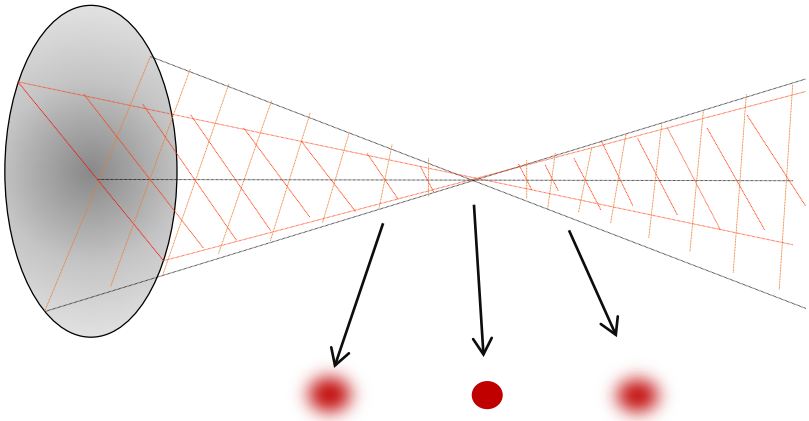


Adjustments – stigmatism

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:



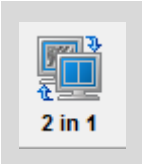
Imaging – 2 in 1

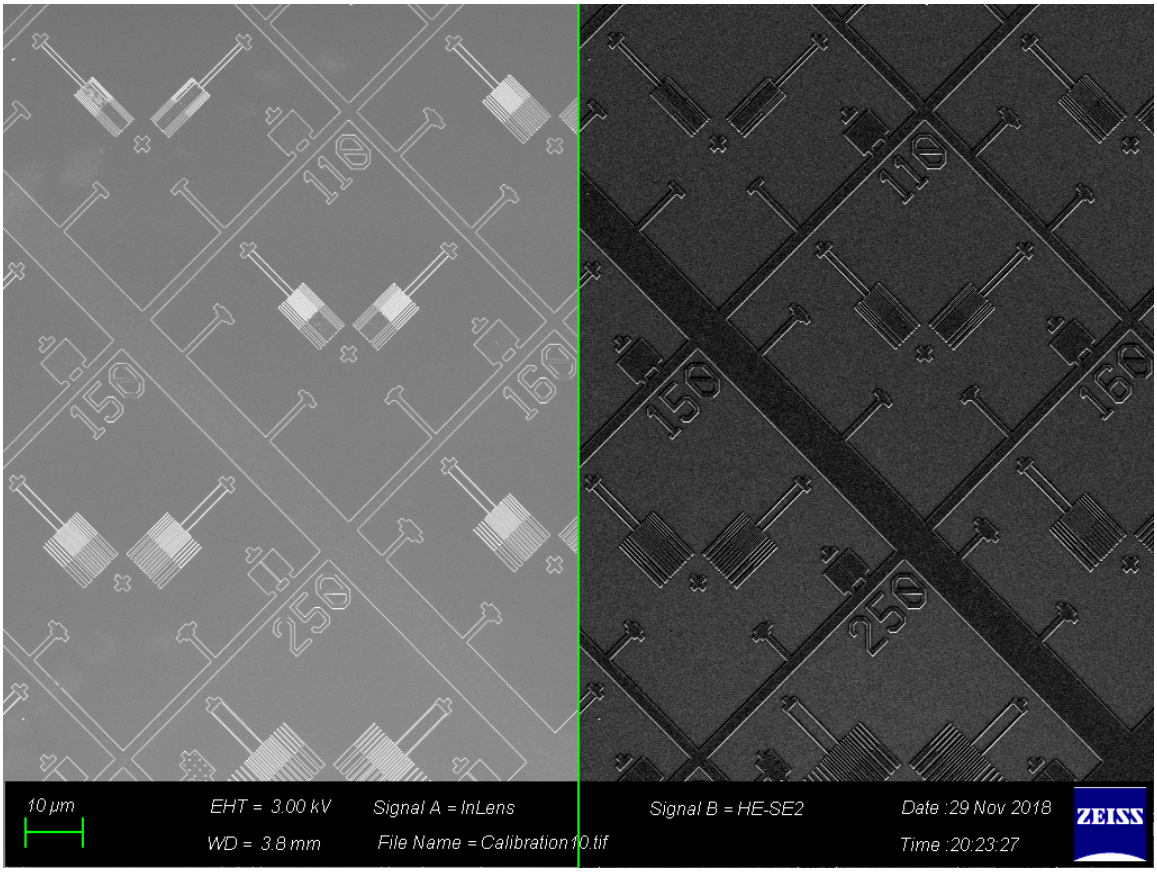
In this mode two different signals (e.g. In-Lens and Backscattered) are collected at the same time and displayed in the same window.

To get this click on the 2 in 1 icon on the toolbar

Then click on View -> DataZone -> Load User DataZone -> 2-in1

Return DataZone and imaging mode to “Normal” when finished.





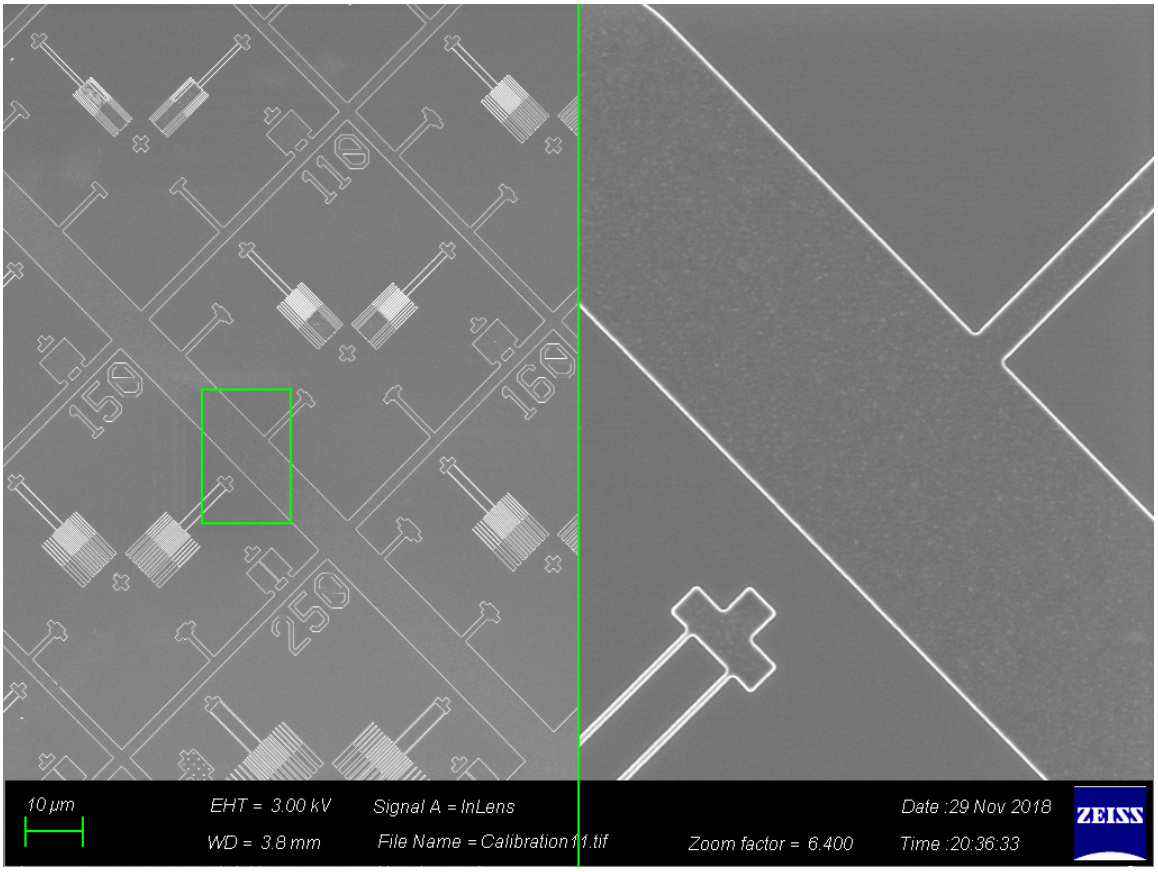
Imaging – Dual Mag

In this mode two different magnification images are collected and displayed in the same window.

To get this click on the DualMag icon in the toolbar

Then click on View -> DataZone -> Load User DataZone -> DualMag
The “Zoom factor” in the DataZone reflects the relative scale
A scale bar can be inserted in the magnified image.

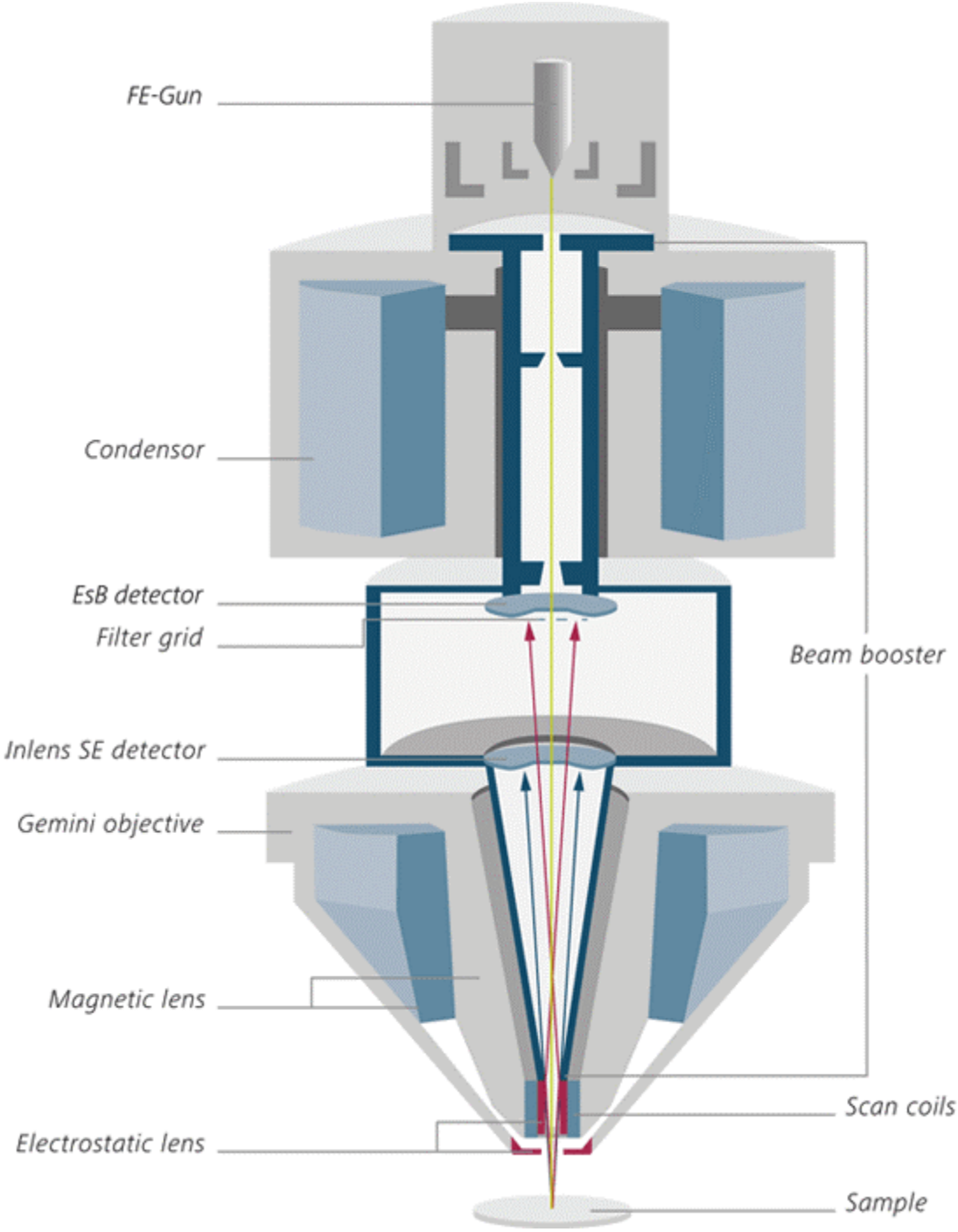
Return DataZone and imaging mode to “Normal” when finished.



Gemini 500 SEM

Reference

part of the CCMR EM Facility



Choosing an aperture

	Low Current Mode	
Aperture (µm)	Probe size (nm)	Current (pA)
7	1.08	3.5
10	1.04	6.6
15	1.66	13.5
20 (center)	1.1	37
30	9.3	51.2
60	71.9	205
120	574	778
	High Current Mode	
Aperture (µm)	Probe size (nm)	Current (pA)
7	3.6	6.5
10	2.6	12.2
15	1.9	24.8
20 (center)	1.5	40.7
30	1.2	94.2
60	1.9	377
120	10.1	1440

	Best Imaging Modes	
Aperture (µm)	Probe size (nm)	Current (pA)
20 (low current)	1.1	37
30 (high current)	1.2	94.2
	Best X-ray Modes	
Aperture (µm)	Probe size (nm)	Current (pA)
30 (high current)	1.2	94.2
60 (high current)	1.9	377
120 (high current)	10.1	1440