Standard Operating Procedure

Hitachi UHR CFE SU8230 SEM

Yale West Campus
Materials Characterization Core
ywcmatsci.yale.edu

ESC II, Room E119F
810 West Campus Drive
West Haven, CT 06516

Version 1.5, February 2017

> *Please **FOLLOW the SOP strictly** to keep the facility in good condition. Any **explorations are strongly prohibited** unless permitted by lab manager*
> *__NEVER__ use your own USB drive on the SEM computer. Data can be retrieved from Yale data server*
> *__NEVER__ surf the web on the SEM/EDS computer in order to minimize the risk of the computer being hacked*

> *Yale West Campus MCC facility users must acknowledge MCC in their publications that rely significantly on MCC resources. The general acknowledgement for SEM should read:*
> "The micrographs were taken using the Hitachi SU8230 CFE SEM at Yale West Campus Materials Characterization Core (MCC)."
> *The core reserves the right to use the micrographs for core promotion*

Table of Contents

Hitachi SU8230 Standard Operating Procedure

1 Introduction

1) Instrument features:
   > Cold field emission (**CFE**) e-beam source → high resolution on conductive surfaces (**0.8 nm** on Au clusters/magnetic tape)
   > Sliding-in annular Energy Dispersive Spectroscopy (**EDS**) detector → high elemental mapping resolution
   > Sliding-in annular Photo Diode **PD-BSE** detector → much high intensity backscattered electron detection than regular SE detectors
   > Scanning Transmission Electron Microscopy (**STEM**) detector →high resolution compositional contrast imaging, ideal for EDS mapping
2) Location

   Materials Characterization Core
   Room E119
   810 West Campus Drive
   West Haven, CT 06516

3) Primary Staff Contact

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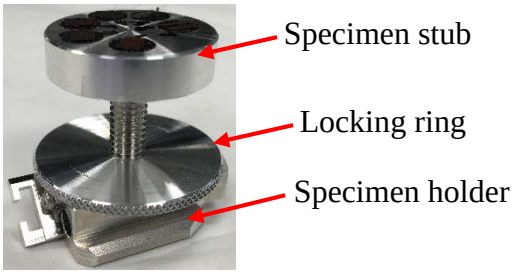
The Yale West Campus MCC Facilities are operated for the benefit of all researchers. If you encounter any problems with this facility, please contact the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact a staff member.

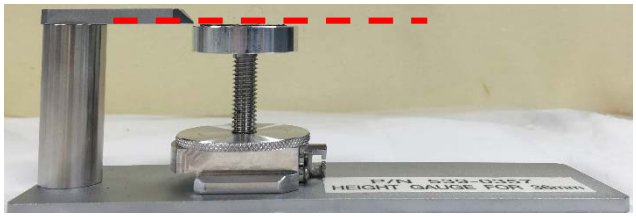
Notice: Please **follow** strictly the **SOP** to keep the facility under good condition. We **DO NOT** recommend user explorations on program unless endorsed by core manager.

2 Specimen Preparation[1]

1) **Always wear gloves** for vacuum sample preparation!! **Change gloves** if touched computer keyboard and mouse.
2) **The sample for SEM needs to be completely dried!**
   a) The **powders samples** can be dripped and dried on Si substrate. Alternately, powders can be **sprinkled** on **Conducting Graphite Paint** (supplied in the Core) directly applied on the specimen stub, or on to double sided conducting carbon tab.
   Note: Do not press the particles firmly as it may change their surface morphology.
   Note: The **Conducting Graphite Paint** is highly recommended to fix the samples especially **magnetic** particles for **high magnification** (>**100 k**) measurement.
   Warning: use **maximal** pressure **dry N_2** gas in the fume hood to **blow off loose particles** on powder samples before introduction into SEM chamber. **Loose particles will do damage to turbo pump in the specimen chamber (SC) and contaminate the vacuum including the lens system.**
   b) The solid samples, large size flakes, single crystals can be fixed directly onto the sample holder using **Conducting Graphite Paint**.



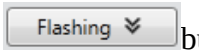
3) Attach the specimen stub to the specimen holder; **DO NOT** overtighten the locking ring.
4) Adjust the height of specimen so that the **highest point on the sample** matches the **lower surface of the height gauge**.
   a) **Caution**: if the paste at the edge of sample surpasses the sample surface, then align the paste to the height gauge.
   b) **Warning**: **Failure** to follow the instruction may lead to severe damage to the lens system, and the **repair fee** will be charged to PI's account.)



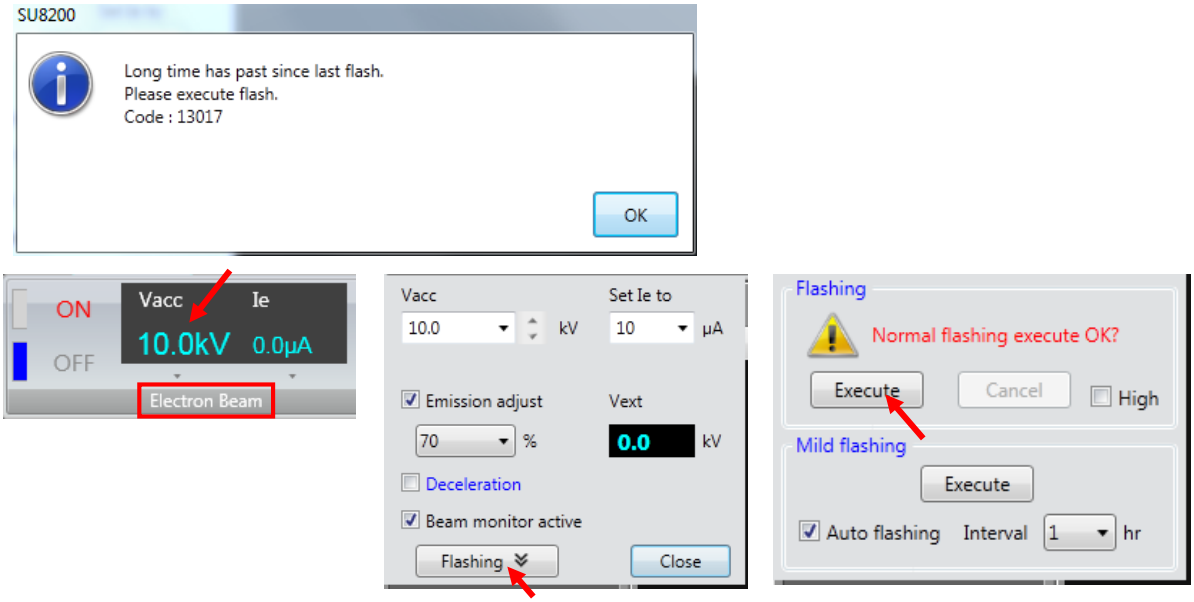
[1] **Always wear gloves** for your sample preparation in your own lab or in MCC! Warnings will be given for violations and the **user account will be revoked** after three warnings with notice to PI. Further training at PI's expense will be required to resume the account.

5) Bring the specimen stub inside the fume hood and blow off loose particles on the sample surface using the N_2 nozzle.[2]

3 Starting Instrument

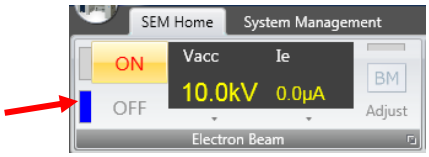
1) **Login** inside your reserved time box in **FOM calendar**.
2) **Sign in** on the **logbook** and put down date, usage time, sample materials, Specimen Chamber (**SC)** pressure, imaging modes (SEM, PD-BSE, STEM or EDS), and report any issues during measurement.
3) If the **PC_SEM** program is not open, click **PC_SEM** icon on desktop, choose or type **WC MCC** as profile name and hit **OK** button to login, no password required. (If the computer is logged off, then choose the profile **PC-SEM** and type **hitachi** to login.)
4) If a flashing message in yellow "**Execute Normal Flashing**" appears on top of the imaging window, click **OK** on the popup window, click the **Electron Beam** window and click 

button to open the Flashing window. Make sure the **Vacc** is **OFF (blue bar on)**, then click **Execute** button to flash the tip.



4 System Status Check

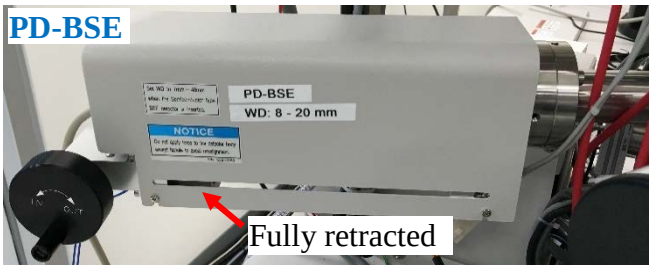
1) Check the **Electron Beam** window below: accelerating voltage **Vacc** should be **OFF** in the HV indication area with blue bar highlighted. If **Vacc** is **ON**, click the **OFF** button.

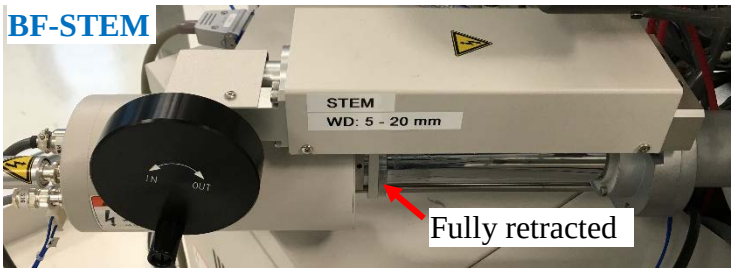


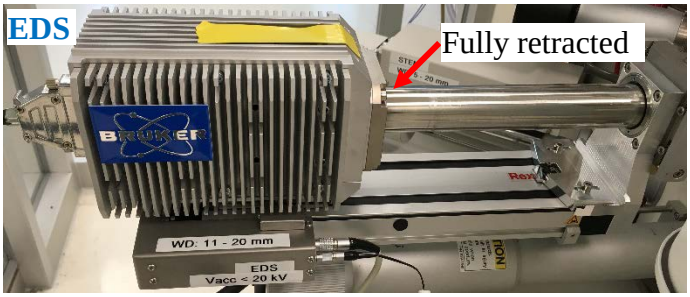
[2] **This step is crucial to keep the SEM chamber vacuum at good pressure,** which in turn improves the imaging resolution with less surface contamination and keeps the SEM lens system at good condition.

2) Turn on the Specimen Chamber **SC** chamber scope LCD (the switch is at the top left corner in the back)
   a) **Caution:** the specimen holder should be empty and at the exchange **EXC** position
   b) **Caution:** no other detectors (**PD-BSE** or **EDS**) underneath the pole piece.

3) Check if **PD-BSE**, **STEM** and **EDS** detectors are **fully retracted** outside the SEM chamber





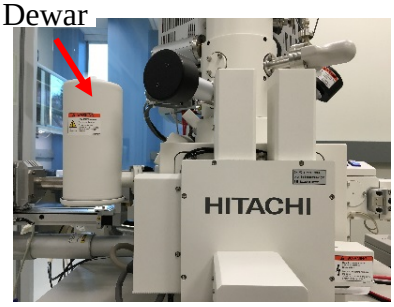


4) Check the Specimen Chamber **SC** pressure, which should read **LE-4 Pascal**. Fill the anti-contamination trap dewar with liquid nitrogen.[3]

[3] **Note:** this step is highly recommended for high magnifications >**100 k** or low accelerating voltages < **1 kV**.

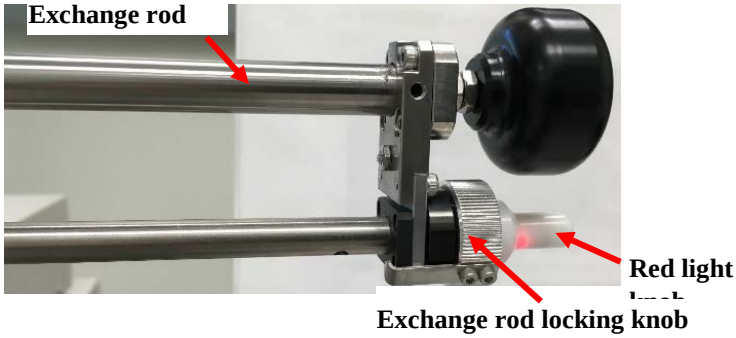
5) **Caution**: check the LN2 level momentarily during measurement to make sure the dewar is not **EMPTY**, otherwise the **quick outgassing** from the cold trap inside **SC** chamber will lead to pressure burst above **10^{-3} Pa** and **shutdown** the beam.

Dewar

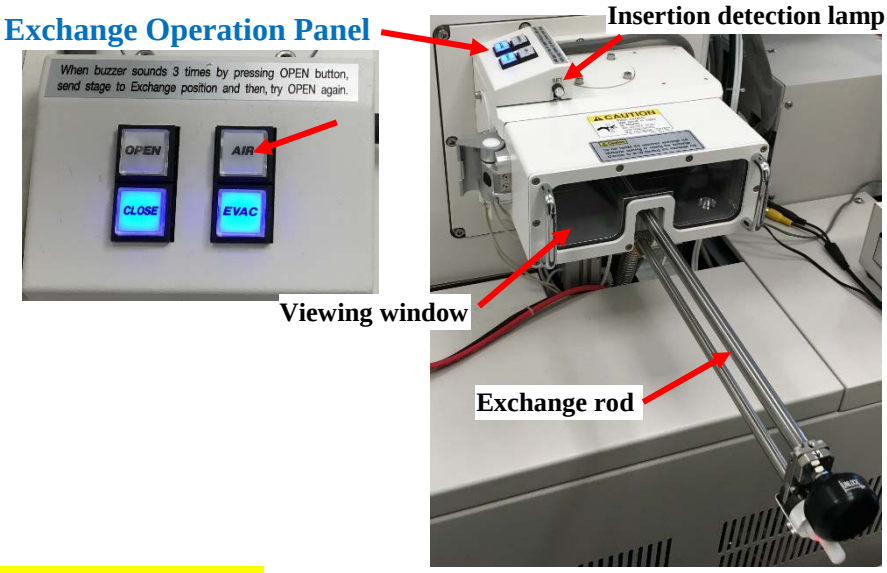


5 Loading the Specimen

1) Turn the **exchange rod locking** knob ***clockwise*** to lock the rod and make sure the **red light** on the locking knob is **ON**.



2) Press the **AIR** button on the **Exchange Operation Panel**. **Wait until** the buzzer sounds when air introduction into the specimen exchange chamber is complete.



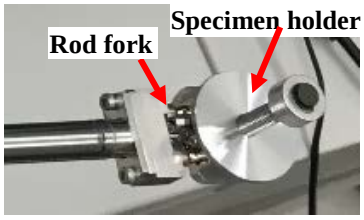
3) **Push with your thumb** to open the exchange chamber door.

   Caution: **DO NOT** hold the **exchange rod** to open the door, which will bend the rod with time and fail the sample transfer.

4) Insert the specimen stage onto the exchange rod
   a) Turn the **exchange rod** locking knob ***counterclockwise*** to release the rod, and push the rod to see the fork.

b) Turn the specimen holder **lock/unlock** knob ***clockwise*** to the **unlock** position and insert the rod fork into the holes of the specimen holder. Turn the knob ***counterclockwise*** to the **lock** position and confirm that the holder is locked to the rod.

Specimen holder knob (top view)

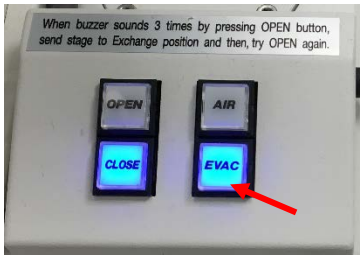


Warning: it is crucial to **Make Sure** that the sample holder is at the **Lock** position for sample transfer. **Violation** will lead to transfer failure and parts damage on the SEM stage.

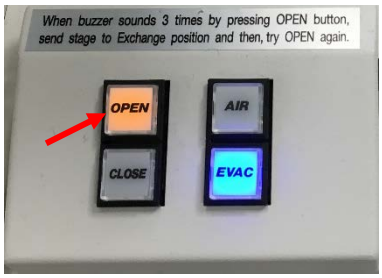
5) Pull the specimen **exchange rod** back into the airlock door and turn the exchange rod locking knob ***clockwise*** to lock the rod. The **red light** on the locking knob should be **ON**.

6) **Hold** and **Press** the **exchange chamber** to close the door. Continue **holding** the specimen exchange chamber and pressing the **EVAC** button on the **exchange operation panel**. **Wait until** the buzzer sounds indicating the chamber is evacuated back into vacuum
Caution: **DO NOT** use the exchange rod to close the door as this will lead to rod bending with time.

Exchange Operation Panel



7) Press **OPEN** button on the exchange operation panel. **Wait until** the buzzer sounds and the gate valve is open.

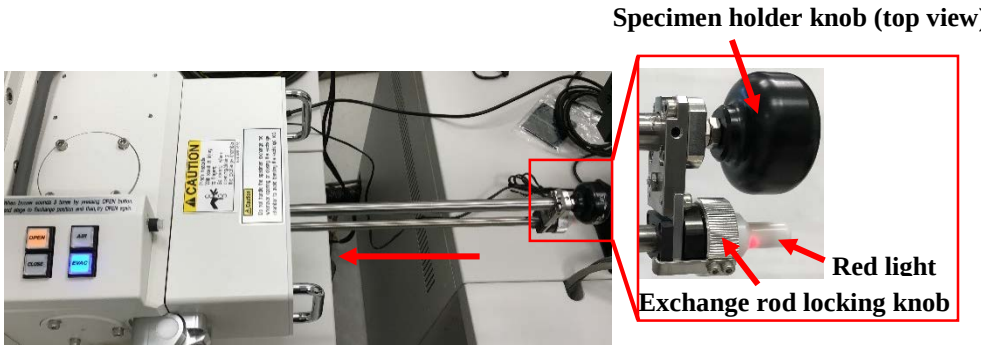


8) Turn the exchange rod locking knob ***counterclockwise*** to release the lock. Push the rod carefully into the **SC** chamber until the **insertion detection lamp** above the exchange chamber is lit in **blue**.

Warning:

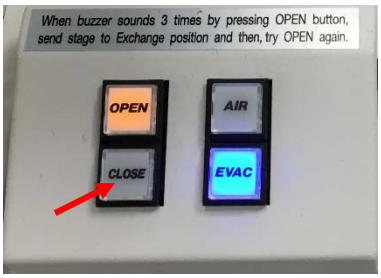
> **DO NOT turn** the **specimen holder knob** while pushing the rod into the **SC** chamber. This may cause accidental switch of **Lock** position to **Unlock** on the rod leading to sample transfer **failure** and mechanic **damage**.

> **Always Hold** and **Push** the **knob** during transfer to prevent rod **accidental sliding** into **SC** due to pressure imbalance between exchange and SC chambers.



9) Turn the **specimen holder lock/unlock knob** ***clockwise*** to **UNLOCK** position. Carefully retract the rod all the way to the back and turn **exchange rod locking knob** ***clockwise*** to lock the rod. The **red light** on the locking knob should be **ON**.

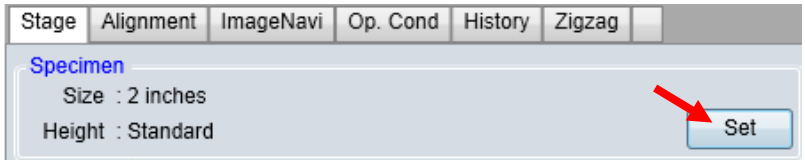
10) Press the **CLOSE** button on the exchange operation panel and wait until the buzzer sounds, indicating the sample transfer is complete.

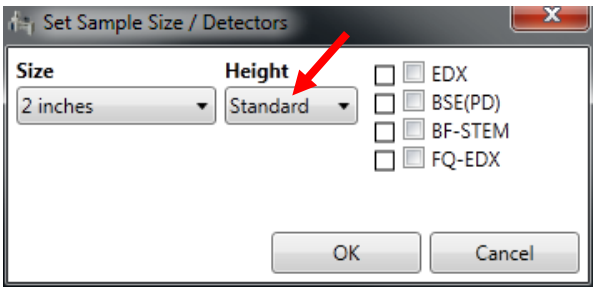


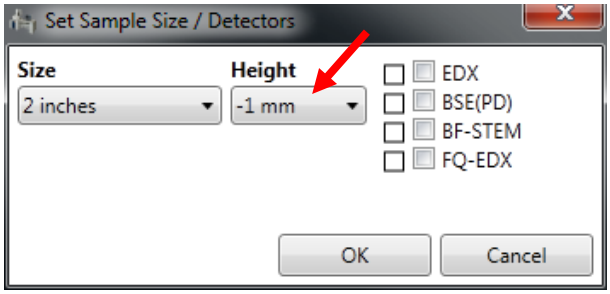
6 Image Observation

1) Click the **HOME** button on **PC-SEM** software top menu (**Caution**: **DO NOT** repeatedly click this button, which may lead to **STOP** button next ineffective). Use **SC** chamber scope LCD to see the sample holder moving from the exchange **EXC** position to **HOME** (measurement) position underneath the pole piece.

2) Click **Set** button in the **Stage** tab to set the specimen stub **Size** and **Height**
   a) **Caution**: for **safety** purpose, always choose the specimen stub **one size** up, e.g.: choose **2 inches** for **1 inch** specimen stub).[4])
   b) For **Height** setting, **Standard** is recommended with carefully adjusted sample height using height gauge.
   Warning: If sample height is slightly low than the height gauge, e.g. 1 mm, then -1 mm should be chosen.
   c) Check the boxes on right side if additional detectors will be in use. This will set up a safe **Z** movement range for detectors. To **AVOID damages to detectors**, please select **Z** within the range for different detection modes.

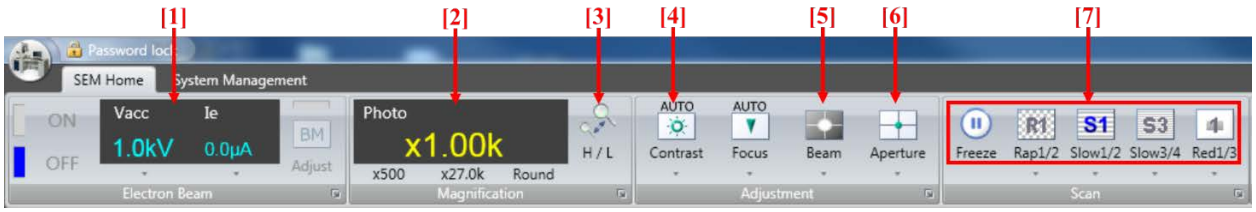






3) Confirm and set operating conditions.

[4] **Failure** to follow the instruction may lead to severe damage to the lens system; the user account will be revoked and the repair fee will be charged to PI's account.

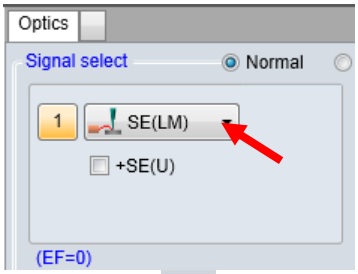


a) **[1]** Choose accelerating voltage **Vacc** (typical values: **1kV, 5 kV**, **10kV** or **15kV** for SEM imaging,
Note: always try small voltage first to avoid sample surface over-charging and **ebeam induced carbon deposition** (black imaging box)
Caution: **DO NOT** turn **Vacc ON** at this stage; and set the emission current **Ie** to **10 μA.**

b) Confirm the **Probe current** is checked at **Norm**.

c) Click **[3]** 

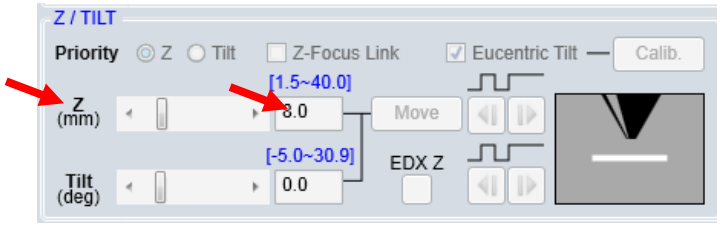
to choose **Lower Magnification** with **LM** appeared in window **[2]**, and make sure that the **SE(LM)** detector appears in **Optics** tab



d) Scan Mode: choose **Rapid Scan Mode** 

[7] to start with

e) Set **Z height** (**Caution: the smallest Z height allowed is 5 mm**.[5])

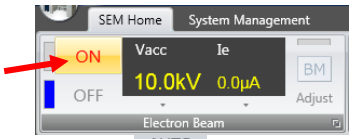


Z height setting restrictions (Severe damage to lens may happen with z < 5 mm):
Regular SEM: 5 – 20 mm
EDS: 11 – 20 mm
PD-BSE: 8 – 20 mm
BF-STEM: 5 – 20 mm

[5] **The Z height < 5mm** may lead to sample collision with lens. User's account will be suspended for damage caused by SOP violation and the repair expenses will be charged on user PI's account.

4) Check the Specimen Chamber (**SC**) pressure, which should read **LE-4 Pascal** on **EVAC CONTRO**L panel.[6]

5) Click the **ON** button to turn on **Vacc** after **SC** pressure reaches **LE-4 Pascal**.



6) Click **[4]** 

on PC_SEM window menu bar or **AUTO** button **[P-1]** on the **Manual Operation Panel** to adjust the image brightness/contrast. The **BIRGHTNESS** and **CONTRAST** knobs can be used separately to do manual adjustment.

Manual Operation Panel

7) Roll the track ball on the **STAGE CONTROLLER** to find the field of interested in LM mode:

[6] **It is crucial to wait until the Specimen Chamber (SC) pressure is LE-4 Pa before turning on HV.** This usually takes up to **5 minutes** after sample transfer, and **2 minutes** with liquid nitrogen in the dewar. **Turning on HV in bad SC pressure** higher than LE-4 Pa will affect imaging resolution and shorten filament lifetime. Warnings will be given for violations and the user account will be revoked after three warnings with notice to PI. Further training at PI's expense will be required to resume the account.

a) Adjust magnification with the **MAGNIFICATION** knob **[P-3]** on the **Manual Operation Panel**.

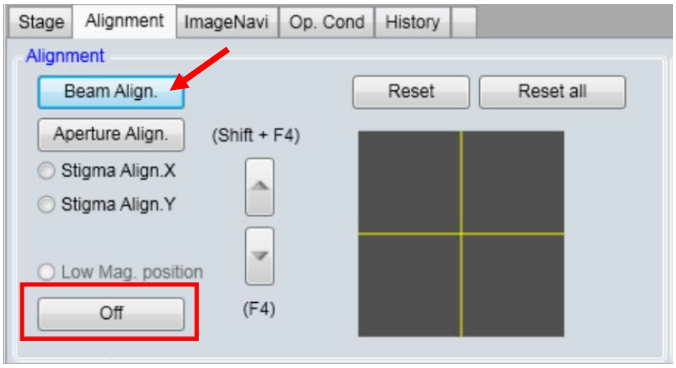
b) Adjust focus using **FOCUS COARSE** and **FINE** knobs **[P-4]**. Move the stage to look for the field of interest in **LM** mode, and then click **[3]** 

to switch to **High Magnification (HM)** mode.

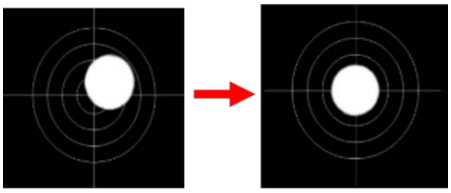
8) In **HM** mode, change the magnification and adjust **FOCUS** knob on **Manual Operation Panel**

a) If image **drifts** (swaying or heaving):

> Click **Alignment** tab and click **Beam Align** button. The **ALIGNMENT** LED on the **Manual Operation Panel [P-2]** should be **ON**. Bring the circular image to the center of the image area by adjusting **X** and **Y** knobs on **[P-2**.
Notice: skip this step if **Vacc** is not changed



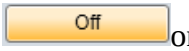
Beam alignment



Beam alignment image

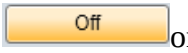
> Click **Aperture Align** button below and adjust **STIGMA/ALIGNMENT X /Y** knobs **[P-2]** to minimize the wobbling motion in image

> Click **Stigma Align X/Y** button below and adjust **STIGMA/ALIGNMENT X /Y** knobs **[P-2]** to minimize the wobbling motion in image

> Click 

on **Alignment** tab

b) If image **distorts** (stretching), correct **astigmatism**:

> Make sure the **Alignment** LED **[P-2]** is **OFF**, otherwise 

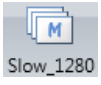
on **Alignment** tab

> Use the **STIGMA/ALIGNMENT X /Y** knobs **[P-2]** alternating with **FINE FOCUS** knob **[P-4]** to reduce distortion and obtain the sharpest image.

c) **Repeat** steps a) and b) at **each high magnification**

9) Select the field of view, confirm image with slow scan 

or 

and then, click the **Capture** button 

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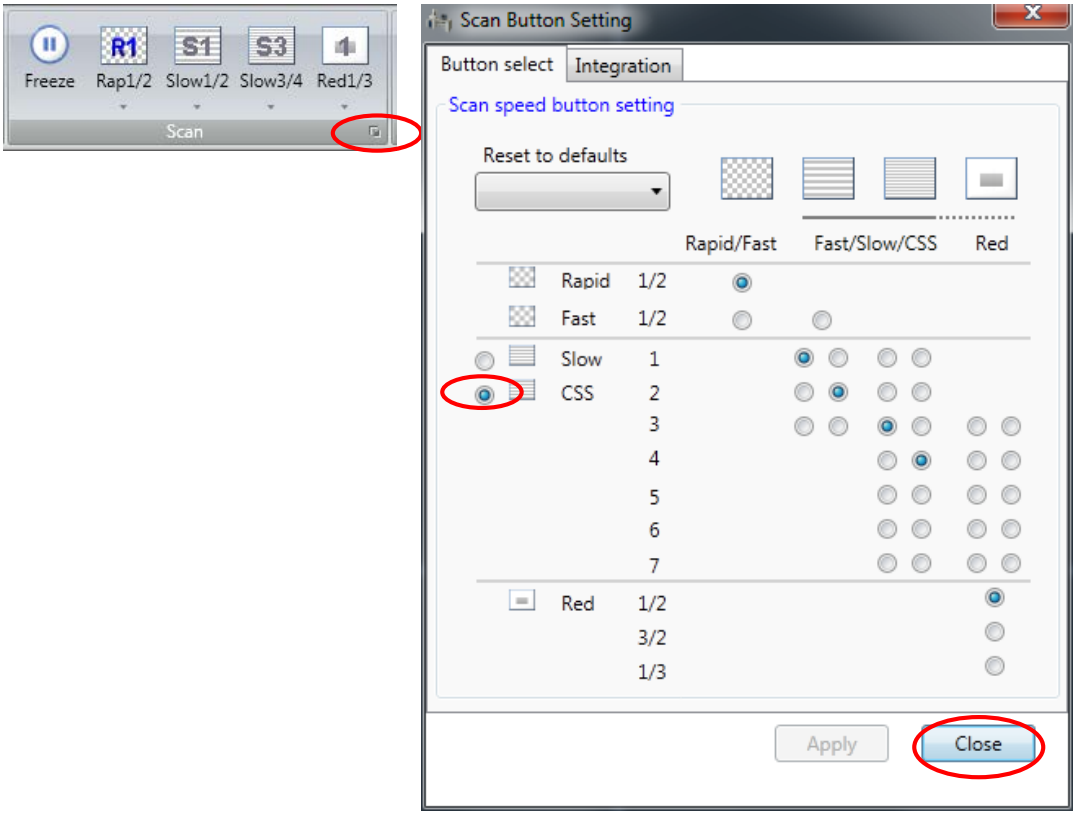
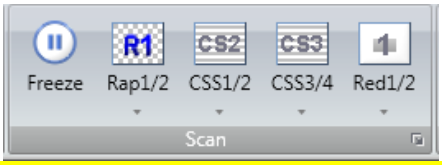
10) Image capturing settings for charging samples:

> Choose **CSS** (Charge Suppressed Scan) mode:

a) Clicking on the small box in the **Scan Menu** to open the Scan Button Setting window. Check the radio button next to CSS.
b) Hit **Apply** button and **Close**.

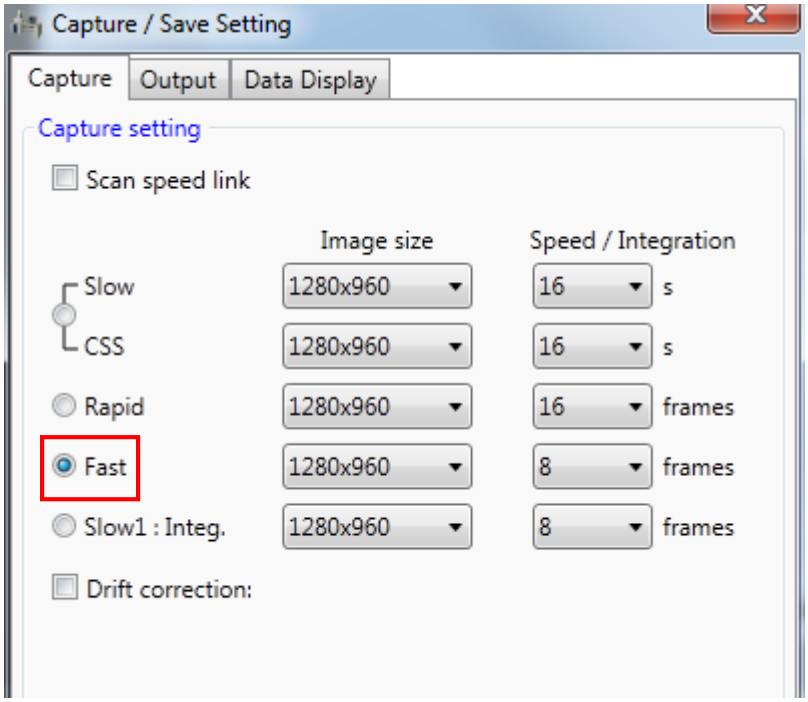


c) The CSS Scan menu should appear as follows:



d) If the charging is still strong on the surface, change the scan mode from line scan to frame mode:

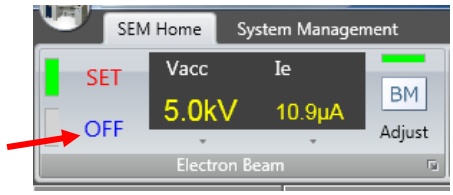
> Click the small box by Capture menu to the Capture/Save Setting window. Choose **Fast** capture mode with 8 frames.



11) To save data, click **ALL** button on the bottom of the image thumbnail column and click →**PCI** button, the collected images will be transferred into the **Quartz PCI** program
12) In **Quartz PCI** program top menu bar, click **File** and select **Export All…** on the dropdown menu, then choose export path and file format.

7 Closing SEM measurement

1) Click the **OFF** button to turn off **Vacc**.



2) Fully **retract** **PD-BSE** or **EDS** detectors first if used.
3) Click the **EXC** button on **PC-SEM** software top menu to move the specimen stage to the exchange position.

4) To take out the specimen from the specimen chamber, follow the **reversed** order from sample insertion:
    a) Press **OPEN** button on the exchange operation panel. **Wait until** the buzzer sounds and the gate valve is open.
    b) Turn the **exchange rod locking knob** ***counterclockwise*** to **release** the lock. Push the rod carefully into the **SC** chamber until the **insertion detection lamp** above the exchange chamber is lit in **blue**.
    c) Turn the **specimen holder lock/unlock knob** ***counterclockwise*** to **LOCK** position. Carefully pull out the rod all the way to touch the **exchange rod locking knob** and turn **exchange rod locking knob** ***clockwise*** to lock the rod. The **red light** on the locking knob should be **ON**.
    d) Press the **CLOSE** button on the exchange operation panel and **wait until** the buzzer sounds, indicating the sample transfer is complete.
    e) Press the **AIR** button on the **exchange operation panel**. **Wait until** the buzzer sounds when air introduction into the specimen exchange chamber is complete.
    f) **Push with your thumb** at highlighted spot to open the exchange chamber door.
    g) Turn the exchange rod locking knob ***counterclockwise*** to **release** the rod, and push the rod out of the open airlock door
    h) Turn the specimen holder **lock/unlock** knob ***clockwise*** to **UNLOCK** position and remove the specimen stage from the exchange rod.
    i) Pull the **specimen exchange rod** back into the airlock door and turn the **exchange rod locking knob** ***clockwise*** to lock the rod. The **red light** on the locking knob should be **ON**.
    j) Press to close the specimen exchange chamber door, **hold the door and press** the **EVAC** button on the **exchange operation panel**. **Wait until** the buzzer sounds indicating the chamber is evacuated.
5) **Turn off** the SC **chamberscope LCD**.
6) **Leave the** PC-SEM program **ON**

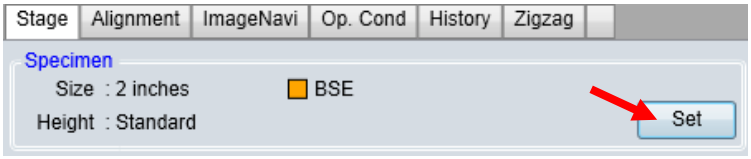
7) **Use ONLY** the core USB flash drive to transfer data from SEM computer to the workstation in the core, and then use either your own USB flash drive or internet to retrieve data.
8) **Log off** in your reserved time box in **FOM calendar** off the SEM monitor.

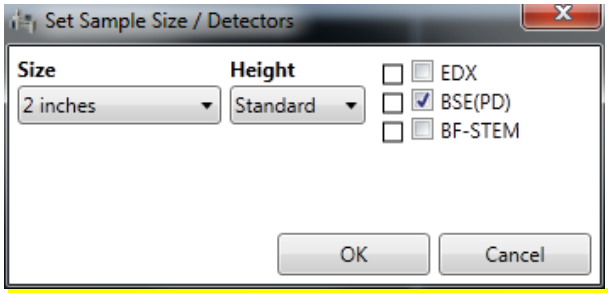
8 Checklist after Experiment

1) **Sign off** the **logbook** and report any problems.
2) **Remove** samples from the stub on the specimen holder, and **clean** the holder with Kimwipes using Methanol/IPA.
3) **Store** the specimen holder in assigned organizer box.
4) **Clear** the SEM work bench.

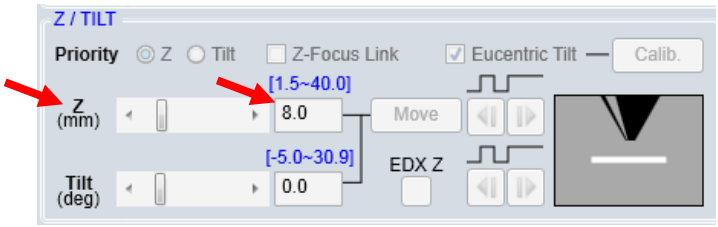
9 Photodiode Back Scattered Electron (PD-BSE) detection

1) Make sure **Vacc** is **OFF**
2) In **PC_SEM** program with the sample holder at **EXC** or **HOME** position, click **Set** button in the **Stage** tab and check the **BSE** box





3) **To AVOID damages to detectors, choose Z range: 8-20 mm, typically 8 mm**



4) Select **Vacc ≥ 15 kV** and regular **Ie=10 µA** as the **PD-BSE** requires high e-beam kV
5) Select **Dual Screen** mode on the **PC_SEM** top menu bar, and choose 

detector for the first screen and 

detector the second

6) Switch to **LM** mode and turn **Vacc ON**
7) **Make sure the Z ≥ 8 mm, crank (move) slowly** the **PD-BSE detector** to the measurement position
   Caution: cranking slowly prevents the vibrations of detector and **SC** chamber, **stop** cranking once feel stopped
8) Monitor the movement of **PD-BSE** detector until it stops between sample and lens on chamber scope **LCD** screen and then **TURN OFF LCD** screen
   Warning: the **PD-BSE detector** is very **sensitive** to ambient light; the **LCD** screen must be **turned off** before PD-BSE imaging
9) Click 

beside 

to activate **SE** imaging:

a) Find interested areas on the sample inside the **annular PD-BSE** detector in **LM** mode; adjust focus and switch to **HM** mode.
b) Get a **GOOD** image in **HM** mode
Caution: **Always** switch to **SE** window to adjust image quality for **PD-BSE** imaging

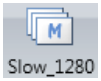
10) Click 

beside to activate **BSE** imaging:

a) Click or **AUTO** button on the **Manual Operation Panel** to adjust the image brightness/contrast. The **BIRGHTNESS** and **CONTRAST** knobs can be used separately to do manual adjustment

b) Select the field of view, confirm image with slow scan 

or 

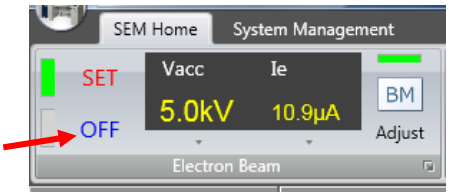
and then, click the **Capture** button 

.

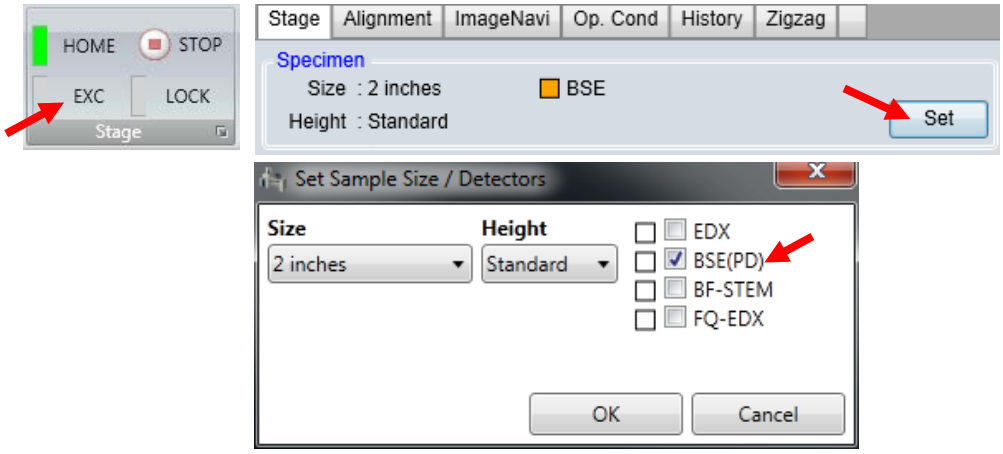
Notice: **do not** use 

rapid scan mode for **PD-BSE** imaging

11) To quit **PD-BSE detection** mode:
a) Switch to **LM** mode
b) In **PC_SEM** program, click the **OFF** button to turn off **Vacc**



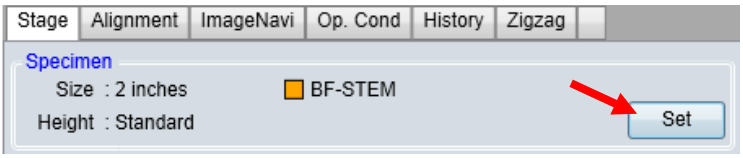
c) Turn the chamber scope **LCD ON**
d) **Crank to fully retract the PD-BSE detector**
e) Click the **EXC** button to move the specimen stage to the exchange position. Click **Set** button in the **Stage** tab; uncheck **BSE(PD)** box, and then following regular SEM instructions to take the sample out from chamber.

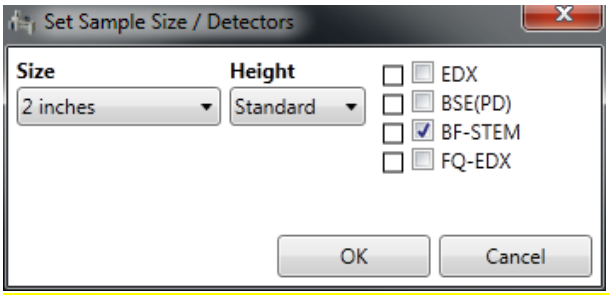


10 Scanning Transmission Electron Microscopy (STEM) detection

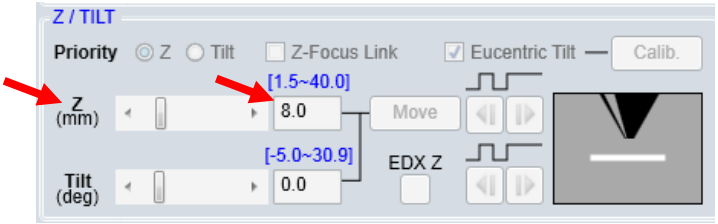
Notice: The STEM sample holder has standard **36 mm**, so **NO Height Gauge** is required.

1) Make sure **Vacc** is **OFF**
2) In **PC_SEM** program with the sample holder at **EXC** or **HOME** position, click **Set** button in the **Stage** tab and check the **BF-STEM** (Bright Field) box





3) To **AVOID damages** to STEM detector**,** choose **Z range: 8-20 mm,** typically **8 mm** (especially if coupled with **EDS mapping** to avoid EDS detector damage**)**

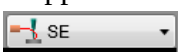


4) Select **Vacc ≤ 20 kV** and regular **Ie=10 µA** in **STEM** **(especially if coupled with EDS mapping to avoid EDS detector damage)**
5) Select **Dual Screen** mode on the **PC_SEM** top menu bar, and choose 

detector for the first screen and 

detector the second

6) Switch to **LM** mode and turn **Vacc ON**
7) **Make sure the Z ≥ 8 mm, crank (move) slowly** the **STEM detector** to the measurement position
   Caution: cranking slowly prevents the vibrations of detector and **SC** chamber, **stop** cranking once feel stopped
8) Click 

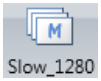
beside 

to activate **SE** imaging:

a) Find interested areas on the sample in **LM** mode; adjust focus and switch to **HM** mode.
b) Get a **GOOD** image in **HM** mode
Caution: **Always** switch to **SE** window to adjust image quality for **STEM** imaging

9) Click  beside  to activate **STEM** imaging:

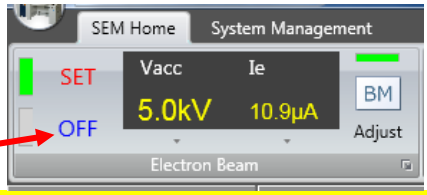
a) Click  or **AUTO** button on the **Manual Operation Panel** to adjust the image brightness/contrast. The **BIRGHTNESS** and **CONTRAST** knobs can be used separately to do manual adjustment

b) Select the field of view, confirm image with slow scan  or  and then, click the **Capture** button  .

Notice: **do not** use  rapid scan mode for **STEM** imaging

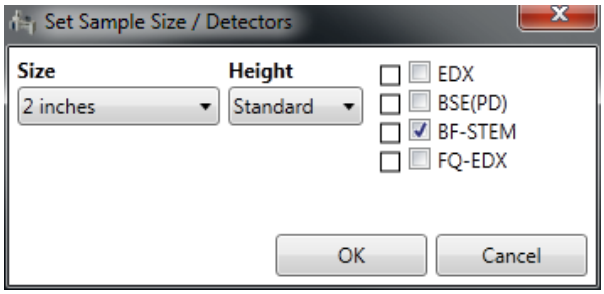
10) To quit **STEM detection** mode:

a) In **PC_SEM** program, click the **OFF** button to turn off **Vacc,** switch **Vacc** back to **15 kV**



b) Crank to fully retract the STEM detector

c) Click the **EXC** button to move the specimen stage to the exchange position. Click **Set** button in the **Stage** tab; uncheck **BF-STEM** box, and then following regular SEM instructions to take the sample out from chamber.



11 Energy Dispersive X-ray Spectroscopy (EDS)

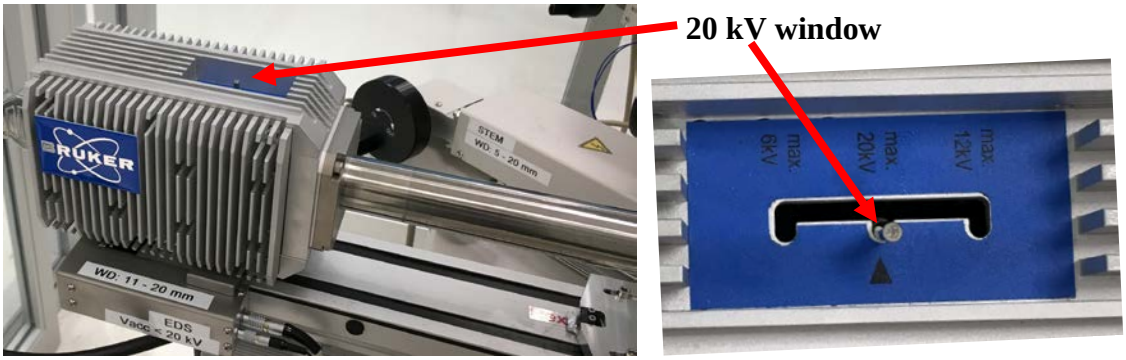
Both regular **SE** (Secondary Electron imaging) sample holders and **STEM** (Scanning Transmission Electron Microscopy) holders can be used in **EDS** mode. The **EDS mapping** resolution can be improved to **sub-µm** on **STEM** samples.

1) Make sure **Vacc** (e-beam accelerating voltage) is **OFF** (check in PC_SEM program)
2) On PC_SEM top menu bar, **set Vacc to 15 kV**
   Note: Vacc can be set to smaller voltages if light elements (Si, Ti…) or intermediate elements (Cu…) are interested only. **Contact Core manager (Min Li)** for detailed instruction.

Warning: It is extremely important to choose the Vacc < 15 kV in EDS mode. The EDS detector will be burned once exposed to high kV e-beam. Violation will lead to detector damage and repair charge will be applied to user's PI account.

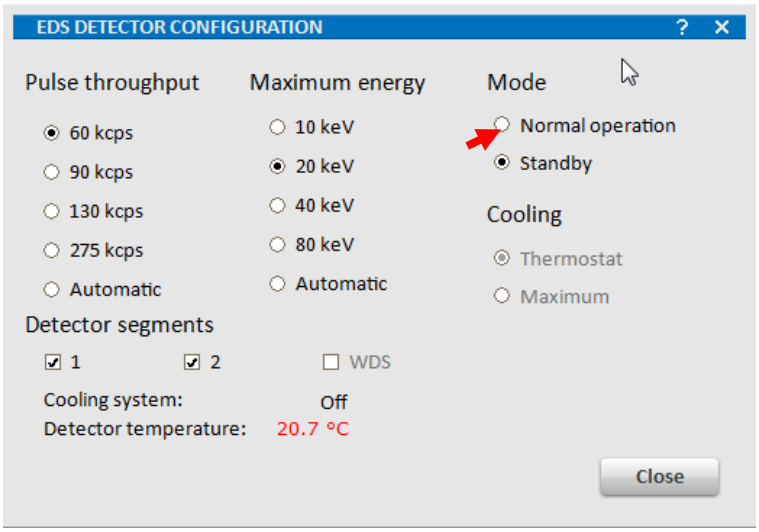
3) Log into **EDS** monitor through FOM Screen Locker or user's own FOM account.
   a) The EDS software **Esprit** window should be always kept **ON**.
   b) In case the Esprit program was closed and the EDS computer was logged off, select the profile **PC-SEM** with password **hitachi**
4) Check EDS detector window position and **make sure it is set at 20 kV:**

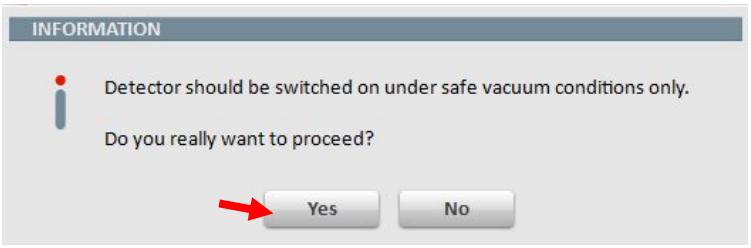
Warning: It is extremely important to set the detector window at 20 kV with the thickest window protection from e-beam damage. Violation will lead to detector damage and repair charge will be applied to user's PI account.



5) Switch **EDS** detector in operation mode and set up parameters:
   a) In **EDS Esprit** operating program, click the triangle in the **EDS** tab at the bottom left corner to open the **EDS DETECTOR CONFIGURATION** window, check **Normal operation**, read the **INFORMATION** window and hit **Yes** only if **SC** pressure is at **LE10-4 Pascal** and the sample is inside main chamber.
   Warning: EDS detector cooling in low vacuum such as during and right after sample transfer will lead to detector contamination.

b) Select **60 kcps** in **Pulse throughput** and **20 keV** in **Maximum energy** if SEM beam energy is above **10 keV**. The **Cooling** setting should be on **Thermostat.**



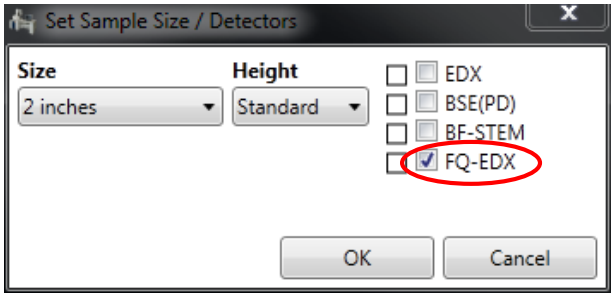


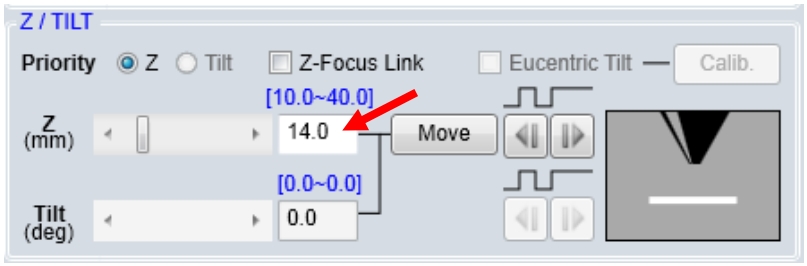
c) Close both **INFORMATION** and **EDS DETECTOR CONFIGURATION** window.

d) Watch the EDS detector **Temperature** reaches the operating temperature of **-20 ± 0.5 °C** before turning on the SEM beam.
Warning: earlier E-beam switch on may cause damage to the EDS detector.

6) In **PC_SEM** program with the sample holder at **HOME** position, click **Set** button in the **Stage** tab and check the **FQ-EDX** box. The stage **Z position** should move to the default height at **14 mm**.

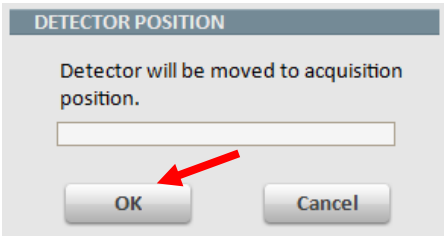
Warning: DO NOT change the **Z position < 11 mm**. This will cause the sample holder crashing into the **EDS detector**. This **severe SOP violation** will lead to user account suspension and charge on PI's account.





7) Check the working distance (**W.D.**) in **PC_SEM** and make sure it is set above **11 mm**. If not, change it in **High Magnification** mode.

8) In EDS **Esprit** program and click  button; click **OK** on **DETECTOR POSITION** window to move the detector to the acquisition position right above sample.



9) Check EDS detector and **make sure** it is at operation temperature **-20 ± 0.5 °C**

10) In **PC_SEM** program, **make sure the Vacc is set to or below 15 eV**

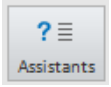
   a) Switch **Vacc ON** and change the emission current **Ie** to **30 µA**

   b) Change the **Probe current** to **High**.

   c) Choose either **SE** or **STEM** (if STEM holder is being used) detector

d) Select interested area, adjust image quality (focus, stigma) and start SEM scan on **Rapid Scan Mode** 

11) **Turn off** the chamberscope from the back of the monitor.
Note: This step is crucial, or **the EDS detector will be flooded by ambient signals** leading to fat peaks in spectra.

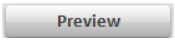
12) EDS scan
Switch to **EDS Esprit** program and select interested scan modes (Spectra, Object, Line scan and Mapping). **Always click** 

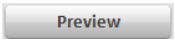
for detailed instruction

a) **Spectrum Acquisition Mode (always perform this mode for dead time testing before moving on to other modes**):
Note: this mode should be chosen only if the entire imaging area is **homogeneous**, otherwise use the **Objects mode** below instead.

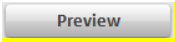
> Click 

on the left side menu to enter **Spectra** workspace

> Click 

button to acquire live spectrum. To stop preview, hit 

again.

> With **Preview** mode on, check if the **Dead time** is ~**30 – 50%** for regular samples. If not, stop preview, go to PC_SEM, increase (dead time increases) or lower the SEM emission current, adjust focus and stigma and click 

to check again.

Note:

> **Dead time** adjustment using Spectrum Acquisition mode must be performed prior to other EDS modes.

> The dead time on STEM samples is usually lower than **30%**, so no need to adjust.)

Warning: Dead time above 50% could lead to signal pile-up to induce false peaks in EDS.

> Click 

button to start spectral scan

> To add collected spectra into **project** or **report**, hit the 

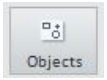
button on the top right corner of the spectral workspace

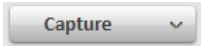
> To save the data in Bruker spectra format (***.spx**) or export to ***.txt** or ***.xlsx** format, click the lower 

button.

b) **Objects Mode** (spectrum acquisition from objects in image):
Note: this mode should be chosen for **inhomogeneous** surface spectral analysis.

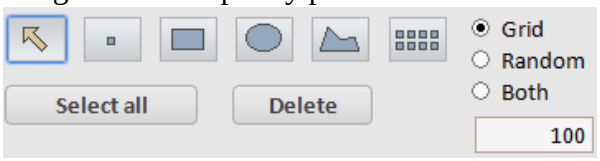
> Adjust Dead time to be ~ ~**30 – 50%** in **Spectrum Acquisition Mode**

> Hit 

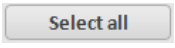
on the left side menu to enter **Object** workspace and hit 

button to capture an image.

> Select the desired object type on the bottom menu bar and click on captured image above to specify positions.



Note: the EDS lateral resolution is ~ **1 µm** which is variable on **Vacc** (e-beam accelerating voltage), choose **Point** object mode  if the interested feature is too small. Allow longer collection time if the Point or small square or circle objects are chosen. Generally check

> Click 

to highlight all objects and click 

> To save object data, click the 

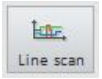
button on the top right corner of the workspace window.

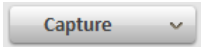
> To save the spectrum, click the lower spectrum chart 

.

c) **Line Scan Mode**:

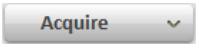
> Adjust Dead time to be ~ ~**30 – 50%** in **Spectrum Acquisition Mode**

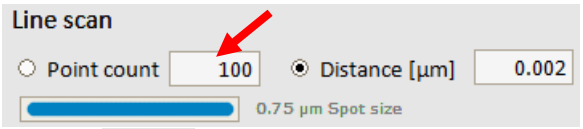
> Hit 

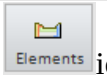
on the left side menu to enter **Line Scan** workspace and hit 

button to capture an image.

> Highlight the line and drag and adjust the endpoints to the desired position

> Set **Point count** of the line scan and click 



> Use the 

icon to identify elements

> To save line scan data, click the 

button on the top right corner of the workspace window.

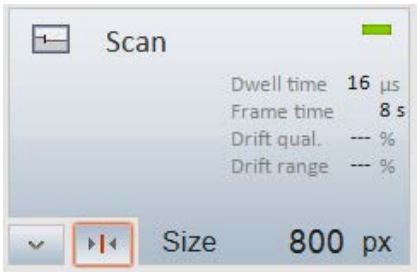
> To save the profile, click the lower profile chart 

.

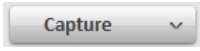
d) **Mapping Mode**:

> Adjust Dead time to be ~ ~**30 – 50%** in **Spectrum Acquisition Mode**

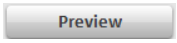
> Click  button on **Scan** tab to activate image drift correction. Make sure the button is highlighted in **red** to enable **drift correction**.

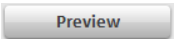


> Hit 

on the left side menu to enter **Mapping** workspace and hit 

button to capture an image.

> Click 

button and adjust image; hit 

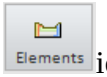
again to stop preview

> Click 

button to start **Mapping**. To stop **Mapping**, click 

again.

Note: typical data collection time depends on signal intensity (**5 mins ~ 15 mins** or even longer). Before stop mapping, hit **Spectrum** tab on the far right side of **Map** tab and check if the interested elemental peaks have good S/N ratio.

> Use the 

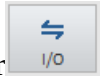
icon to identify elements

> To save map data, click the 

button on the top right corner of the workspace window.

> To save the map image, click the lower image window 

.

> To save individual element image in the thumbnail on the bottom, click the thumbnail bar 

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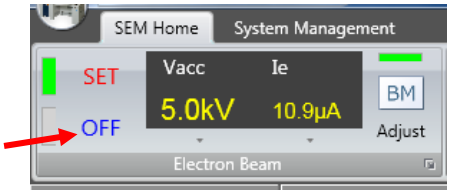
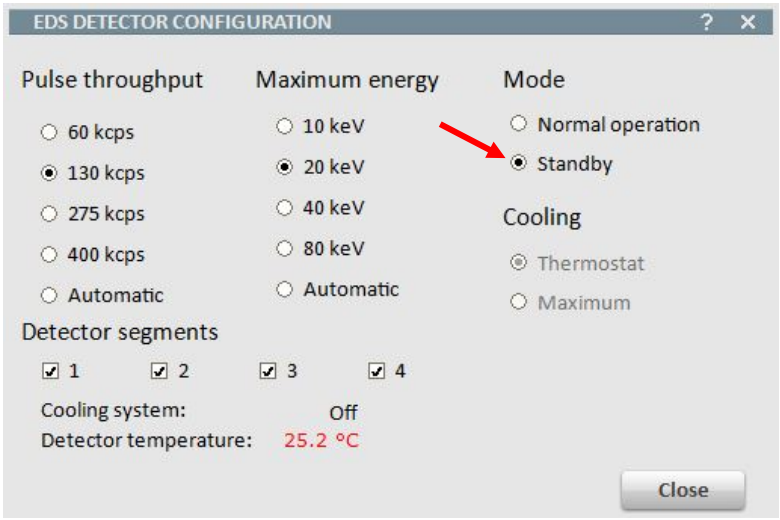
13) To quit **EDS detection** mode:

a) In **PC_SEM** program, click the **OFF** button to turn off **Vacc**, change **Vacc** back to **10 kV** and the **Ie** back to **10 µA**

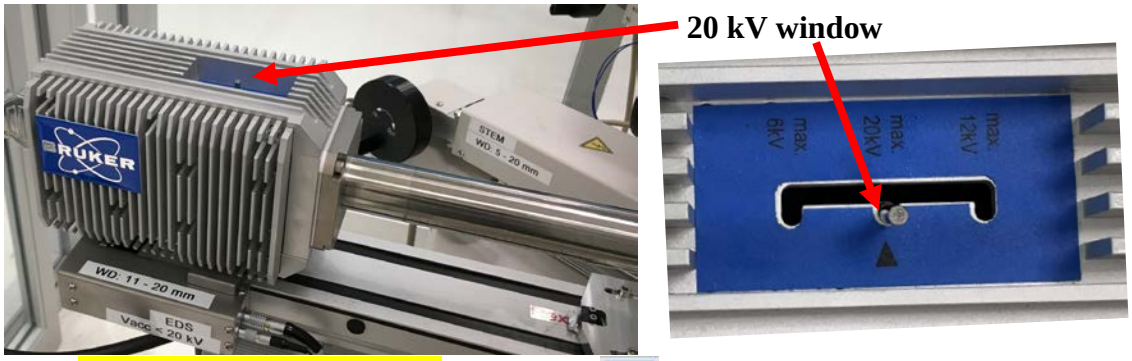


b) Click **Standby** button in **EDS DETECTOR CONFIGURATION** window to switch the EDS detector to **Standby Mode**



c) Check EDS detector window position and **make sure it is at 20 kV:**

Warning: It is extremely important after EDS to make sure the detector window is set at 20 kV with the thickest window protection from e-beam damage. Violation will lead to detector damage and repair charge will be applied to user's

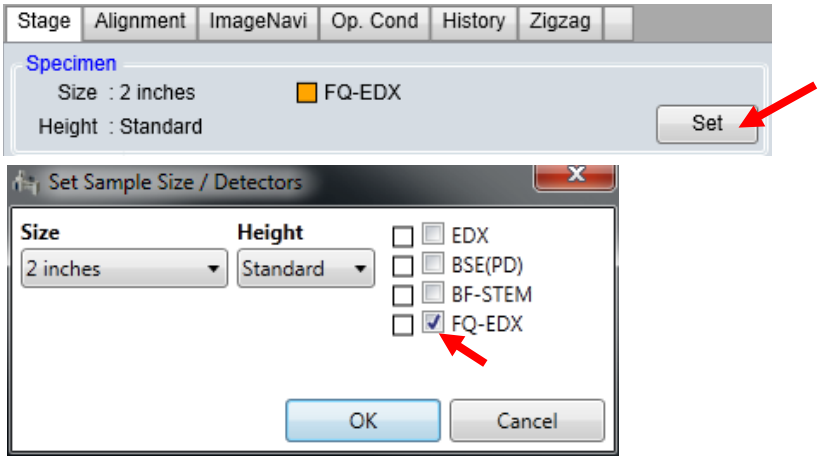


d) **Fully retract EDS detector** by clicking  in **EDS Esprit** program

e) Change the **Probe current** back to **Norm**.

f) In **PC_SEM** program, click the **EXC** button to move the specimen stage to the exchange position.

g) Click **Set** button in the **Stage** tab; uncheck **EDX** box, and then following regular SEM instructions to take the sample out from chamber.



h) Before sample transfer, wait the EDS detector temperature rises above **0 ˚C**.
Warning: sample transfer while EDS detector is still being cooled below **0 ˚C** will induce contamination on the detector when the Specimen Chamber is open.

i) **Log off** SEM computer in your FOM account while the EDS detector warms up

j) Finish sample transfer

k) **Turn off Chamberscope LCD**

l) Keep **Esprit** program **ON** and **Log off** EDS computer on screen

m) Clear the work bench and dispose **gloves**, Kimwipes…