# **Standard Operating Procedure I:** SEM, BSE and STEM





ESC II, Room A119F 810 West Campus Drive West Haven, CT 06516

- > **FOLLOW the SOP strictly** to keep the instrument in good condition. Any violation will lead to user account suspension.
- > **NEVER** use your own USB drive on instrument computer. Data can be transferred with the Jump Drive provided by the Core.
- > **NEVER** surf the web on the instrument computer to minimize the risk of the computer being hacked.
- > **NEVER** allow other users to get access to instrument computer on your reservation.
- > **REPORT** any issues to Core director immediately so they can be fixed on time.

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# **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: high signal intensity from backscattered electron
  - > Scanning Transmission Electron Microscopy (STEM) detector: high resolution compositional contrast imaging, ideal for EDS mapping
- 2) Location

Materials Characterization Core Room A119F 810 West Campus Drive West Haven, CT 06516

3) Primary Staff Contact

Dr. Min Li Tel: 203-737-8270 Email: min.li@yale.edu Office: ESC II, Room A119D

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.

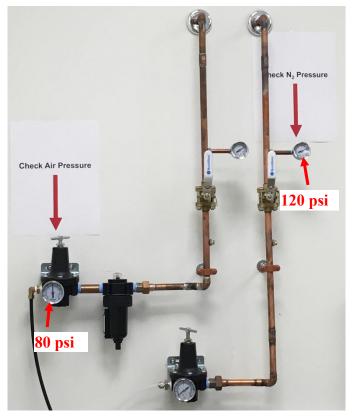
**Warning**: Please **follow** strictly the **SOP** to keep the facility at good condition. We **DO NOT** recommend user explorations on program unless endorsed by core director.

- 2 Initial System Status Check
  - Check if the small SC LED is flashing and the Specimen Chamber (SC) pressure reads LE-4 Pascal. If not, stop and report to Core manager immediately.

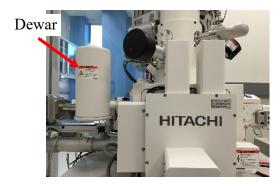


 Check building supplied Compressed Air (CA) and Nitrogen (N2) pressure on the wall, make sure the CA pressure reads around 80 psi and N2 around 120 psi. If both gauges read zero, contact Core Manager immediately.

Warning: Operating without compressed air and nitrogen will lead to system damage.



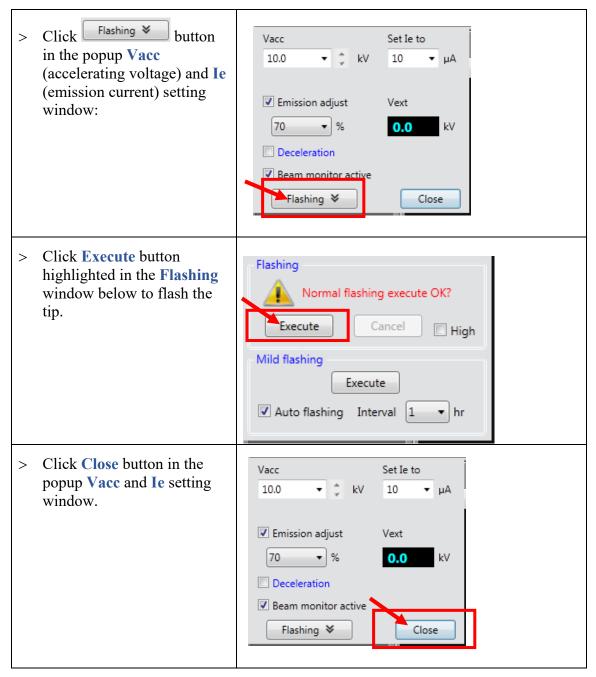
 Check the LN2 dewar below and make sure it is high enough to support the measurement. A full tank can support ~1 hour so keep checking LN2 in every half hour to avoid pressure rise to shut down the electron beam.
 Warning: when opening the LN2 storage tank on the ground, pull the lid slowly out of the tank. Do Not shake the lid which could break the foam cylinder attached to the lid.



# 3 Starting Instrument

- 1) 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.
- 2) Unlock the SEM computer using FOM calendar only. The FOM screen lock window NetID login is not working.
- 3) If the PC\_SEM program is closed, 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:

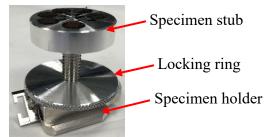
>	Click <b>OK</b> on the popup window on the right side	SU8200 Long time has past since last flash. Please execute flash. Code : 13017
>	Click inside the <b>Electron</b> <b>Beam</b> window as highlighted in red	ON Vacc Ie 10.0kV 0.0µA Electron Beam



## 4 Sample Preparation

- 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 tape. **Note:** 
    - **Do not press powders too hard** as it may change surface morphology.

- The **Conducting Graphite Paint** is highly recommended to fix the samples especially **magnetic** particles for **high magnification** (>100 k) measurement.
- 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** (see photo below).

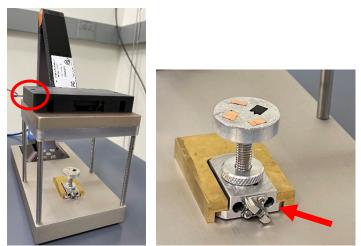
Warning: Samples mounted above the dashed line will crash into the lens system or detectors (EDS, STEM), and the repair fee will be charged to PI's account.)



5) Clean sample holder: bring the specimen stub inside the fume hood and blow off loose particles on the sample surface using the N<sub>2</sub> gun.

# 5 Taking Navigation Photo

1) **Wear gloves** and fully push to insert the sample holder base into the Navigation Camera slot with two banana pin holes facing outwards as highlighted below:



2) Turn on the camera light at the top left corner of the stand as circled above.

3) Go back to PC-SEM program and click **CameraNavi**, the last tab on the right side of the window as shown below and Click the Movie button at the bottom:

	Alignment	ImageNavi	Op. Cond	History	Zigzag	CameraN	
Page		Movie	Su	b-Screen		Alignme	
Page	Delete	Movie	Su	b-Screen	Scanı	Alignme	
Page X: 0.0		Movie	Su Horiz: 1				fram
X:0.0			Horiz: 1			ning image f	fram

4) Click the Movie button in above window to take sample holder video as shown below: (The Movie button was replaced by the Capture button).



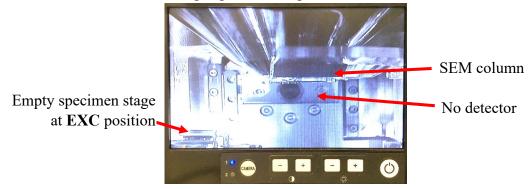
5) Ignore the **Navigation not possible** message in the window below. It will disappear when the sample holder is at **Home** position in the **SC** chamber and the **HV** is turned on. You may **Delete** the picture below and replace the sample holder and restart **Movie** if needed.



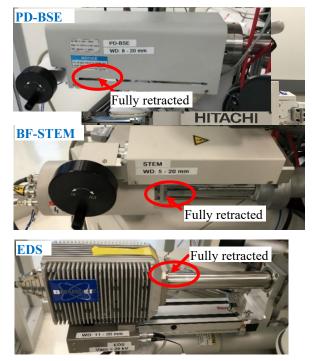
6) Turn off the camera light.

## 6 Sample Loading

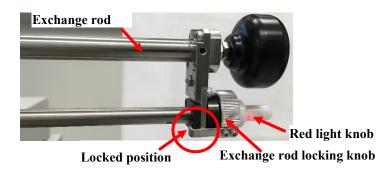
- 1) Remove the sample holder from the **Navigation Camera** base and place it on the SEM table.
- 2) Turn on the small LCD on the table by pressing the switch at the **top left corner** in the back. This unit includes the Specimen Chamber (**SC**) camera display and the camera power supply.
  - > Check and make sure the specimen stage is at the exchange EXC position and no sample holders on the stage, as highlighted in the picture below:
  - > Check and make sure no other detectors (PD-BSE or EDS) underneath the electron column, as highlighted in the picture below:



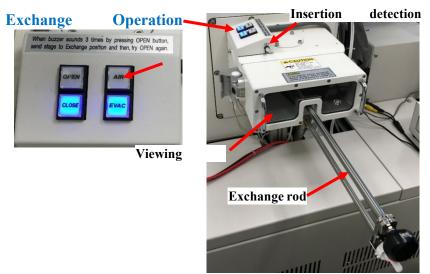
3) Check and make sure the PD-BSE, STEM and EDS detectors are fully retracted on the left side of SEM chamber:



4) Check to make sure the exchange rod is locked. If not, turn the exchange rod locking knob *clockwise* to lock the rod and make sure the red light on the locking knob is ON.



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



6) **Press highlighted corner with your thumb (preferred) or grab the handle** 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.



- 7) Insert the specimen stage onto the exchange rod:
  - a) Turn the **exchange rod** locking knob *counterclockwise* to release the rod and push the rod forward to find the fork.



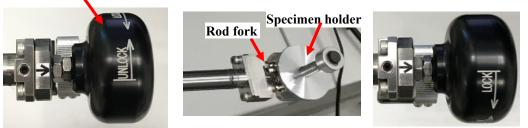
Exchange rod locking knob



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

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

Specimen holder knob (top view)



8) Pull the specimen **exchange rod** all the way 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**.

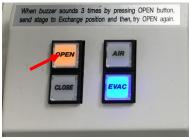




9) Close and hold exchange chamber door with right hand and use left hand as shown below to press the EVAC button on the exchange operation panel. Wait until the buzzer sounds and the EVAC button LED changes to stable blue, indicating the chamber is evacuated back into a good vacuum. Caution: DO NOT hold the exchange rod to close the door as this will lead to rod bending with time.



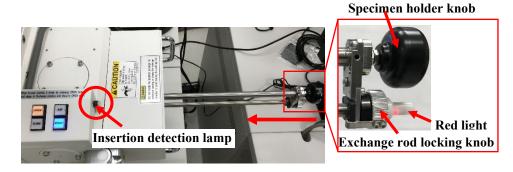
- 10) If it takes more than 15 mins to hear the beep, remove the sample from the exchange chamber. It means your sample can not be accepted by our system. Please contact manager for more info.
- 11) Press **OPEN** button on the exchange operation panel. **Wait until** the buzzer sounds and the gate valve is open.



12) **Turn** the exchange rod locking knob *counterclockwise* to release the lock. **Push** the transfer rod forward slowly into specimen chamber until the **insertion detection lamp** on top of the exchange chamber is lit in **blue**.

#### Warning:

- > **DO NOT** rotate the specimen holder knob while pushing the rod into specimen chamber. This may cause accidental switch of **Lock** position to **Unlock** on the rod leading to sample transfer failure and mechanic damage.
- > **Always** hold the knob when pushing the transfer forward to prevent rod accidental sliding into specimen chamber.



- 13) Turn the specimen holder knob *clockwise* to UNLOCK position. With left hand holding the door, right hand grabs the knob and slowly pushes against glass window with the knuckles and pull 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.
- 14) Press the **CLOSE** button on the exchange operation panel and wait until the buzzer sounds, indicating the sample transfer is complete.



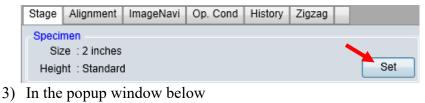
15) Turn off small LCD from the back top left corner. Leaving the LCD on will damage the EDS detector even at retracted position.

#### 7 Image Observation

 Click the HOME button on the menu at the top right of PC-SEM window (Caution: DO NOT repeatedly click this button as this may lead to STOP button next ineffective).



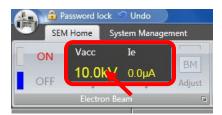
2) Go to Stage tab and click Set button to set the specimen stub Size and Height



-	ple Size / Detectors		x
Size 2 inches	Height ▼ Standard	BSE(PD)	c)
a)	b)	🗖 🔲 FQ-EDX	
		DK Cance	el

- a) Choose the Size of specimen stub one size up for safety purpose, e.g.: choose 2 inches for 1 inch specimen stub).
- b) Set **Height** to **Standard. Warning**: make sure the specimen stub has been carefully aligned using height gauge.
- c) Confirm that the boxes next to detectors (EDX, BSE, BF-STEM and FQ-EDX) are not checked.
- d) Click OK button to close the Set Sample Size/Detectors window above.

- 4) Setting imaging parameters.
  - a) Set accelerating voltage V<sub>acc</sub>:



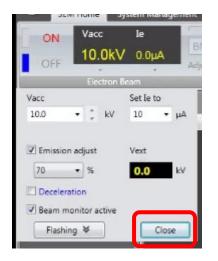
> Click inside Electron Beam window in highlighted rectangle region above and choose Vacc from dropdown list. (typical values: 1kV, 5 kV, 10kV or 15kV)

#### Warning:

- > **Do Not** type in arbitrary numbers, as the beams from dropdown list have been calibrated by service engineer.
- > Always try small voltage first to avoid surface damage, over-charging and ebeam induced carbon deposition (black imaging box)
- b) Set emission current Ie:
  - > Click inside **Electron Beam** window above to choose  $I_e$  at 10  $\mu$ A.

Note: Consider smaller values if surface charging or carbon deposition is a concern.

c) Click Close button to close the dropdown Electron Beam setting window.



d) Confirm the LM (Lower Magnification mode) is active inside Magnification

window below. If not, click H/L to switch back to LM mode.



- R1
- e) Choose Rapid Scan Mode Rap1/2 in the window below to start with.



f) Choose the **SE(LM)** (Secondary Electron Low Magnification) detector in Optics tab below. If not, click and choose from dropdown list.

Optics		
Signal select	Normal	0
SE(LM)	×	
(EF=0)		

g) Set the Probe current to High in the Operation condition window below.
 Note: high probe current is recommended at lower magnification to improve signal/noise ratio.

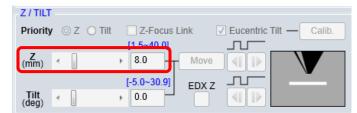
Probe current	🔘 Norm	High
Cond. Lens1	5.0 🔻	\$
[2 W.D.	.8-40.0mm](Ful	l) mm
	ABCC lin	k

- h) Confirm that Cond Lens 1 is set at "5" in the Operation condition window above.
- i) Set sample surface Z height (defined as the distance from the bottom of electron column to sample surface). To avoid sample collision with detectors, always check the table below before changing Z height value:

```
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
```

#### Warning:

- > The default height is 8 mm, good in most imaging cases
- > The smallest Z height allowed is 5 mm.



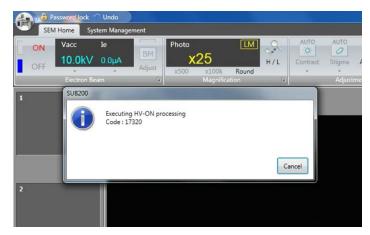
 Check and confirm the Specimen Chamber (SC) pressure has reached LE-4 Pascal on EVAC CONTROL panel as highlighted below. Never turn on the electron beam if the pressure is not ready. The electron gun chamber will be contaminated quickly.



 Click the ON button to turn on beam voltage V<sub>acc</sub> in the Electron Beam window below: Note: the process takes seconds, please be patient and wait till the popup window disappears.



7) Waiting for the "Executing HV-ON processing" window to disappear.



8) Go back to the CameraNavi tab window, the **Navigation not possible** message should disappear on the bottom left corner of the window below. Click on intended samples on the picture to navigate the stage.



- 9) Click Contrast on the top menu bar or AUTO button on the Manual Operation Panel below if the image is too bright or dark. The BIRGHTNESS and CONTRAST knobs can be used separately for manual adjustment.
- 10) Roll the **track ball** on the **STAGE CONTROLLER** to find the field of interested in LM mode:

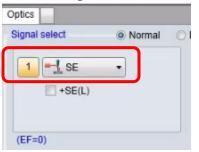


## **Manual Operation Panel**

- 11) Adjust magnification using MAGNIFICATION knob on the Manual Operation Panel.
- 12) Adjust focus using FOCUS COARSE and FINE knobs. Move the stage to look for the

field of interest in LM mode, and then click H/L to switch to High Magnification (HM) mode.

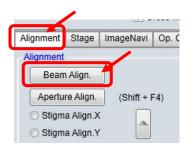
13) Check the **Optics** tab and make sure the SE mode is checked as below:



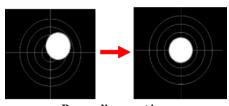
14) In **HM** mode, check and make sure the **W.D.** (Working Distance) is set to 8 mm.

Operating condition Probe current	(Low-Mag mo	ode	le] © High
Cond. Lens1	5.0 -	•	
[2	8-40.0mm](Fi	III)	0
W.D.	8.0 -	m	mm
	ABCC li	nk	ĸ

- 15) Change the magnification and get sharply focused image using FOCUS, STIGMA X and STIGMA Y knobs on Manual Operation Panel.
- 16) If image **drifts** (swaying or heaving) during adjusting:
  - Click Alignment tab and click Beam Align button. The ALIGNMENT LED on the Manual Operation Panel should be flashing.



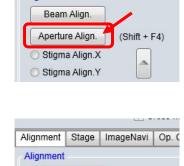
 Bring the circular image to the center of the image area by adjusting X and Y knobs on the panel.



Beam alignment image

- Click Aperture Align button below and adjust STIGMA/ALIGNMENT X /Y knobs on the panel to stabilize the image wobbling.
- > Click Stigma Align X button and adjust STIGMA/ALIGNMENT X/Y knobs to minimize the image wobbling if needed. You need to lower the magnification below 50k to observe the changes.
- > Click Stigma Align Y button and adjust STIGMA/ALIGNMENT X/Y knobs to minimize the image wobbling if needed. You need to lower the magnification below 50k to observe the changes.

> Click off on Alignment tab. Make sure the Alignment LED on Manual Operation Panel is OFF



Beam Align.

Aperture Align.

Stigma Align.X

Stigma Align.Y

Stage ImageNavi Op. C

nift + F4)

Alignment

Alignment

Alignment	Stage	ImageNavi	Op. C
Alignment			
Beam	n Align.		
Apertu	re Align.	(Shift + I	F4)
O Stigm	a Align.X	<ul> <li></li></ul>	
🔘 Stigm	a Align.Y	· 📋	
O Low N	/lag. pos	ition	
	Off	(F4)	

**S**3

**S1** 

- 17) Switch back to high magnification. Readjust the FOCUS, STIGMA X and STIGMA Y knobs on Manual Operation Panel to get the final image before saving.
- 18) Save image:
  - a) Select the field of view, confirm image with slow scan Slow1/2 or Slow3/4.

M

- b) Click the **Capture** button Slow\_1280.
- c) Click  $\mathbb{R}_{un}$  button next to  $\mathbb{R}_{un}^{\mathbb{R}}$  to resume live image scan.
- 19) Image capture settings for charging samples (image distorts, streaky, drifts during scan):
  - a) Choose CSS (Charge Suppressed Scan) mode:

> Clicking on the small box in the **Scan Menu** to open the Scan Button Setting window.



> Check the radio button next to CSS. Hit **Apply** button and **Close** in the window below:

🚑 Scan Bu	itton Setti	ng				×
Button sel	lect Inte	gration				
Scan speed button setting						
Reset	t to defau	lts •				-
			Rapid/Fast	Fast/S	Slow/CSS	Red
12	Rapid	1/2	۲			
83	E Fast	1/2	$\bigcirc$	$\bigcirc$		
	Slow	1		0 0	$\odot$ $\odot$	
	CSS	2		0	$\odot$ $\odot$	
		3		$\odot$	0	$\odot$ $\odot$
		4			0	$\odot$ $\odot$
		5			$\odot$	$\odot$ $\odot$
		6			$\odot$	$\odot$ $\odot$
		7			$\odot$	$\odot$ $\odot$
	Red	1/2				۲
		3/2				$\odot$
		1/3				$\odot$
				Apply		Close

> The CSS Scan menu should appear as follows:



- b) If the charging is still strong on sample surface, change the scan mode from line scan to frame mode:
  - > Click the small box in the **Capture** menu below:



> In the Capture/Save Setting window below, choose Fast capture mode with a minimum 8 frames or more as needed and close the window.

👘 Capture	e / Save S	etting	×
Capture	Output	Data Display	
Capture	setting —		
Scan	speed lir	nk	
		Image size	Speed / Integration
L Slow		1280x960 🔻	16 🔻 s
Lcss		1280x960 🔹	16 🔹 s
🔘 Rapi	d	1280x960 -	16 • frames
Fast		1280x960 -	8 • frames
© Slow	1 : Integ.	1280x960 🔻	8 <b>v</b> frames
🔲 Drift	correctio	n:	

c) If captured images using a) and b) still appear streaky, blurry due to charging, click R1 button below once to switch to R2 mode, wait several seconds till image contrast is acceptable and then click Save button to save the image.

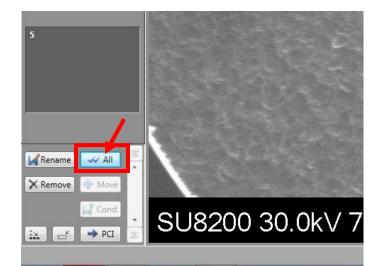


#### 20) Data save:

a) Click the **OFF** button to **turn off** electron beam in the **Electron Beam** window below before data saving:



b) Click ALL button on the bottom of the image thumbnail column below:



c) Click →PCI button in above window, the collected images will be transferred into Quartz PCI program below. Click File in the menu and select Export All... on the dropdown menu, then Browse to choose Export to Path and File Format, Export Text (imaging parameters) and hit OK.

🕖 Quartz PCI - Ir	nage Managemen	t System					
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			다인님	1 -17	<u> (φ</u> μ «	% & ¢	5 & 4
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Export All	1.00	10 m 10	100	1	- /	l	X
Destination	to Path F:\SEM u	sers			Browse	ОК	
	to CD/DVD			C		Canc	el
File Format:	TIFF Images (*.TI	F) 🔻	Options			Help	
Apply to:	🔽 Images	Exp	port Text				
	Spectra	🔽 Ap	ply Overlay				
	Reports	Sec	cure PDF				
Contraction of the	and the second	and the second	Contraction of the local division of the loc			- China -	

d) **Clear the thumbnail images** from the left column by clicking **Remove** button.

# 8 Closing SEM measurement

 Make sure the electron beam has been turned off with a solid blue bar appearing next to OFF in the Electron Beam window below:



2) Click inside Electron Beam window highlighted above and set electron beam voltage  $(V_{acc})$  to 10 kV, and emission current Ie to 10  $\mu$ A



3) If **Deceleration Mode** was used, uncheck the Deceleration box as shown below:

JEM HU		узленн манау	jemen
ON	/acc	le	
1	0.0kV	Au0.0	
OFF	-	•	A
	Electron B	leam	
Vacc		Set Ie to	
10.0 +	1 kV	10 •	μА
Emission adj	ust %	Vext	kV
70 •	20	0.0	KV.
Deceleration	22		
Beam monito	or active		
Flashing &	5	Clos	e
		Clos	e

4) Switch imaging mode to Lower Magnification (LM) Mode



- 5) Click **Freeze** button to stop scanning. The **Run** button will appear after clicking.
- 6) Click the **EXC** button on **PC-SEM** top menu to see the specimen stage moving to the exchange position. Wait till status vertical bar next to EXC button stops flashing.



7) Resume the default Scan modes as follows:



8) Click the small box in the **Scan Menu** to open the Scan Button Setting window and change the setting back to Slow:

👘 Scan Butto	n Settin	g				×
Button select	Integ	ration				
Scan speed	Scan speed button setting					
Reset to defaults					-	
			Rapid/Fast	Fast/S	Slow/CSS	Red
535	Rapid	1/2	۲			
833	Fast	1/2	$\bigcirc$	$\bigcirc$		
$\bigcirc$	Slow	1		0 0	$\odot$ $\odot$	
	CSS	2		0	$\odot$ $\odot$	
		3		$\odot$ $\odot$	0	$\odot$ $\odot$
		4			0	$\odot$ $\odot$
		5			$\odot$	$\odot$ $\odot$
		6			$\odot$	$\odot$ $\odot$
		7			$\odot$	$\odot$
=	Red	1/2				۲
		3/2				$\odot$
		1/3				$\bigcirc$
			(	Apply		Close

- 9) **Check and resume** the default line scan mode in the **Capture**:
  - > Clicking the small square next to the **Capture** as highlighted below:



> In the **Capture/Save Setting** window below, choose **Slow/CSS** as highlighted in the window:

tan Capture / Save Setting									
Capture Output									
Capture setting									
Scan speed link									
Image size Speed / Integration									
Slow	1280x960 -	16 🔻 s							
L <sub>CSS</sub>	1280x960 -	16 • s							
Rapid	1280x960 -	16 • frames							
○ Fast	1280x960 -	8 • frames							
Slow1 : Integ.	1280x960 -	8 • frames							
Drift correction:									

10) Uncheck the ON box in the Image Shift/Rotation window as follows:



11) Check and confirm in Stage > Set: Set Sample Size/Detectors window below that all detector check boxes are unchecked.

👍 Set Sample Size / Detectors								
Size 2 inches	Height	EDX BSE(PD) BF-STEM FQ-EDX						
		OK Cancel						

12) Click the CameraNavi tab below and click the Delete button to remove the sample photo.



13) Turn on small chamber LCD monitor from the top left corner on the back.

14) Put on gloves and remove samples from the specimen chamber:

- > Press **Open** button on the exchange chamber and wait for the beep,
- > Unlock the transfer rod and push the rod all the way into the specimen chamber until the blue SET light on,
- > Turn to **Lock** position on the black knob,
- > Extract the rod all the way to the back and **lock** the rod,
- > Press the **Close** button and wait for the beep,
- > **Turn off** small chamber LCD
- > Press the Air button and wait for the beep,
- > Using the right hand to open the exchange chamber,
- > Turn to **Unlock** position on the black knob and extract the sample holder out from the rod,
- > Close the exchange chamber door and hold with right hand, then press the **Evac** button,
- > Release the right hand until door closes by itself.

15) Keep the PC-SEM window fully maximized.

- 16) Upload data to box.yale.edu, or use ONLY the Core USB flash drive for data transfer.
- 17) Log off FOM from SEM computer or user's FOM SEM calendar. Make sure the FOM Screen Lock window below appears.

**Note:** if reporting issues, please check "**Something wrong**" in FOM logoff window and describe the issues in the empty box.

Select Login Option

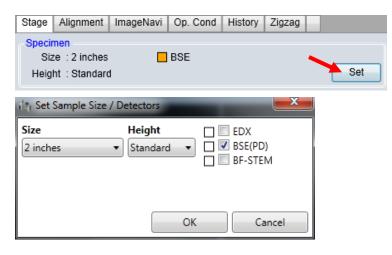
Login with Yale NetID

Login with FOM username and password

- 18) Sign off logbook.
- 19) **Remove** ONLY samples from the stub on the specimen holder and **clean** the holder with Kimwipes using **IPA**. **Do Not** dissemble the sample holder.
- 20) Store the specimen holder in assigned organizer box.
- 21) **Clear** the SEM work bench.

22) Confirm the **Chamber LCD** has been turned off before leaving.

- 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) Check to make sure sample Z height is set at 8mm.



- 4) Select Vacc at 15 kV and regular Ie=10  $\mu$ A as the PD-BSE requires high e-beam kV
- 5) Select **Dual Screen** mode on the **PC\_SEM** top menu bar as shown below, and choose



- 6) Switch to LM mode and turn  $V_{acc}$  ON
- 7) Slowly rotate the knob shown below to move the PD-BSE detector to the measurement position. Stop turning quickly once feel stopped.



- 8) Check to make sure the small chamber LCD is OFF. The PD-BSE is very sensitive to infrared light generated from camera inside.
- 9) Click beside set 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 PD-BSE to activate **BSE** imaging:
  - a) Click Contrast 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

**S1** 

**S**3

b) Select the field of view, confirm image with slow scan Slow1/2 or Slow3/4 and then,

click the Capture button Slow\_1280.

Warning: do not use Rap1/2 rapid scan mode for PD-BSE imaging.

M

R1

- 11) To quit **PD-BSE detection** mode:
  - a) Turn off electron beam voltage, Vacc



- b) Switch to LM mode.
- c) Turn on the small chamber LCD
- d) Rotate slowly to fully retract the PD-BSE detector
- e) Click the **EXC** button to move the specimen stage to the exchange position. **Wait until the green status bar stops flashing** next to **EXC** button.



f) Click Set button in the Stage tab shown below; uncheck BSE(PD) box, and then follow Section 7 Closing SEM measurement and 8 Checklist after Experiment to close the measurement.

Stage	Alignment	ImageNavi	Op. Cond	History	Zigzag	
	men ze : 2 inches ht : Standaro	1	BSE		4	Set
itaj Set	Sample Size	/ Detectors			X	
Size 2 inch	es	Height Standard		EDX BSE(PD BF-STE FQ-ED	M	
			OK	C	ancel	

Notice: The STEM sample holder has a standard height of 36 mm, so no need to use Height Gauge.

- 1) Make sure Vacc is OFF.
- 2) Make sure the small chamber LCD is OFF.
- 3) 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 as shown below:

Stage	Alignment	ImageNavi	Op. Cond	History	Zigzag	
	nen ze : 2 inches ht : Standard	1	BF-STEM		•	Set
f <sub>ff</sub> Set	Sample Size	/ Detectors			×	
Size 2 inch	es	Height Standar	d <b>v</b>	EDX BSE(PI BF-ST FQ-ED	EM	
			ОК		Cancel	

4) Make sure the **Z height** is set at **8 mm**. Never change the Z height larger than 20 mm to damages the STEM detector below the stage.



- 5) Select  $V_{acc} \le 20 \text{ kV}$  and regular Ie=10  $\mu$ A in STEM
- 6) Select **Dual Screen** mode on the **PC\_SEM** top menu bar, and choose detector for the first screen and **BF-STEM** detector the second



- 7) Check and make sure the SC chamber pressure reaches LE-4 Pa.
- 8) Switch to LM mode and turn on electron beam voltage  $V_{acc}$ .



10) Click beside se imaging:

- a) Find interested areas on the sample in LM mode; adjust focus and switch to HM mode.
- b) Following instructions in Section 6 Image Observation to get a well focused image in HM mode

**Caution**: **Always** switch to **SE** window to adjust image quality for **STEM** imaging

11) Click beside **BF-STEM** to activate **STEM** imaging:

- a) Click Contrast 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 adjustments
- b) Select the field of view, confirm image with slow scan Slow1/2 or Slow3/4 and then,

click the **Capture** button Slow\_1280.

**R1** 

Notice: do not use Rap1/2 rapid scan mode for STEM imaging

M

12) To quit STEM detection mode:

AUTO

a) Turn OFF electron beam voltage  $V_{acc}$ , and switch  $V_{acc}$  back to 10 kV, and 10 uA.



b) Slowly retract the STEM detector until stopped

**S**3

**S1** 

c) Click the **EXC** button to move the specimen stage to the exchange position. Wait until the green status bar stops flashing next to EXC button.



d) Click Set button in the Stage tab; uncheck BF-STEM box, and then follow Section
 7 Closing SEM measurement and 8 Checklist after Experiment to close the measurement.

Stage	Alignment	ImageNavi	Op. Cond	History	Zigzag	
	men ze : 2 inches ht : Standaro		BF-STEM		•	Set
iter Set	Sample Size	/ Detectors Height		EDX	×	
2 inche	es	Standard		BSE(PI BSE(PI BF-STE FQ-ED	EM	
			ОК		ancel	