Standard Operating Procedure II:
EDS (Bruker Flat-Quad)

Yale WEST CAMPUS
Material Characterization Core
ywcmatsci.yale.edu

ESC II, Room A119F
810 West Campus Drive

Version 1.41, October 2022

- **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.

Table of Contents

Energy Dispersive Spectroscopy (EDS) 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 A119
   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

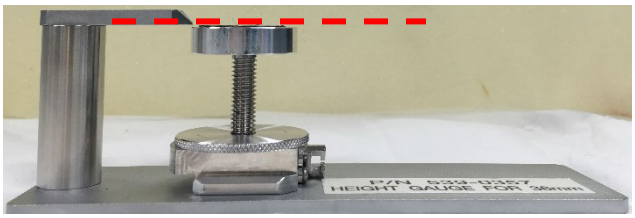
The Yale West Campus MCC Facilities are operated for the benefit of all researchers. If you encounter any problems with this facility, please contact the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact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

Note: TEM samples can also be used with a dedicated **TEM sample holder** for **EDS**. The **EDS mapping** resolution can reach below **100 nm** with TEM samples using **STEM-EDS** mode.

1) **Always wear gloves** for vacuum sample preparation! **Change gloves** if touched computer keyboard and mouse.
2) Follow instructions from SEM SOP to prepare samples. Use **copper tapes to ground** the **sample surface** with the holder to reduce the surface charging.
3) **Align sample surface strictly with the height gauge** tip as shown below.
   Warning: Samples mounted above **Height Gauge** tip will **hit** the EDS detector and the **repair fee ($40,000)** will be charged to PI's account**.**
   Note: no need to use height gauge if **TEM sample holder** is used.



4) **Clean sample holder**: bring the specimen stub inside the fume hood and **blow off** loose particles on the sample surface using the **N_2 gun**.

3 Starting SEM Instrument

Follow **Section 3 Starting Instrument** in the separate SEM SOP.

4 SEM System Status Check

Follow **Section 4 System Status Check** in the separate SEM SOP.

5 EDS Sample loading

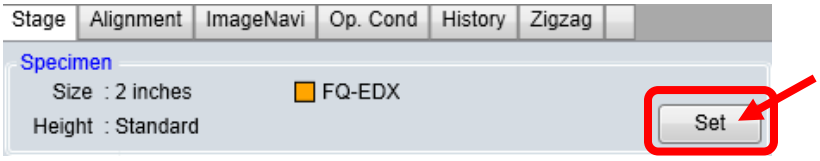
1) Follow **Section 5 Loading the Specimen** in the separate **SEM SOP** and leave the sample at exchange position
2) **Turn off** the **small LCD** from the back (top left) to avoid EDS detector damage and EDS spectra distortion.
   Warning: The LCD unit also provides power to the infrared camera inside main chamber. If it is left on during SEM/EDS scan, the camera will continuously emit infrared light to **flood the EDS detector even at fully extracted position** and shorten its lifetime quickly.

6 EDS setup on SEM computer

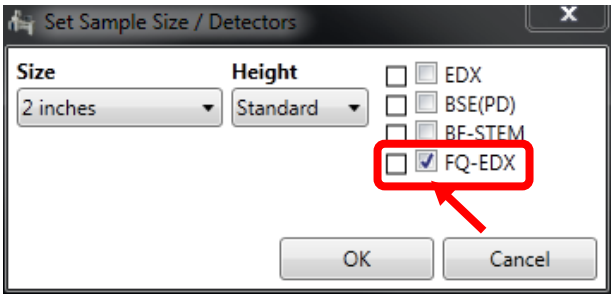
1) In **PC_SEM** program on the SEM computer (left side), click **HOME** button to move the sample holder to **HOME** position
2) **Turn off** the small **LCD** by pressing the switch in the back top-left corner of the monitor.
   Note: This step is crucial, otherwise **the EDS detector will be flooded by ambient**

signals leading to fat peaks in spectra.

3) Click **Set** button in the **Stage** tab in the popup window below:

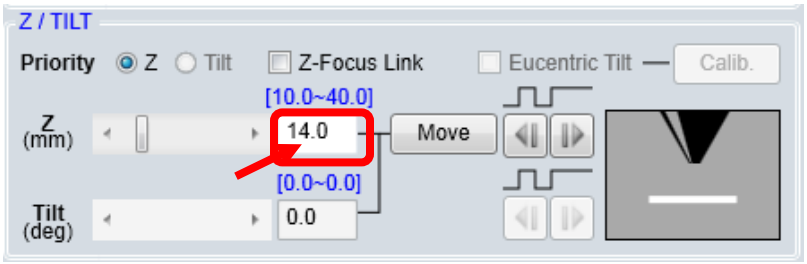


4) Check the **FQ-EDX** box in the popup window below and hit OK.



5) The stage **Z position** should move to the default height at **14 mm**.as highlighted in the window below:

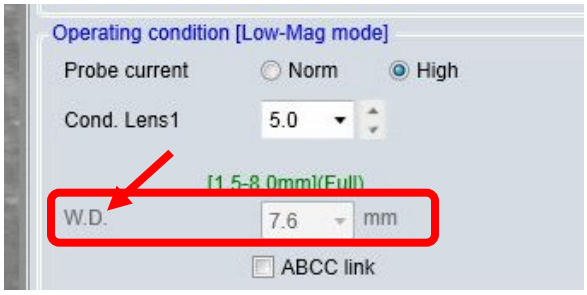
Warning: Never change the **Z position < 11 mm**. This will cause the sample holder crashing into the **EDS detector**. This **severe SOP violation** will lead to user account suspension and charge on PI's account.



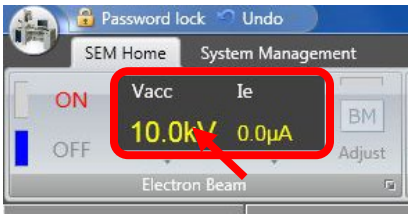
6) Check the working distance (**W.D.**) in **PC_SEM** below and make sure it is set at **15 mm**.

If not, click 

on the top menu bar to switch imaging mode from **LM** (low magnification) to high magnification, then change **W.D.** in the window below. Switch back to **LM**.



7) **Change Vacc (accelerating voltage in PC-SEM window)** as highlighted below



with the instructions listed below:

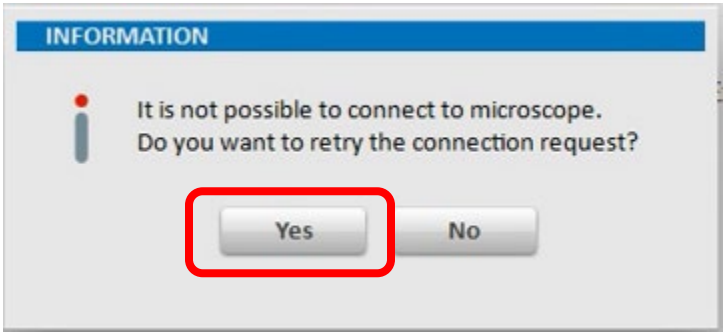
a) Check the EDS element table and choose a set of characteristic X-ray emission lines for intended sample elements that have least energy overlapping.

b) Choose the **Vacc** **at least 2x** the highest X-ray line in matrix elements. **NEVER** choose e-beam voltage above **20 keV!!** **The EDS detector will be burned!!**
Tip: smaller beam voltage means higher EDS special/mapping resolution.

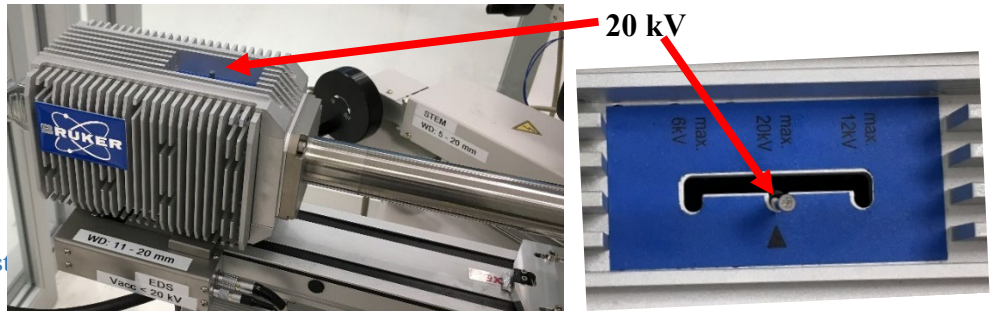
7 EDS setup on EDS computer

1) Log into **EDS** computer through **FOM Screen Locker**.
   a) The EDS software **Esprit** window should be always kept **ON**.
   b) If the **Esprit** program was closed and the **EDS** computer was logged off, select the profile **PC-SEM** with password **hitachi**

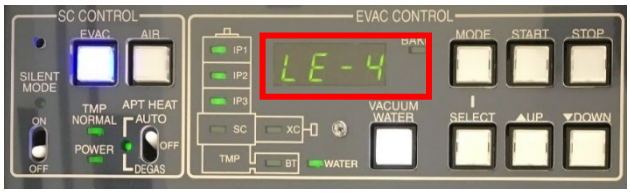
2) Clear **Yes** button on the popup **INFORMATION** window below to connect to microscope (SEM)



3) Check **EDS** detector window position on the detector highlighted on the pictures below and **make sure it was set by previous user at 20 kV.** If not, report to manager immediately.
Warning: It is the serious SOP violation if forgot to switch the detector window back to 20 kV after use. This may lead to detector damage and repair charge will be applied to user's PI account.

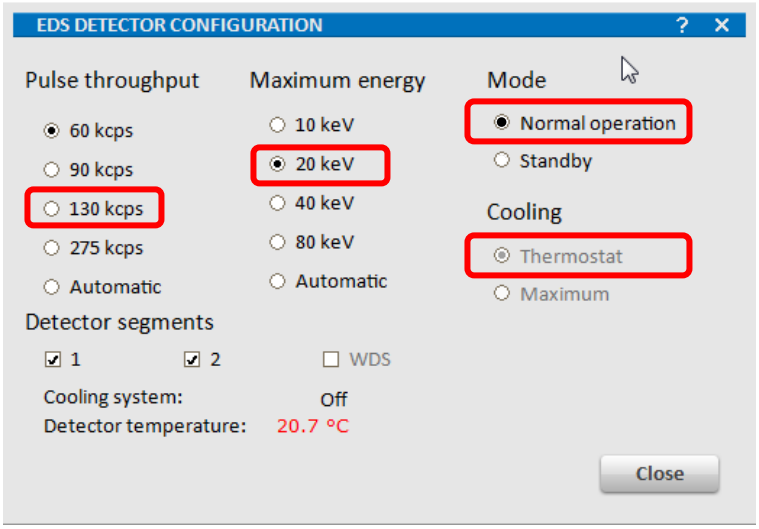


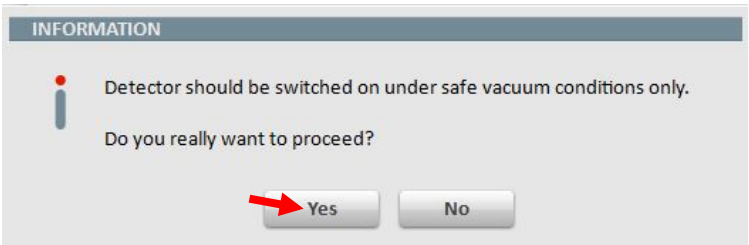
4) **Check to make sure** the Specimen Chamber **SC** pressure is at **LE-4 Pascal**.



5) Switch **EDS** detector in operation mode and set up **detector configuration parameters**:
   a) In **EDS Esprit** operating program, click the triangle in the **EDS** tab at the bottom left corner as indicated below:

   b) In the **EDS DETECTOR CONFIGURATION** popup window below:
      > Select **130 kcps** in **Pulse throughput** and **20 keV** in **Maximum energy.**
      > Make sure the **Thermostat** is checked in **Cooling** setting.
      > Select **Normal operation** in Mode setting. Select **Yes** in the popup **INFORMATION** window.





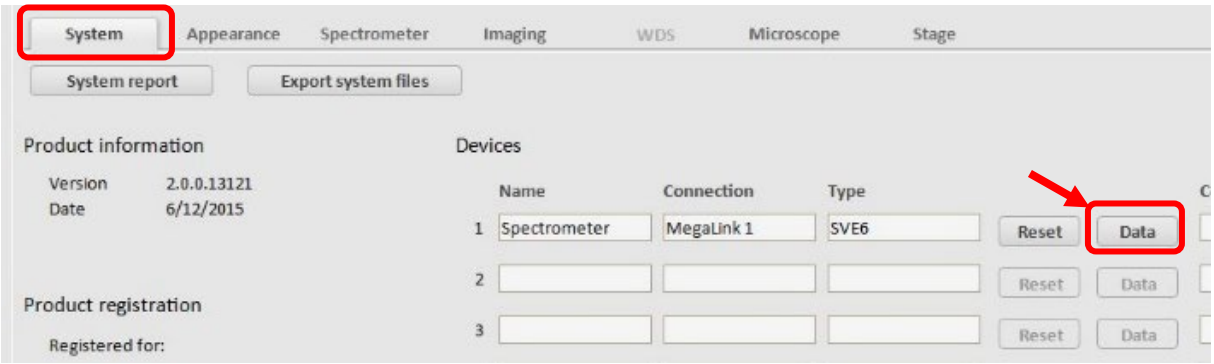
c) Close both **INFORMATION** and **EDS DETECTOR CONFIGURATION** window above.

d) The EDS detector will be cooled to the operating temperature of **-20 ± 0.5 ºC** in about 5 minutes.

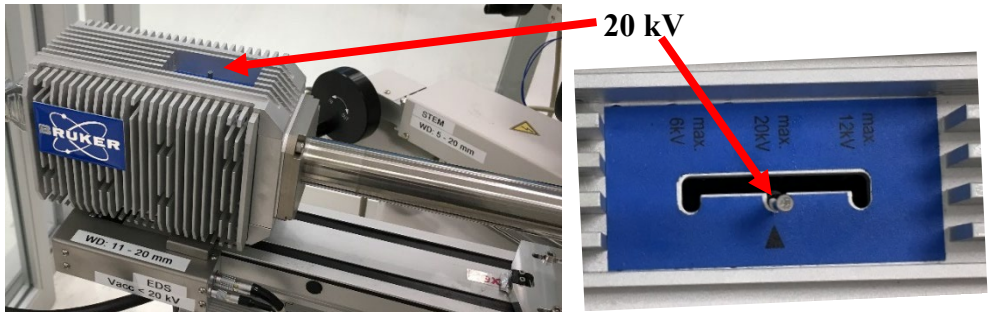
6) **EDS** detector window setting at different overvoltage/e-beam voltage:

a) If the **beam voltage ≤ 6 kV**:

> Enter **Esprit System** window by clicking System button on the **bottom left** of the side menu and clicking System tab as shown below. Click Data button to open **Detector Data** window and select "**1µm Mylar**" from dropdown list.



> Go back to **EDS detector head** on **SEM** chamber as shown below, switch the detector window from 20 kV to **6 kV**.



b) If the **beam voltage is between 6 kV and 12 kV**:

> Enter Esprit **System** window by clicking System button on the left side menu

and clicking System as shown below. Click Data to open **Detector Data** window and select "**1+2 µm Mylar**" from dropdown list.

> Go back to **EDS detector head** on **SEM** chamber, switch the detector window from 20 kV to **12 kV**.

c) If the **beam voltage is larger than 12 kV** **(Never change voltage to above 20 kV to damage the detector)**:

> Enter Esprit **System** window by clicking System button on the left side menu and clicking System as shown below. Click Data to open **Detector Data** window and select "**1+6 µm Mylar**" from dropdown list.

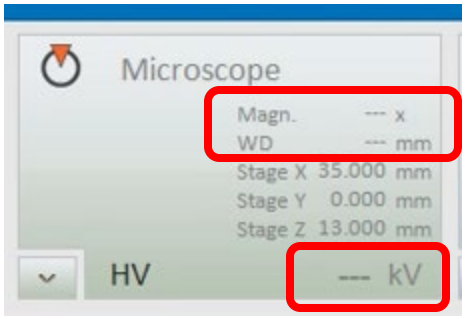
> Go back to **EDS detector head** on **SEM** chamber, make sure the detector window switch is set at **20 kV**.

d) If higher Vacc is needed in the middle of EDS scan, switch the EDS detector window to higher voltages first to protect the EDS detector.

e) If lower Vacc is needed in the middle of EDS scan, change the Vacc to lower voltages first to protection the EDS detector.

8 EDS measurement

1) **During EDS detector cooling**, turn on e-beam in **PC_SEM** program and find the area of interest.
2) Check EDS detector and **make sure** it has reached the operation temperature **-20 ± 0.5 °C**
3) Check the **Microscope** tab below and make sure the **HV** (beam voltage Vacc in PC_SEM computer), **Magn.** (Magnification) and **WD** (W.D.) follows the numbers in **PC_SEM** program.



4) **If HV, Magn and ED failed to match SEM program:**

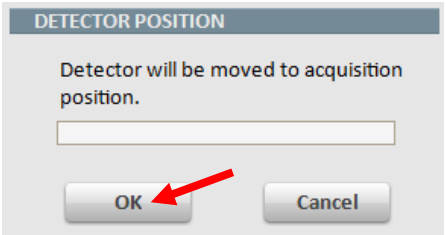
> clicking System button on the **bottom left** of the side menu and clicking Microscope tab, then click Initialize button. If no error popup window appears, wait for several minutes, and monitor HV, Magn and WD changes on the Microscope tab. Restart Esprit program if needed.

> If the following **ERROR** popup window appears,

Click **OK** button and follow the steps below:

a) Turn off beam voltage (Vacc) and close PC_SEM program and Esprit program,
b) Restart PC_SEM computer first,
c) Wait till the windows system is back on SEM computer, restart EDS computer,
d) Start PC_SEM program and wait until the program is completely back,
e) Start EDS program.

5) Click  button in **EDS** tab in **Esprit** program and click **OK** on **DETECTOR POSITION** window to move the detector to the acquisition position right above sample.

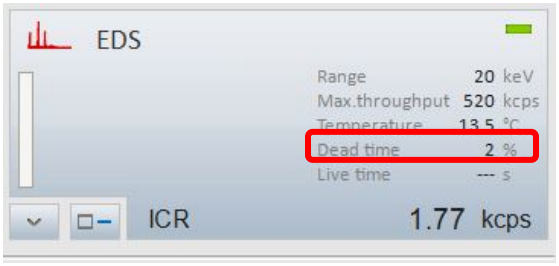


6) If the user decide to run EDS in the middle of SEM imaging, make sure to check **FQ-EDS** mode in **PC_SEM** program **Stage > Set** setting window to lower the sample stage to required EDS stage height (14 mm). Please check **Section 6 from Step 1-4)** on page 3.

Warning: **Failure to check FQ-EDS box** will lead to EDS detector crashing into sample stage. The repair cost will be charged on PI's account.

7) **EDS dead time** adjustment (Read steps below and move to Step 7) Spectrum Acquisition mode and follow steps a)-c)):

> As shown in the **EDS** tab below, the **Dead time** in percentage denotes the signal processing capability of the EDS controller. The higher number indicates the higher signal counts and the possibility of the **signal pileup or false peak** appearance. The **Dead time** should be kept around **30%** for Spectrum Acquisition Mode and **10%** for Mapping, Objects and Line Scan Mode.



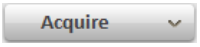
8) **Spectrum Acquisition Mode**:

Note: Make sure the imaging area for spectrum collection is **homogeneous** if using this mode. Consider **Objects mode** next for inhomogeneous surfaces.

a) Go back to **PC_SEM** window, decide the image magnification and briefly adjust focus and stigma to get a **good** SEM image. No need to adjust aperture alignment and stigma alignment.

b) In **EDS Esprit** window click 

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

c) Click 

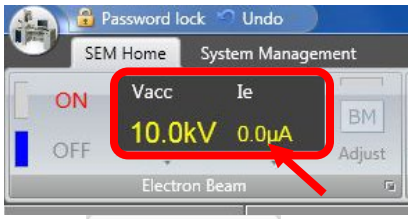
button and check and adjust the **Dead time** within ~**10 – 30%**:

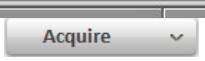
> If the **Dead time** is below **10%**, in **PC_SEM** window (do the opposite steps if **Dead time** is above **30%**):

> Hit 

again to stop the acquisition. Increase the e-beam **emission current** (maximum **30 µA**). Click 

to resume acquisition and check the dead time.



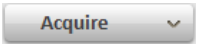
> Hit 

again to stop the acquisition. Choose **High Probe current**. Click 

to resume acquisition and check the dead time.

> **If the dead time is still below the required values,** click 

again to stop the acquisition. Change **Cond. Lens 1** setting from "**5**" to "**1**" or

"2" in the window above, the smaller number the higher the signal intensity. Click 

to resume acquisition and check the dead time.

d) If the interested peaks appear smooth, click 

again to stop acquisition.

e) To add collected spectrum into **project** or **report**, hit the 

button on the top right corner of the spectral workspace

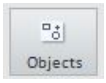
f) To save the data in Bruker spectra format (***.spx**) or export to ***.txt** or ***.xlsx** format, click the lower 

button.

9) Objects Mode:

Note: this mode should be chosen for **inhomogeneous** surface spectral analysis.

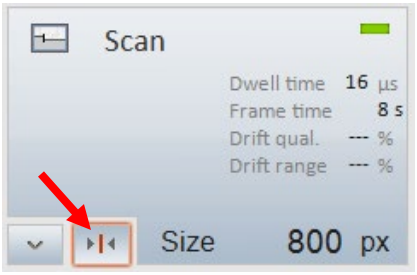
a) Adjust **Dead time** within **10 – 30%** in **Spectrum Acquisition Mode** above

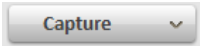
b) Hit 

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

c) In **PC_SEM** window adjust focus and stigma to get **sharp** SEM image

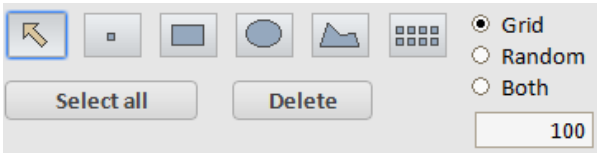
d) Click  button on **Scan** tab to activate image drift correction. Make sure the button is highlighted in **red** to enable **drift correction**.



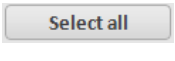
e) Go back to **EDS Esprit** window and click 

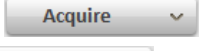
button to capture SEM image.

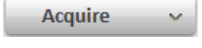
f) Select the desired object type on the bottom menu bar below and click on captured image above to specify positions. As the EDS spatial resolution is e-beam voltage/overvoltage dependent and typically around 1 µm, draw lines well inside the interested objects.



> Choose **Point** object mode  only if the interested feature is too small.

g) Click 

to highlight all objects and click 

h) If the interested peaks look smooth, click 

again to stop acquisition. Allow longer collection time if the point or small square or circle are

used.

i) Click the 

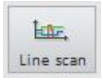
button on the top right corner of the workspace window to save object data.

j) To save the spectrum only, click 

at the top right corner of the spectrum window.

10) Line Scan Mode:

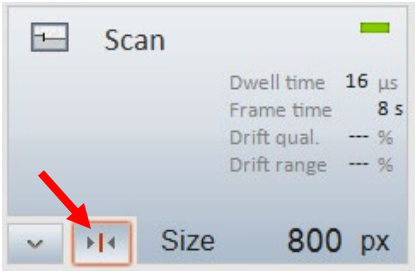
a) Adjust Dead time within ~ **10 – 30%** in **Spectrum Acquisition Mode**

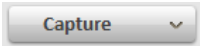
b) Hit 

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

c) In **PC_SEM** window adjust focus and stigma to get **sharp** SEM image

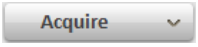
d) Click  button on **Scan** tab to activate image drift correction. Make sure the button is highlighted in **red** to enable **drift correction**.

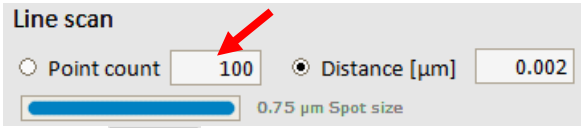


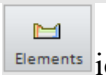
e) Go back to **EDS Esprit** window and click 

button to capture SEM image.

f) Highlight the line and drag and adjust the endpoints to the desired position

g) Set **Point count** of the line scan and click 



h) Use the 

icon to identify elements

i) If the interested peaks look smooth, click 

again to stop acquisition.

j) To save line scan data, click the 

button on the top right corner of the workspace window.

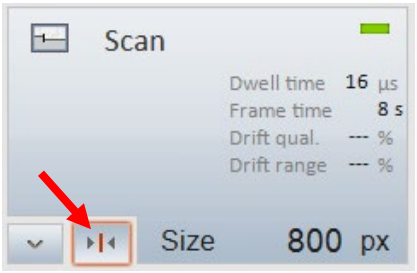
k) To save the profile, click the 

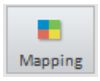
at the top right corner of the lower profile window.

11) Mapping Mode:

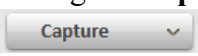
a) Adjust Dead time to be just above **10 %** in **Spectrum Acquisition Mode**

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

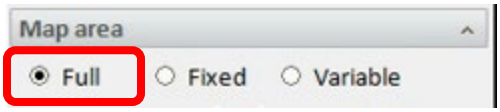


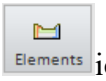
c) Hit  on the left side menu to enter **Mapping** workspace

d) In **PC_SEM** window adjust focus and stigma to get **sharp** SEM image

e) Go back to **EDS Esprit** window and hit  button to capture SEM image.

f) Click  button to start **Mapping**. If a green box appears inside the SEM window, click the ∨ on the bottom right corner of the  and check **Full** below:

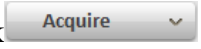


g) Use the  icon to identify elements in spectrum window during mapping.

h) **Before stop mapping**:

> Click **Spectrum** tab above the top right corner of **Map** window, check if the interested elemental peaks are smooth in the spectrum.

> Check if the mapping resolution has not improved with time. Typical mapping collection time depends on signal intensity (**~ 10 – 30 mins** or even longer).

i) Click  **again** to stop **Mapping**.

j) To save map data, click the  button on the top right corner of the workspace window.

k) To save the large combined map image, click the  on the top right corner of the image window.

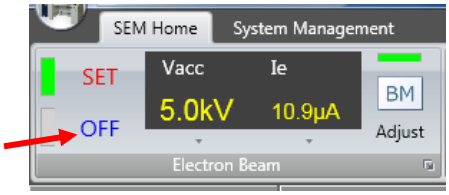
l) To save individual element image in the thumbnail on the bottom, Shift+ left click

all thumbnails and click the 

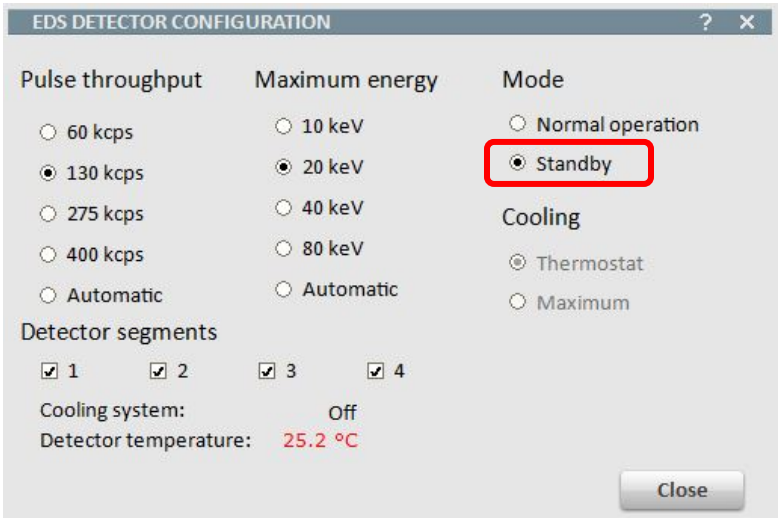
on the right side of thumbnail window.

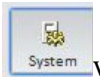
9 Closing EDS detection:

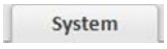
1) In **PC_SEM** program, click the **OFF** button to **turn off** electron beam

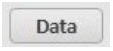


2) Click **Standby** button in **EDS DETECTOR CONFIGURATION** window to switch the **EDS** detector to **Standby Mode**



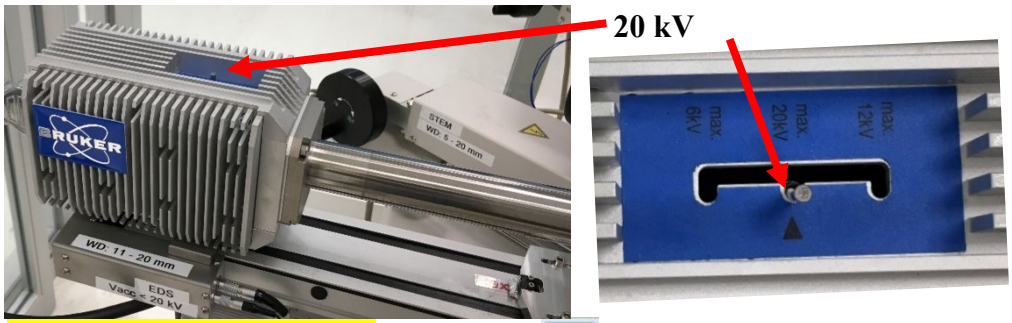
3) In **EDS Esprit** 

window, go to 

tab, click 

button to open **Detector Data** window and select "**1+6 µm Mylar**" from dropdown list and click **OK**.

4) **Check EDS detector window position** and **make sure it is switched back to 20 kV** to avoid detector damage.



5) **Fully retract EDS detector** by clicking  in **EDS Esprit** program and **wait until the detector is fully retracted**.

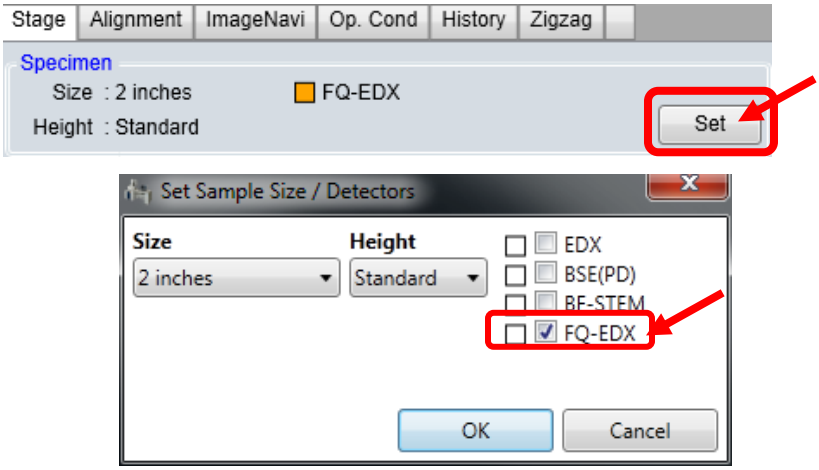
6) In **PC_SEM** window:
   > Change **Vacc** back to **10 kV** and the **Ie** back to **10 µA**
   > Change the **Probe current** back to **Norm**.

   > Change the **Cond. Lens1** back to "**5.0**" in the window below in both **High Magnification** mode and **LM** mode:

   > In **LM** mode, click the **Freeze** button below:

   > Click the **EXC** button to move the specimen stage to the exchange position.
   Note: click **Home** button if continue to do SEM imaging.

> Click **Set** button in the **Stage** tab; uncheck **FG-EDX** box below.



7) Check the **SEM SOP** closing steps to make sure all parameters are resumed to default settings.
8) **DO NOT** log off FOM from SEM computer while waiting for the EDS detector warmup to above **20 ˚C** (~ **20 minutes, be patient!**). **Never start sample transfer** while EDS detector is still being cooled. This will cause detector contamination.
9) During EDS detector warmup:
    > Make sure sample holder is at exchange (**EXC**) position
    > Data transfer is complete
10) Check to make sure the **EDS** detector temperature rises above **20 ˚C**.
11) Turn **ON** small LCD
12) Finish sample extraction from SEM chamber. Check SEM SOP if needed.
13) Turn **OFF** small LCD
14) Clean the sample holder and work bench.
15) Make sure both **PC-SEM** and **Esprit** program windows are not minimized.
16) Upload data to box.yale.edu.
17) Log off FOM on both **SEM** and **EDS** computers.
18) Sign off the logbook.