SEM Training Notebook

Lab Manager: Dr. Perry Cheung MSE Fee-For-Service Facility Materials Science and Engineering University of California, Riverside

June 6, 2019 (rev. 3.7)

Before you begin...

Complete the required safety training modules on UC Learning

- Laboratory Safety Orientation (Fundamentals) 2013
- Hazardous Waste Management
- Compressed Gas Safety
- X-Ray Safety
- □ Submit a copy of your Training Transcript to Lab Manager
- Review the MSE SEM Policies and Regulations
- Fill out the SEM FAU Authorization Form with PI signature
- Fill out the MSE 150, 250, 309 Authorization Form with PI signature
- Receive a user name and temporary password for Faces scheduling
- Arrange a time for SEM training with Lab Manager
- Schedule a 2 hour block on Faces for your training
- □ Familiarize yourself with the graphical user interface (GUI) :A D
- □ Familiarize yourself with SEM fundamentals: E K

A. GUI



B. Floating Toolbar – 1/2



MODE: Opens the context menu for selecting **Displaying Modes**



SPEED: Opens the context menu for selecting predefined Scan Speeds



MAG: Left-click sets the **Magnification** as active function. Right-click opens context menu with predefined values of magnification.



WD: Left-click sets the Focus as active function.



STG: Left-click sets the Stigmator as active function.

B. Floating Toolbar – 2/2



Brightness: Left-click sets the **Brightness and Contrast** control as active function



Auto: Left-click starts Automatic Brightness and Contrast



BI: Left-click sets the Beam Intensity as active function



Manual Column Centering: Left-click starts the manual column centering process



Acquire: Left-click starts the Image Acquisition



C. Sidebar – 2/5

Info Panel shows all the <u>important</u> <u>parameters</u> of the microscope, and at the same time allows a quick set-up of all the most frequently used functions.

	Info Panel	□ ?
Continual	Single	Acquire
Scan Mode:	WIDE F	FIELD
HV:	5.00 k\	(
Magnification:	8.5 x	
View field:	32729	μm
Speed:	1 (0.10	µs/pxl)
WD:	35.863	mm
Depth of Focus	: 737.19	6 mm
Stigmator:	0.0 % /	0.0 %
Shift:	0.0 µm	/-0.0 µm
Rotation:	0.00 de	eg
Beam Intensity	: 15.00	
Absorb. Curr:	30 pA	
Spot Size:	37000.	0 nm

- Continual button stops or starts scanning
- Single button starts scanning of a single frame and then stops scanning
- Acquire button starts the acquisition process
- *HV* button sets the High Voltage value as active function
- Depth of Focus shows estimated range sample surface is in focus
- Absorb. Curr. shows the electron current absorbed by the sample
- Spot Size shows the sample impinging beam size

C. Sidebar – 3/5

Detector Panel shows active detector.

Electron Beam Panel controls filament heating and high voltage range.

SEM D	etector	- ?	0	HV shows
SE	29.9 % / 9	2.4 %	0	Emission
Electro	n Beam		0	Live Time
HV: 5.00 kV	Emission:	102 µA		filament
Live Time: 160 h	Heating:	44.0 %	0	Heating s
HV Q	< Hea	t O	fi	filament h
500 (0 5) kV ×	Adjustmer	nt ≥@		
				Heat star
			<u> </u>	
		<u>H</u>	Adju	<i>ustment</i> op
	HV turns the	high volt	age c	on and off
			ר	
	own selects	nv range		

- SE indicates Secondary Electron detector is active
 - %/% shows Brightness/Contrast
- *HV* shows high voltage value
- o Emission shows current emitted
- Live Time shows total working time of filament
- Heating shows relative value of filament heating current in %

Heat starts or stops filament heating

Adjustment opens context menu

C. Sidebar - 4/5

Vacuum Panel controls the vacuum system.



- Column Pressure indicates the value of the pressure in the column
 - Red = Not Ready
 - Green = Ready
 - Status shows state of vacuum
 - Venting = still venting
 - Venting finished = venting is finished and chamber can be opened
 - **Pumping** = still pumping
 - Vacuum ready = chamber is pumped down to sufficient vacuum
 - Vacuum off = vacuum is in standby mode

C. Sidebar – 5/5

Nano Stage Control controls the specimen stage movement.



D. SEM Image Parameters

	SEM Image Parameters ? ×
Windows Aspect Ratio of Image = 4:3 Resolution = 1024 x 768	Windows 4:3 1024 x 768 Live: 4:3 1024 x 768 Save: 4:3 1024 x 768
Averaging Accumulation = Disable (Prone to vibrational noise and drift)	Averaging Live: Frame accumulation \checkmark 5 \blacklozenge Save: Frame accumulation \checkmark 5 \blacklozenge
Acquisition Keep Actual Speed Keep view field/print magnification	Acquisition Acquisition time: 0.12 s Keep actual speed Keep view field / print magnification
Infobar Texts Show Infobar: Beam Energy, Working Distance, View Field, Detector, Vacuum, Scan Mode	Infobar texts Show infobar Beam energy V Working distance V Magnification Detector V View field Date V Burn Note & Sign texts into image
	Apply

E. Accelerating Voltage – 1/2



E. Accelerating Voltage - 2/2



5 kV

10 kV

20 kV





G. Working Distance vs Focus (W/D) Distance



Actual Working Distance = Distance between objective pole piece and sample and can only be controlled manually with the knob outside the chamber

Focus (W/D) Distance = Distance between objective pole piece and focal point and can only be controlled by the Focus (W/D) button 15

H. Beam Intensity – 1/2



H. Beam Intensity – 2/2

Low Magnification (Minimum < Mag < 10 kX)



At Low Mag, lowering BI doesn't have a dramatic affect on the quality of image...

High Magnification (10 kX < Mag < Maximum)



At High Mag, the BI MUST be chosen correctly!

A grainy image will ALWAYS accompany a reduction in BI, but is easily removed with a drop in scan SPEED! 17



J. Tilt (Advanced Users) - 1/2

0° Tilt (Flat) 15° Tilt 30° Tilt VEGA3 TESCAN VEGA3 TESCAN SEM HV: 15.0 kV WD: 7.61 mm SEM HV: 15.0 kV WD: 7.56 mm SEM HV: 15.0 kV WD: 7.51 mm VEGA3 TESCAN View field: 250 µm Det: SE 50 µm View field: 250 µm Det: SE 50 µm View field: 250 µm Det: SE 50 um SM: RESOLUTION SM: RESOLUTION Print MAG: 508 x Print MAG: 508 x Print MAG: 508 x SM: RESOLUTION

J. Tilt (Advanced Users) - 2/2 **EXCELLENCE IN SCIENTIFIC INSTRUMENTATION**



K. High Resolution Imaging Process Tree

#	Description	Stage	Mag	Focus	Z Knob	BI	Speed	Auto B/C
1	Center tallest part of tallest sample in window	Yes	Yes	Yes		Yes	Yes	Yes
2	Achieve desired working distance			Yes	Yes	Yes	Yes	Yes
3	Center desired sample image in window with desired Mag	Yes	Yes	Yes		Yes	Yes	Yes
4	Increase Mag to ≥ 2X desired Mag		Yes	Yes		Yes	Yes	Yes
5	Beam optimization (if desired Mag ≥ 10 kX)			Yes		Yes	Yes	Yes
6	Achieve best focus			Yes		Yes	Yes	Yes
7	Reduce Mag back to desired Mag		Yes			Yes	Yes	Yes
8	Determine optimal image conditions for BI and Speed and acquire					Yes	Yes	Yes
9	Reduce Mag and acquire image		Yes			Yes	Yes	Yes
10	Move to new	<i>i</i> sample	location -	> Repeat	#3 to #9			21

SEM Operation

- I. Initiate Software
- II. Sample Preparation
- III. Sample Loading
- IV. Turning on HV
- V. Mode
- VI. Beam Intensity
- VII. Brightness and Contrast
- VIII. Mag
- IX. Focusing

- X. Speed
- XI. Working Distance
- XII. Image Preparation
- XIII. Column Centering
- XIV. Stigmation Correction
- XV. Image Acquisition
- XVI. Saving
- XVII. Sample Unloading
- XVIII. Cleanup

I. Initiate Software – 1/1

- 1. Record your time-in on the sign-in sheet located on preparation table
- 2. Sign into Windows using provided **Username** and **Password** located on **monitor** if necessary
- 3. Double-click on VegaTC icon to load software



4. Sign into your user account with your **Username** and **Password**



II. Sample Preparation – 1/2

- **1.** Always wear gloves when dealing with anything that will be placed into or in contact with the SEM
- 2. The specimen should be conductively fixed with double-sided carbon tape to a specimen stub (12.5 mm specimen pin-stubs)



Double-sided carbon tape (DCT)





SEM specimen stub

3. For non-conductive samples, "sandwich" the carbon tape around your specimen to provide a conductive pathway



II. Sample Preparation – 2/2

4. Follow the steps for operating the Sputter Coater (requires training) to deposit a thin-layer of gold

- 5. Magnetic samples will need to be fixed well by a screw holder (provided by user)
- 6. Items located in the cabinet are available for SEM users to help prepare their samples







III. Sample Loading – 1/4 Vacuum 1. Click **VENT** to vent the microscope 2. Click **Yes** to confirm venting) VENT STANDBY Venting finished. Vent the chamber? Yes No 3. Wait until *"Venting finished"* appears 4. Set the tilt of the specimen stage to 0° if not already set to 0° (Advanced Users only) 5. Gently pull the chamber corners toward you to open the chamber TURN CLOCKWISE = **IOWER STAGE**

1.0e+005 Pa

PUMP

III. Sample Loading – 2/4

6. Rotate stage if necessary to access screw port in preferred orientation for maximum clearance

Nano Stage Cont...

7. Using provided tweezers, clamp onto the specimen stub and **blow a stream of air** over the **entire specimen stub** AWAY from the chamber using Airgun

- 8. Loosen the screw first (see example)
- 9. Carefully insert the specimen stub into the specimen stage
- 10. Tighten the screw holding the specimen stub









III. Sample Loading – 3/4

- 11. Ensure that the sample stage is at the lowest position using Z-knob (clockwise)
- 12. Check to see if the O-ring along the chamber door is snugly sitting inside the recessed groove
- Not Snug! Snug Snug
- If loose, WEARING CLEAN GLOVES run your finger = along the O-ring to ensure that it is sitting inside the recessed groove
- 14. Carefully close the chamber door by pushing it towards the chamber

CHECKING THAT THE SAMPLE DOES NOT TOUCH ANYTHING INSIDE CHAMBER





III. Sample Loading – 4/4

15. Ensure there is no gap between chamber and door, else fix O-ring following steps 12 and 13 again



- 16. Place finger against chamber door
- 17. Click **PUMP** to start pumping down chamber =



Column pressure:

VENT

STANDBY

Vacuum ready.

- 18. Wait until bar graph shows red to release finger
- 19. Wait until the bar graph turns green or *"Vacuum ready"* appears (~ 3 min)

4.2e-002 Pa

PUMP

URN CLOCKWISE =

LOWER STAGE



V. Mode – 1/1

- 1. Click *MODE*
- 2. Confirm *Continual Wide Field* option is checked

MODE

3. Choose desired scanning mode (default = Resolution)

	Mode	Characteristics
А	Resolution	High resolution Lower depth of focus
В	Depth	Good resolution Increased depth of focus
С	Field	Lower resolution Large field of view High depth of focus
D	Wide Field	Extra large field of view



4. Right-click on *MAG* and select = *Minimum Magnification* _____

VI. Beam Intensity – 1/1

- 1. Center the SEM window onto your desired sample using the stage control _____
- Click *BI* to adjust beam intensity ______ using the << and >> ______

Recommended Initial **BI** values

Magnification	Beam Intensity
$Min \rightarrow 200$	$18 \rightarrow 13$
200 → 2000	$12 \rightarrow 8$
$2000 \rightarrow 10k$	$10 \rightarrow 7$
$10k \rightarrow Max$	$7 \rightarrow 4$

- 3. Recommend **BI** of **15** to start at low mag
- 4. Change the sensitivity if necessary

Recommended Value = 3



VII. Brightness and Contrast – 1/1

- 1. Click **Auto** to auto adjust the brightness and contrast if too bright or dark as necessary
- 2. Click **Brightness** to manually adjust the brightness and contrast

Hold F12 + 🛑 trackball Contrast: Brightness: Hold F11 + **T** trackball

3. Click on the *IR Camera* button to open up the view of the chamber (if you haven't already)











VIII. Mag - 1/1

1. Click **MAG** to change the magnification



Pad

Reset

х

>>

>

v

OK

Undo

Magnification

3

200.0

<<

2. Turn the trackball from left to right



4. Change the sensitivity if necessary =

Recommended Value = 5

IX. Focusing – 1/1

- 1. Click *WD* to adjust focus distance
- 2. Turn Trackball from left to right to adjust focus (*Resolution* or *Depth* mode only)
- 3. A focused image shows the actual working distance via WD value
- Change the sensitivity if necessary ______
 Recommended Value = 2 for Fine (Mag ≥ 10kX) and 5 for Coarse (Mag ≤ 10kX)
- 5. Double-left-click in the SEM scanning window to create a *Focus Window*
 - Left mouse button inside = move *Focus Window*
 - Right mouse button inside = resize *Focus Window*
 - Double-left-click = remove *Focus Window*
- 6. WD \approx 30 mm when sample is at lowest position







X. Speed – 1/1

- 1. Click **SPEED** to adjust scan speed
- 2. Use Focus Window to determine the effect of *SPEED* and *BI* has on your image quality

Recommendation:

SPEED of 1 - 4 for **initial focusing**

BI setting should be appropriate to **MAG** value

SPEED of higher values looks better but takes longer to focus!

Use higher SPEED values of 5 – 8 when ready to save images

13 min 58 sec

44 min 4 sec

	SPEED	Acquisition Time
	1	0.12 sec
	2	0.30 sec
	3	0.87 sec
	4	3 sec
Ľ	5	16 sec
	6	32 sec
	7	1 min 36 sec
	8	4 min 34 sec

Recommended Initial **BI** values

9

10

	Magnification	Beam Intensity
•	$Min \rightarrow 200$	$18 \rightarrow 13$
	$200 \rightarrow 2000$	$12 \rightarrow 8$
	2000 ightarrow 10k	$10 \rightarrow 7$
	$10k \rightarrow Max$	$7 \rightarrow 4$



XI. Working Distance – 1/3

Use combination of *MAG*, *Stage Control*, and focusing (*WD*)

- a. Identify and bring the **tallest position** of your **tallest sample** to the **center** of SEM scanning window
- b. Increase MAG until distinct features make up majority of window
- c. Check if mode = *Resolution* or *Depth* (if not, keep increasing *MAG*)
- d. If you can't see transition between focus & out-of-focus with WD, you skipped a step!

NOTE: The tallest portion of the tallest sample should be focused since this will crash into the pole-piece first as you raise the stage in the next step.

This DOES NOT have to be the desired position or sample for your images, it is ONLY for setting the safe working distance value!







₩wD





XII. Image Preparation – 1/2

Imaging at **MAG** ≥ 10 kX requires optimization

steps XIII. Column Centering and XIV. Stigmation

Correction after completion of XII. Image

Preparation, else skip and proceed next to XV. Image
Acquisition directly

1. Right-click on *MAG* and select *Minimum Magnification* to see your whole sample again

 Identify an area of interest on your sample to image by using a combination of *MAG*, *Stage Control*, focusing (*WD*), and *BI*



<u>Example</u>





XII. Image Preparation – 2/2

3. Bring the area of interest to the **center** of SEM scanning window and to the **highest desired** magnification (e.g. Desired Mag = 10 kX)

You will **NOT** use the *Stage Control* after this step, so **ENSURE** that the image at the Desired Mag is the one you wish to take before continuing

 Increase MAG by ≥ 2X the desired Mag using the Pad (e.g. New Mag = 20 kX, 30 kX, etc...)

Higher **MAG** yields better results but gets more difficult to optimize

- 5. Reduce *BI* if necessary to increase resolution
- 6. Change scan *SPEED* to 3 or 4 to remove graininess
- 7. Focus (*WD*) your sample again







Recommended Initial **BI** values

Magnification	Beam Intensity
$Min \rightarrow 200$	$18 \rightarrow 13$
$200 \rightarrow 2000$	$12 \rightarrow 8$
$2000 \rightarrow 10k$	$10 \rightarrow 7$
$10k \rightarrow Max$	$7 \rightarrow 4$ 41

XIII. Column Centering – 1/3

- 1. Create a *Focus Window* around a feature of interest
- 2. Click *WD* and bring the feature into focus
- 3. If image moves or shifts as you focus, then column centering needs to be completed and continue to **Step 5**
- 4. If image does not move or shift, proceed to XIV. Stigmation Correction



Under-focused





Over-focused





5. Click *Manual Column Centering* button

- 6. The Manual Centering Wizard will appear
- 7. Click *Next>>*

XIII. Column Centering – 2/3

8. Your image will now "wobble" in and out of focus

If image has any X or Y translation as it wobbles, you will need to remove it



9. Minimize image movement by adjusting the OBJ Centering using the trackball



= Change only X-movement

= Change only Y-movement

XIII. Column Centering – 3/3

- 8. The image should remain stationary with no X or Y translation but only oscillate in/out of focus
- 9. Adjust the sensitivity to finely control the **OBJ Centering** if necessary

Recommended Value = 5 first then 2

- If flashing trackpad is present, click = << Previous and Next >> to reset
- 11. Adjust the *Wobbler sensitivity* to change the extent of "wobble" if necessary at very —— high magnifications
- 12. Click *Finish* when done





XIV. Stigmation Correction – 1/4

- 1. Create a *Focus Window* on a feature of interest
- 2. If Stigmation corrected, a focused image will become **significantly sharper**



3. Click *WD* and bring the feature **in and out-of-focus** (both sides) to check if any **streaking** occurs on **non-straight features** – **stop at Focused position**



4. Any streaks are evidence that *Stigmation Correction* is necessary

XIV. Stigmation Correction – 2/4

- 5. Set **SPEED = 4** + appropriate **BI** (see table)
- 6. Click *WD* and create a *Focus Window*
- Focus on a feature (*WD* Sensitivity = 2) as BEST AS YOU CAN
- 8. Click the *STG* to set as active function
- 9. Set **STG Sensitivity = 6** (slow down trackball for accuracy near "sweet spot")
- 10. Achieve a sharper image by adjusting the Stigmators one at a time (X and Y)



Recommended Initial **BI** values

Magnification	Beam Intensity
$Min \rightarrow 200$	$18 \rightarrow 13$
200 → 2000	$12 \rightarrow 8$
$2000 \rightarrow 10k$	$10 \rightarrow 7$
$10k \rightarrow Max$	$7 \rightarrow 4$

- = Change the X-component
- = Change only Y-component
- **11. CAREFULLY AND SLOWLY** adjust each Stigmator component (X and Y) until you can identify the *"perfect"* or setting with the sharpest image
- 12. REPEAT Steps 6 11 until you no longer see any improvement in sharpness

XIV. Stigmation Correction – 3/4

- 13. If your image still doesn't look "good", 99% it's because of poor STG Correction
- 14. The sequence of *STG Correction* should resemble the following:



15. Repeat the sequence as necessary until the image looks "good"

XIV. Stigmation Correction – 4/4

- 16. Proper STG Correction is EXTREMELY sensitive
- 17. A few % values off from "perfect" setting, and your image will look very blurry!



18. If this is the case, GO BACK AND RE-DO the STG Correction!

XV. Image Acquisition – 1/3

Create *Focus Window* and achieve the 1. **BEST** focus (Recommend Sensitivity = 2)

(Do **NOT** focus again **AFTER** this step!)

Click **MAG** and set back to desired 2. magnification (e.g. Desired Mag = 10 kX)

3. Activate the *Focus Window* over a desired feature

Smaller window = requires less time to refresh



Example







MAG



XV. Image Acquisition -2/3

- 4. Identify maximum **Acquisition Time** for your image (e.g. 2 min) and select corresponding **Speed** (e.g. **SPEED** = 7)
- 5. Adjust the **BI** until a balance between resolution is matched with graininess
- 6. Click *Auto* to auto adjust the brightness and contrast as you change the *BI*
- 7. Click **Brightness** to manually adjust and fine tune the brightness and contrast
- If high resolution is desired but excessive graininess is present, increase the Acquisition Time (e.g. SPEED = 7 -> 8)
- Repeat Steps 5 6 until desired balance between resolution and graininess and is achieved (e.g. see next slide for examples)

1	0.12 sec	
2	0.30 sec	
3	0.87 sec	
4	3 sec	
5	16 sec	
6	32 sec	
7	1 min 36 sec	
8	4 min 34 sec	
9	13 min 58 sec	
10	44 min 4 sec	
		_

SPEED

Acquisition Time





XV. Image Acquisition – 3/3

II 8



XVI. Saving – 1/1

Click **Acquire** to capture image

1.



2. If desired, you may save information = to the image file

Note = the basic description
Sign = the enlarged description
Description = the detailed information
Add = saves the Note or Sign in the list
Delete = deletes the Note or Sign from the list

3. If you choose not to include any Header information, click **OK**



	• [Add Delete
	• [Add Delete
	✓	Add Delete
	~	Add Delete
	~	Import
	Ÿ	
VE042 0011	Courses Mars	10.11.
VEGA3 SBH	Source Mag: Divel Size:	10.14 X
MSE161	Accumulation:	20.07 I µIII 1 frame
MOL TOT	Accounting of the	1 marrie
0.0.10.0	Em Ourset	404.0
0.070.0µm	Em. Current:	101.9 µA
0.0 *	vacuum:	4.3e-002 Pa
 27.51.nA		
20592.0 nm	Stopp (x y z):	
· 211/016%	0.00 0.00 0.00 m	
0.0 °	Stage (rt):	
SE	0.0. 0.0 deg	
	,	C
	VEGA3 SBH 115-0069 MSE161 0.0 / 0.0 µm 0.0 ° urr: 37.51 pA 30582.9 nm : 31.1 / 91.6 % 0.0 ° SE	VEGA3 SBH 115-0069 MSE161 0.0 / 0.0 µm 0.0 * 1.15-0069 MSE161 Accumulation: 0.0 * Vacuum: 1.15-0069 MSE161 Accumulation: 0.0 * Vacuum: 1.15-0069 Vacuum: 1.15-0069 SE2 9 nm 1.15-0069 Stage (x,yz): 1.1 / 91.6 % 0.0 0, 0.0 deg

Line days of Course Million and



XVII. Sample Unloading – 2/3



XVII. Sample Unloading – 3/3

Nano Stage Cont...

- 12. Gently pull the chamber corners toward you to open the chamber
- 14. Rotate the stage if necessary to access screw port ______

15. Using the provided tweezers, carefully remove the specimen stub out of the specimen stage



16. Tighten the screw back so it doesn't stick out

TURN CLOCKWISE =

XVIII. Cleanup – 1/1

- 1. Ensure sample stage is at lowest position
- 3. Carefully close the chamber door and check there is no gap
- 4. Place finger against chamber door



- or "Vacuum ready" appears (~ 3 min) —
- 8. Open File menu and select Logoff, click Yes
- 9. Record your total time used in Sign-in sheet



Vacuum

VENT

Vacuum

VENT

JRN CLOCKWISE

2.9e+000 Pa

PUMP

4.2e-002 Pa

PUMP

No Gap

Column pressure:

STANDBY

Vacuum ready.