SEM Training Notebook

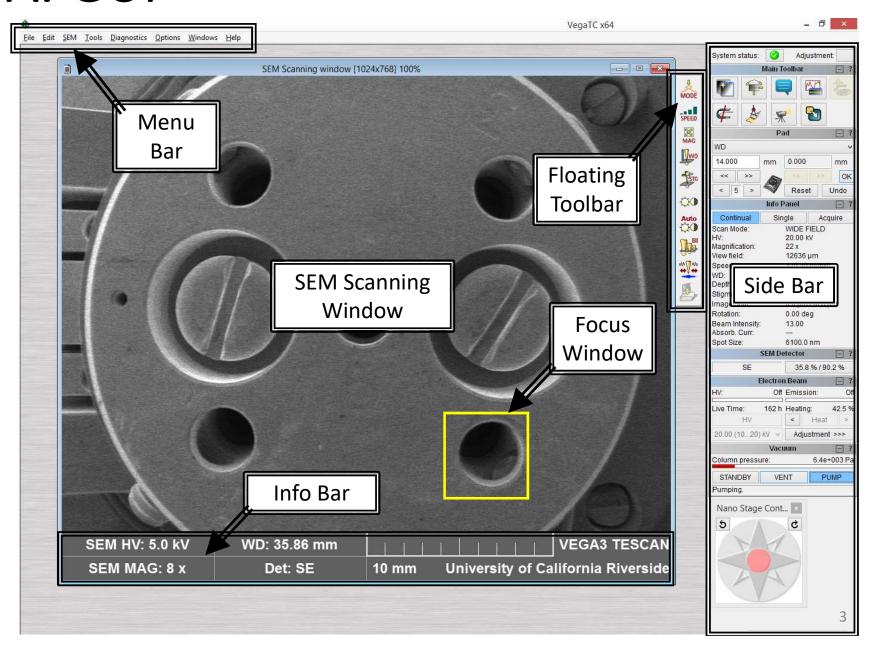
Lab Manager: Dr. Perry Cheung
MSE Fee-For-Service Facility
Materials Science and Engineering
University of California, Riverside

March 8, 2018 (rev. 3.5)

Before you begin...

Con	nplete the required safety training modules on UC Learning			
	Laboratory Safety Orientation (Fundamentals) 2013			
	Hazardous Waste Management			
	Compressed Gas Safety			
	X-Ray Safety			
Sub	mit a copy of your Training Transcript to Lab Manager			
Rev	iew 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			
Rec	eive a user name and temporary password for Faces scheduling			
Arra	ange a time for SEM training with Lab Manager			
Sch	edule a 2 hour block on Faces for your training			
Familiarize yourself with the graphical user interface (GUI) :A – D				
Farr	niliarize yourself with SFM fundamentals: F – 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.

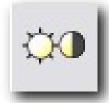


WD: Left-click sets the Focus as active function.



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



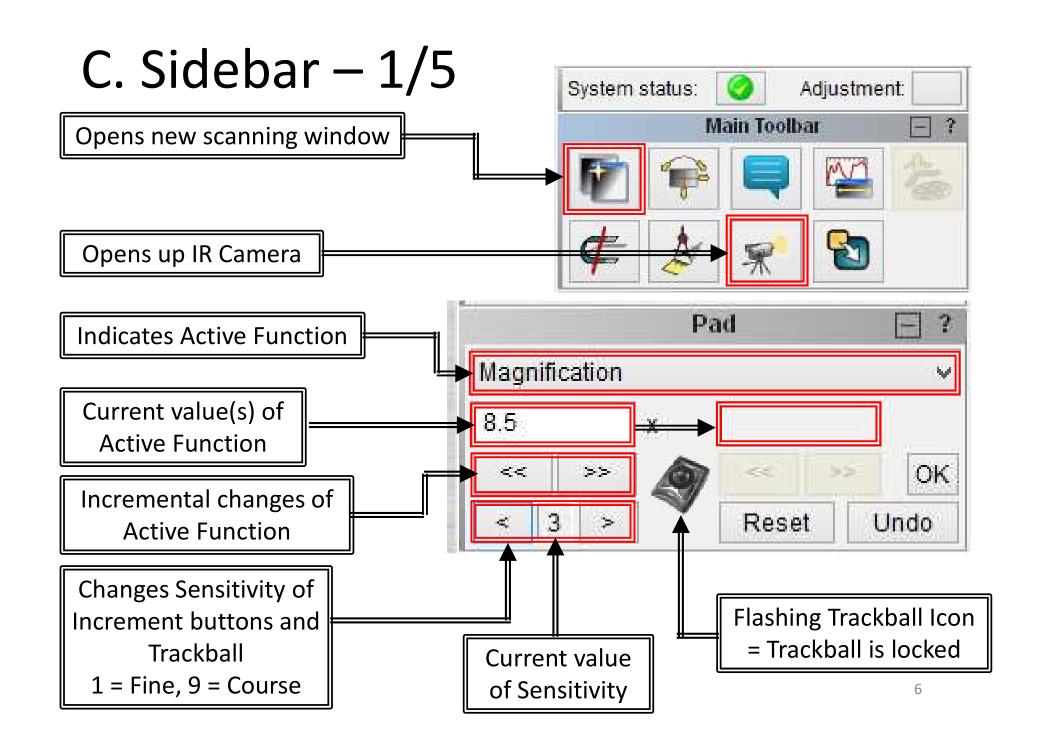
BI: Left-click sets the Beam Intensity as active function



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

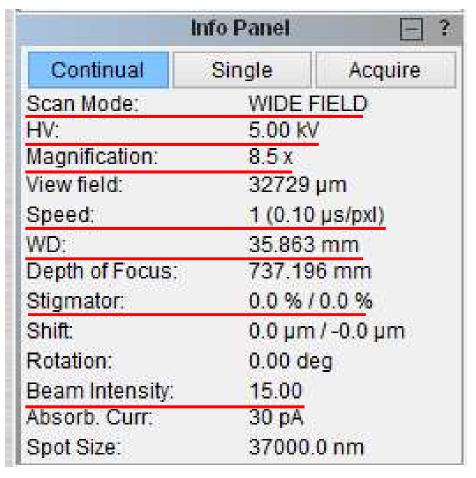


Acquire: Left-click starts the Image Acquisition



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.

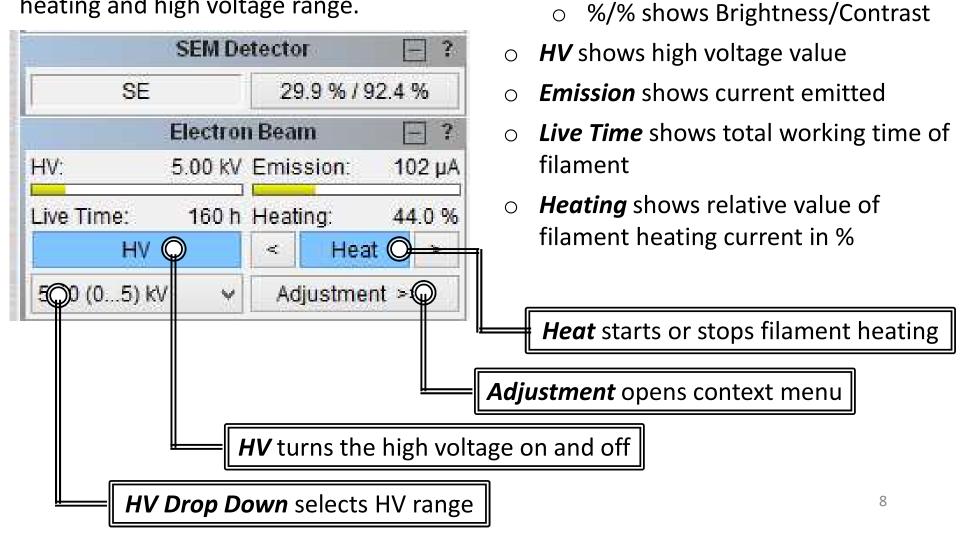


- Continual button stops or starts scanning
- Single button starts scanning of a single frame and then stops scanning
- Acquire button starts the acquisition process
- HV button sets the High Voltage value as active function
- Depth of Focus shows estimated range sample surface is in focus
- Absorb. 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.

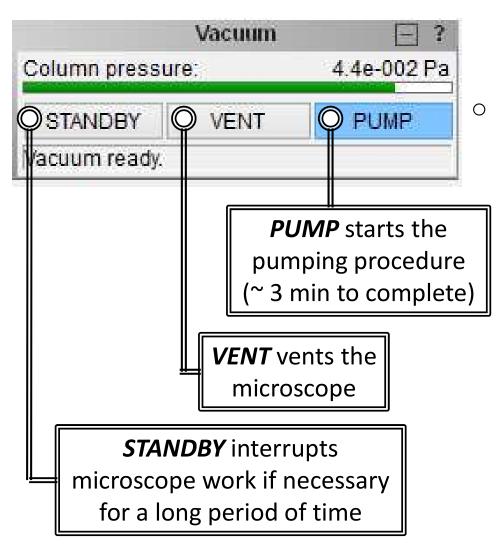


SE indicates Secondary Electron

detector is active

C. Sidebar -4/5

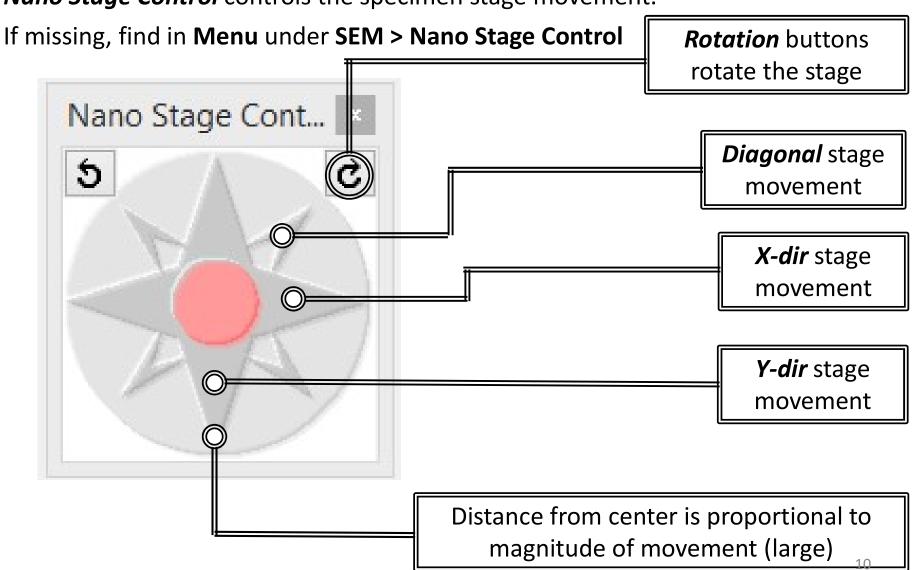
Vacuum Panel controls the vacuum system.



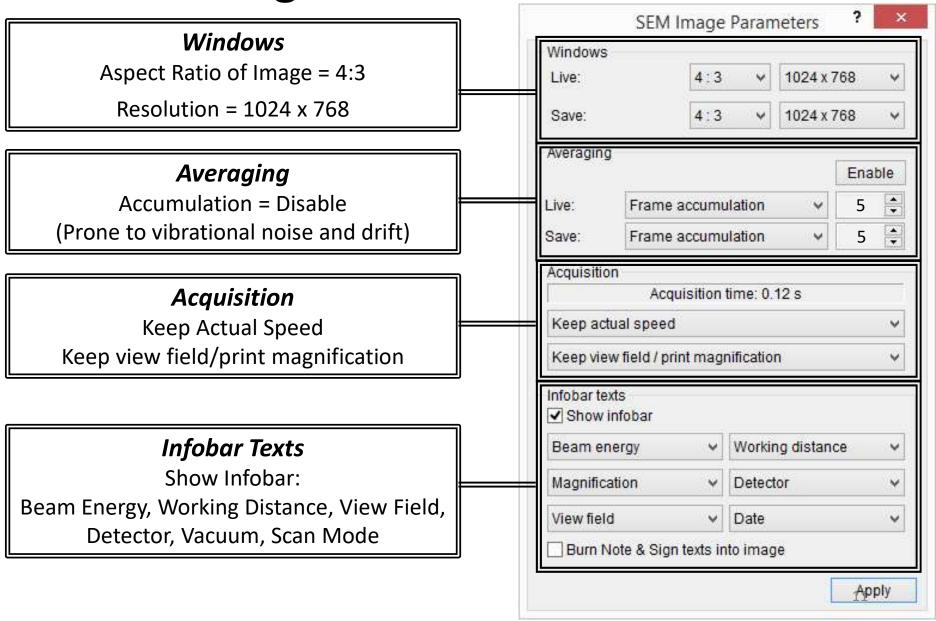
- **Column Pressure** indicates the value of the pressure in the column
 - Red = Not Ready
 - Green = Ready
 - **Status** shows state of vacuum
 - Venting = still venting
 - Venting finished = venting is finished
 and chamber can be opened
 - Pumping = still pumping
 - Vacuum ready = chamber is pumped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

C. Sidebar -5/5

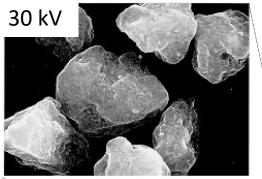
Nano Stage Control controls the specimen stage movement.



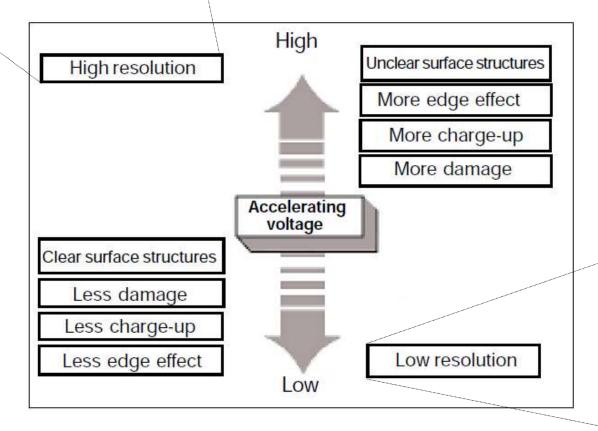
D. SEM Image Parameters

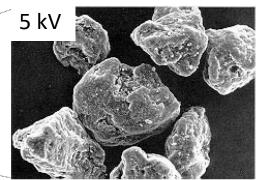


E. Accelerating Voltage – 1/2

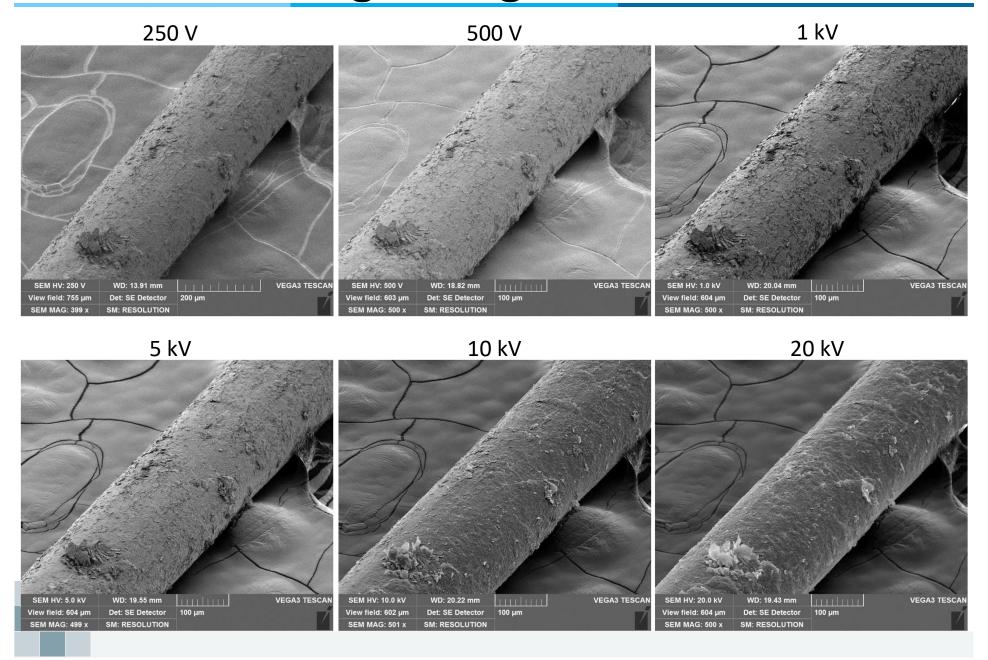


Recommendation: Start at **5 kV** and increase voltage incrementally to balance resolution to surface structures

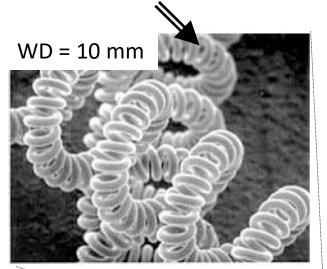




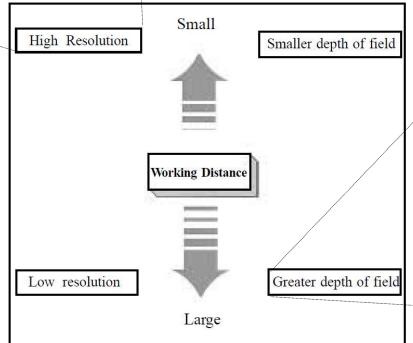


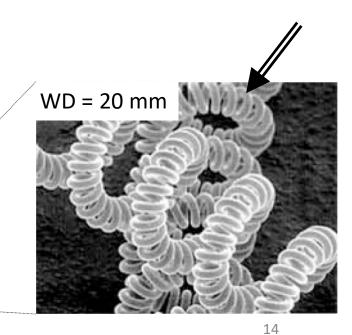


F. Working Distance

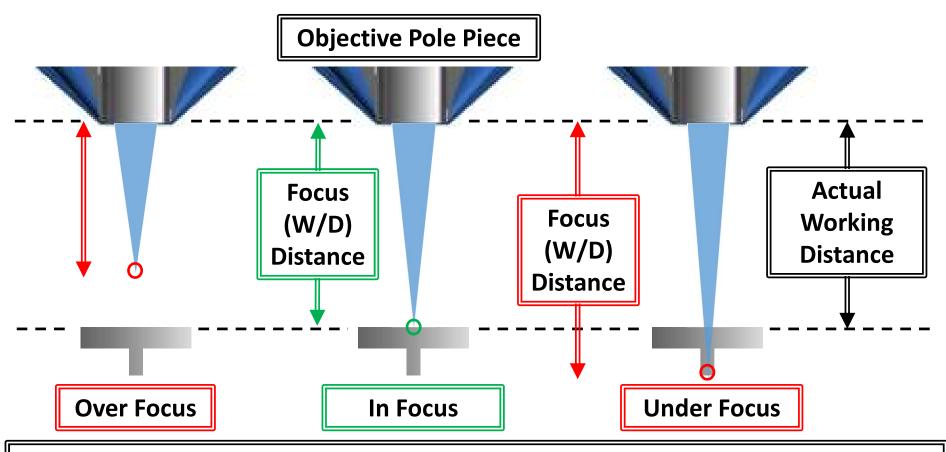


Recommendation: Start at ≈ **10** mm and decrease WD to achieve greater resolution or increase WD to achieve greater depth of field if necessary





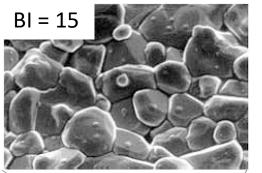
G. Working Distance vs Focus (W/D) Distance



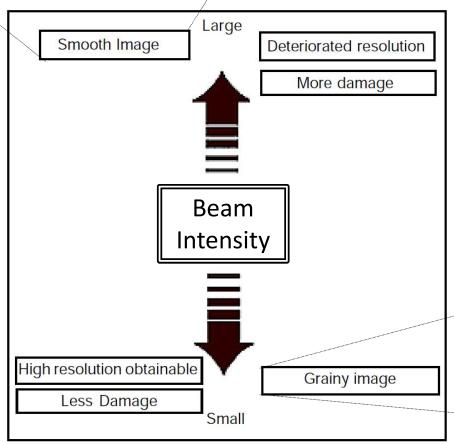
Actual Working Distance = Distance between objective pole piece and sample and can only be controlled manually with the knob outside the chamber

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

H. Beam Intensity – 1/3

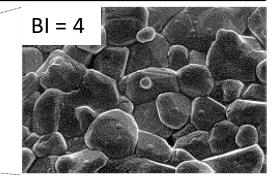


Recommendation: Decrease beam intensity until balance between resolution/grainy image and acquisition time is desired



High BI: Larger spot size for low magnification but poor resolution

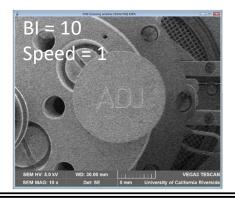
Low BI: Higher resolution but grainier image, need to balance with slower scan SPEED

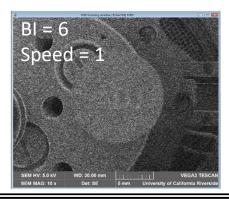


H. Beam Intensity – 2/3

Low Magnification (Minimum < Mag < 10 kX)



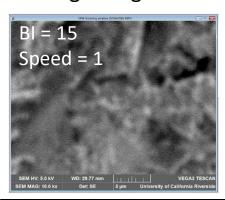


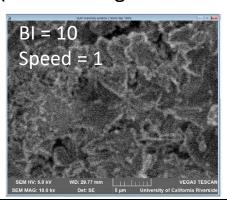


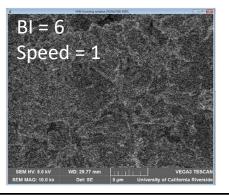


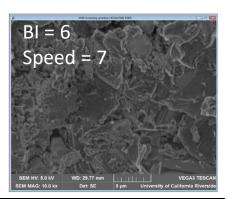
At Low Mag, lowering BI doesn't have a dramatic affect on the quality of image...

High Magnification (10 kX < Mag < Maximum)



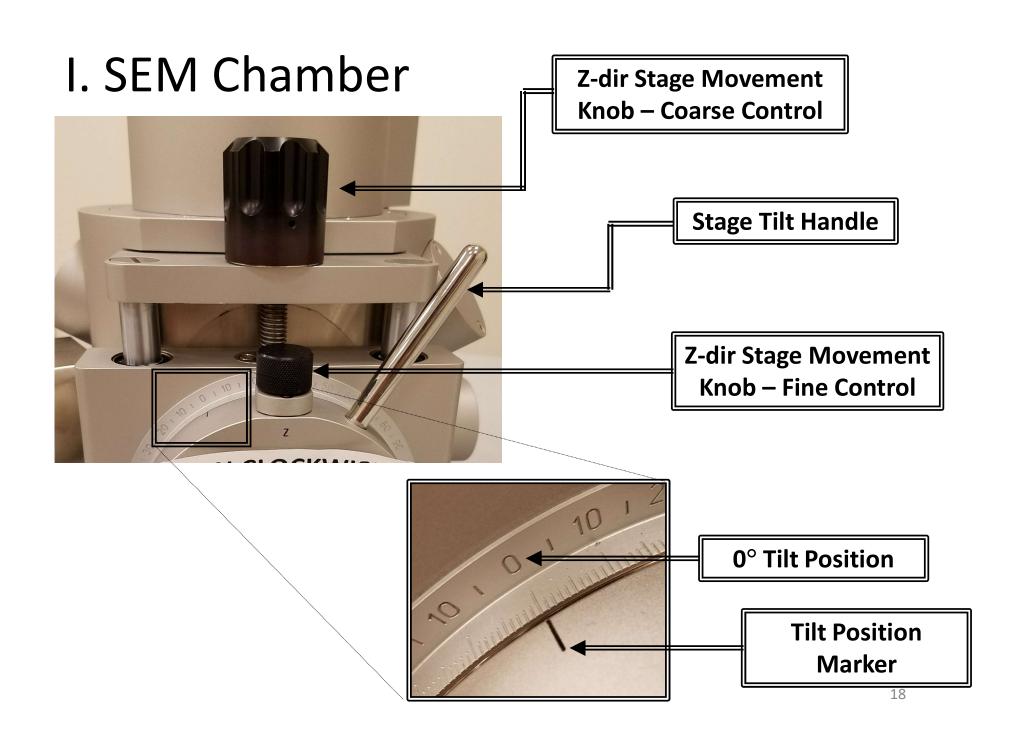






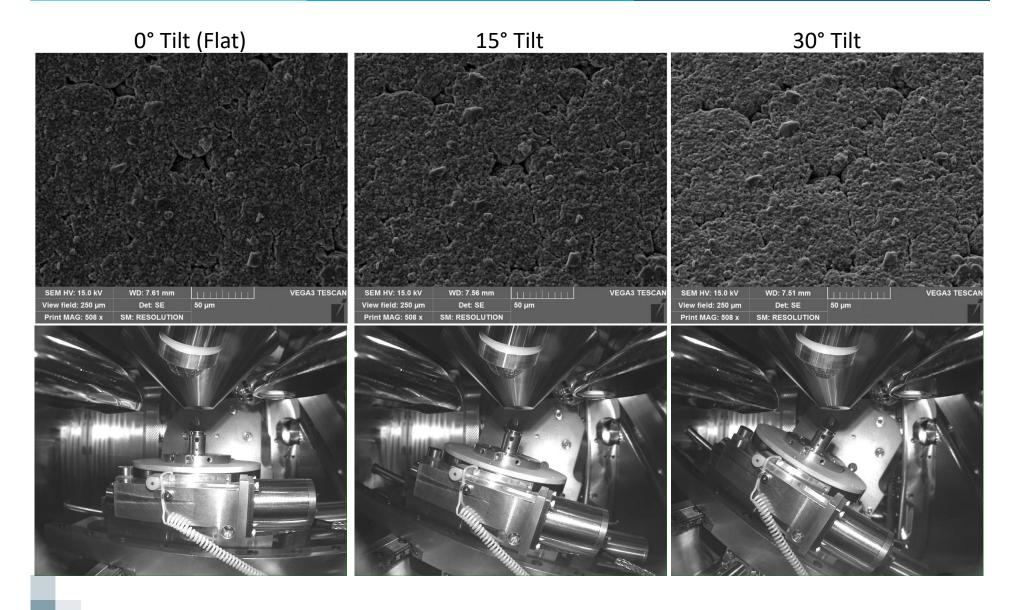
At High Mag, the BI MUST be chosen correctly!

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



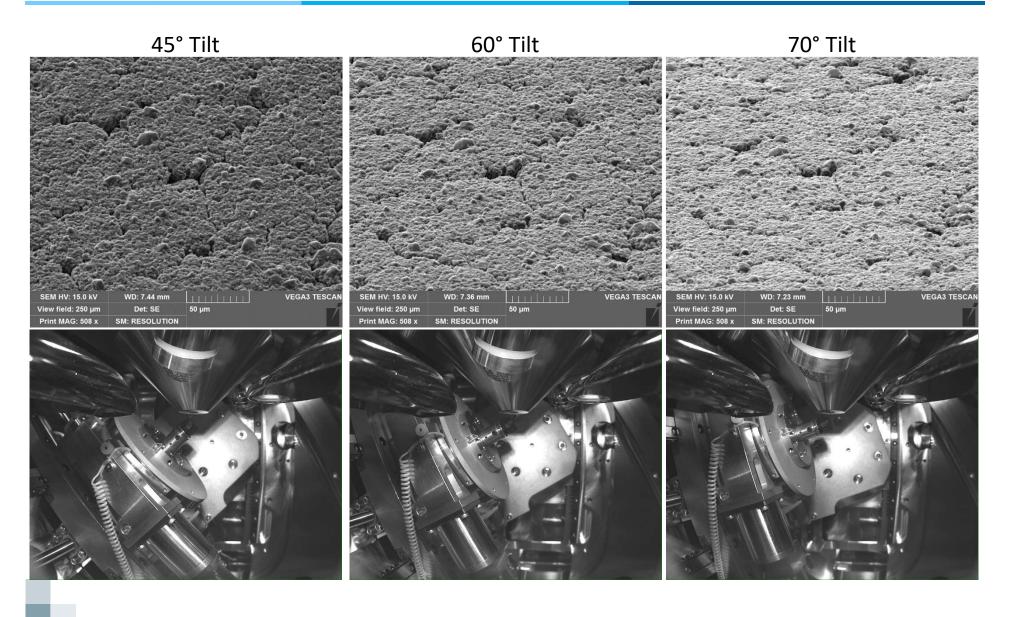
J. Tilt (Advanced Users) - 1/2 TESCAN ORSAY HOLDING EXCELLENCE IN SCIENTIFIC INSTRUMENTATION





J. Tilt (Advanced Users) - 2/2 TESCAN ORSAY HOLDING EXCELLENCE IN SCIENTIFIC INSTRUMENTATION





K. High Resolution Imaging Process Tree

		_						
#	Description	Stage	Mag	Focus	Z Knob	ВІ	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	sample	location -	> Repeat	#3 to #9		á	21

SEM Operation

I.	Initiate Software	Χ.	Speed
II.	Sample Preparation	XI.	Working Distance
III.	Sample Loading	XII.	Image Preparation
IV.	Turning on HV	XIII.	Column Centering
V.	Mode	XIV.	Stigmation Correction
VI.	Beam Intensity	XV.	Image Acquisition
VII.	Brightness and Contrast	XVI.	Saving
VIII.	Mag	XVII.	Sample Unloading
IX.	Focusing	XVIII.	Cleanup

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 – 1/1

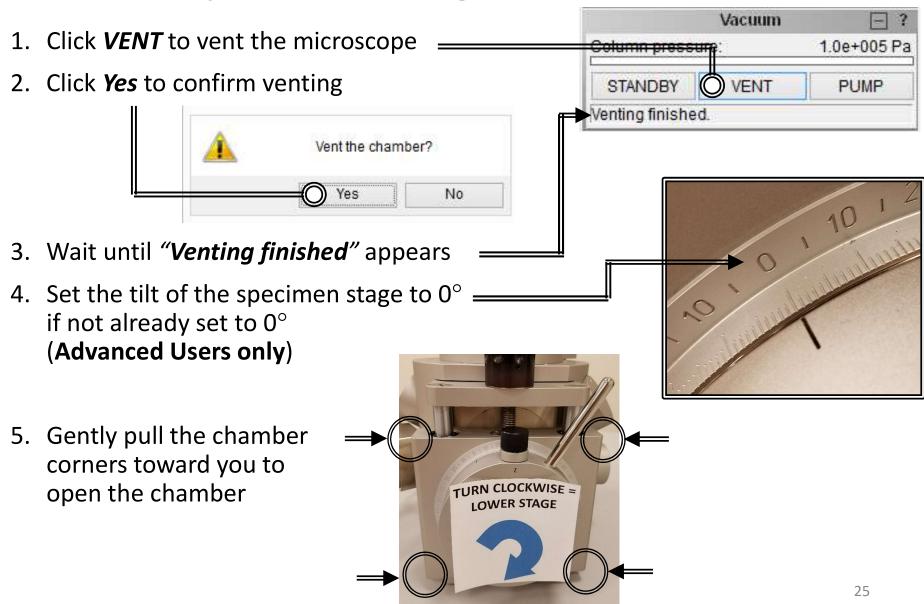
- 1. Always wear gloves when dealing with anything that will be placed into or in contact with the SEM
- 2. The specimen should be conductively fixed or glued to a specimen stub (12.5 mm specimen pin-stubs)

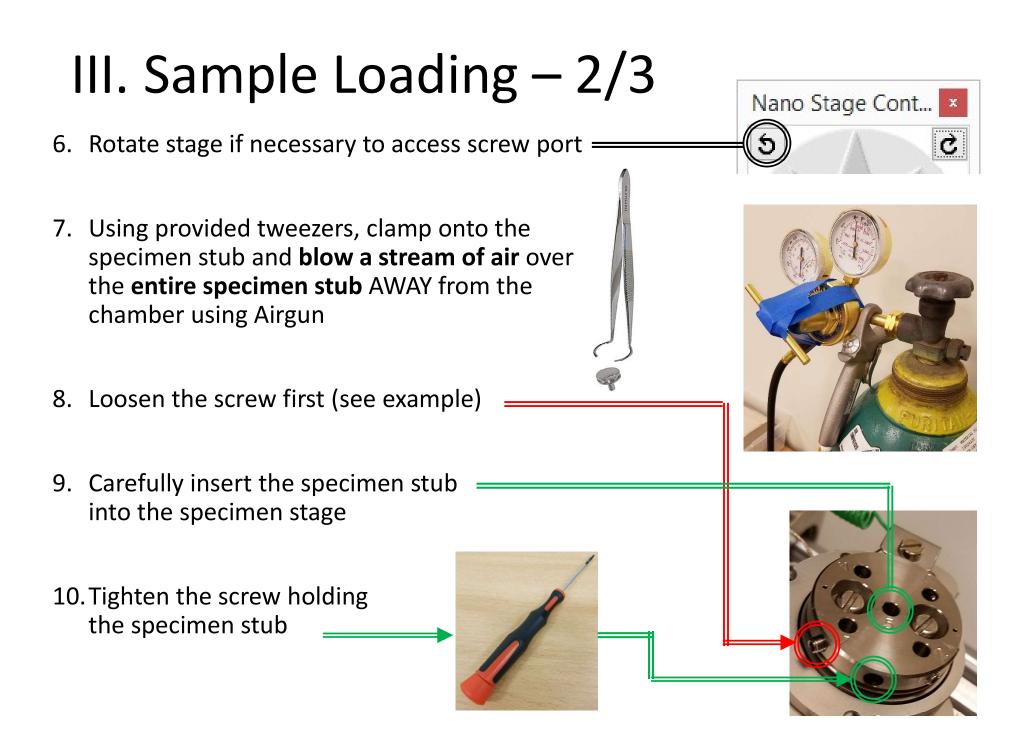


- 3. Non-conductive samples need to be coated by a conductive layer using either a carbon coater or sputter coater (coming soon to MSE)
- 4. Magnetic samples will need to be fixed well by screw holder (provided by user)
- 5. Items located in the cabinet are available for SEM users to help prepare their samples



III. Sample Loading – 1/3





III. Sample Loading – 3/3

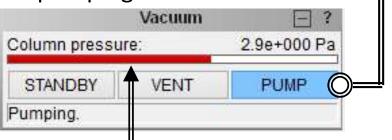
11. Ensure that the sample stage is at the lowest position using Z-knob (clockwise)



12. Carefully close the chamber door by pushing it towards the chamber

CHECKING THAT THE SAMPLE DOES NOT TOUCH ANYTHING INSIDE CHAMBER

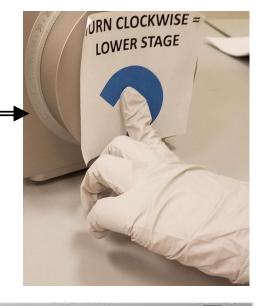
- 13. Place finger against chamber door
- 14. Click **PUMP** to start pumping down chamber

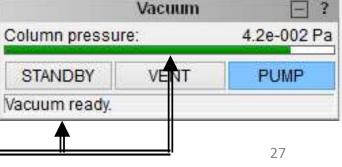


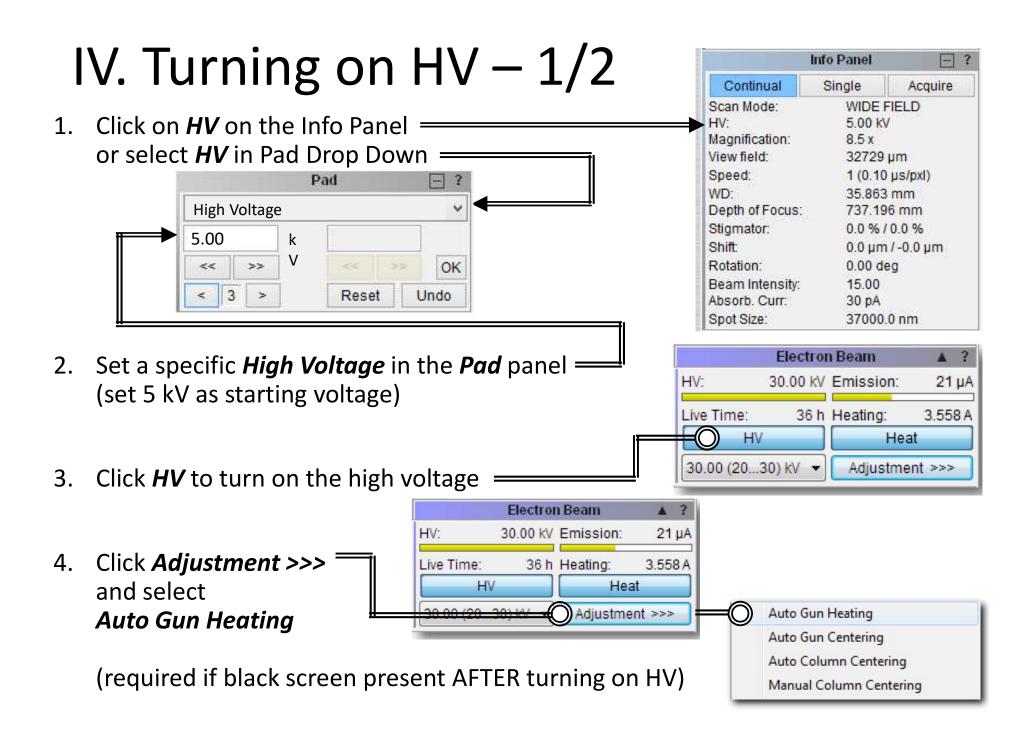
15. Wait until bar graph shows red to release finger

16. Wait until the bar graph turns green or "Vacuum ready" appears (~ 3 min)







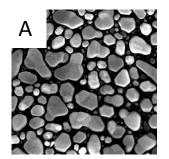


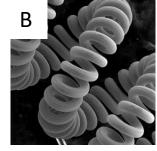
V. Mode – 1/1

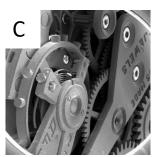


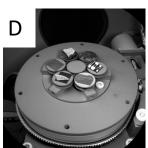
- 1. Click **MODE**
- 2. Confirm *Continual Wide Field* option is checked
- 3. Choose desired scanning mode (default = Resolution)

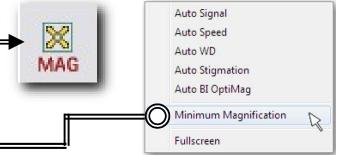
	Mode	Characteristics
А	Resolution	High resolution Lower depth of focus
В	Depth	Good resolution Increased depth of focus
С	Field	Lower resolution Large field of view High depth of focus
D	Wide Field	Extra large field of view











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



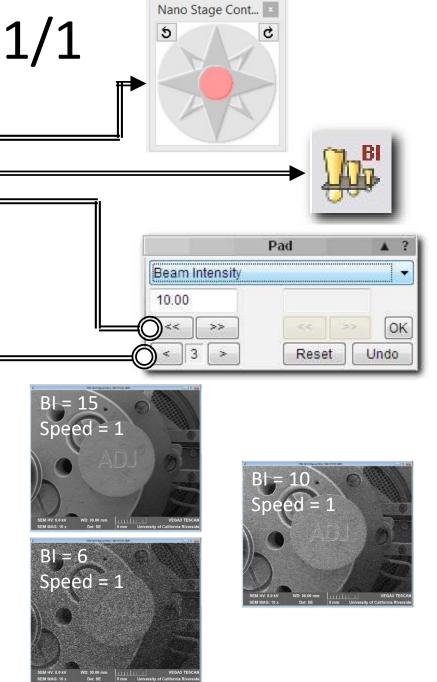
- Center the SEM window onto your desired sample using the stage control

Recommended Initial **BI** values

Magnification	Beam Intensity
Min – 200	13 – 18
200 – 2000	8 – 12
2000 – 10k	7 – 10
>10k	4 – 7

- 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

Click **Auto** to auto adjust the brightness and contrast if too bright or dark as necessary



Click **Brightness** to manually adjust the brightness and contrast 2.



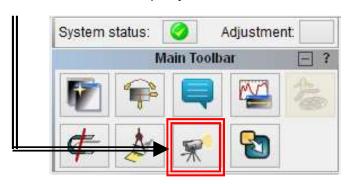
Contrast: Hold F12 + trackball

= Change only Contrast

Brightness: Hold F11 + **1** trackball

= Change only Brightness

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



2. Turn the trackball from left to right



Pad

Reset

OK

Undo

Magnification

200.0

<<

3. Or enter a value directly in *Pad* panel



Recommended Value = 5

IX. Focusing -1/1

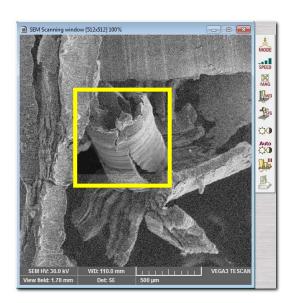




- 1. Click **WD** to adjust **focus distance**
- 2. Turn the Trackball from left to right to adjust focus
- 3. A focused image shows the actual working distance via WD value



- 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!

SPEED	Acquisition Time
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

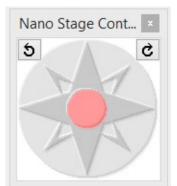
Recommended Initial **BI** values

Magnification	Beam Intensity
Min – 200	13 – 18
200 – 2000	8 – 12
2000 – 10k	7 – 10
>10k	4 – 7

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

XI. Working Distance – 1/3

MAG

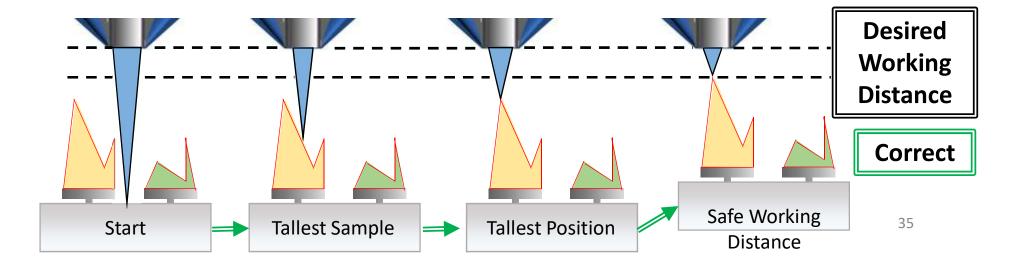


Use combination of *MAG*, *Stage Control*, and focusing (*WD*)

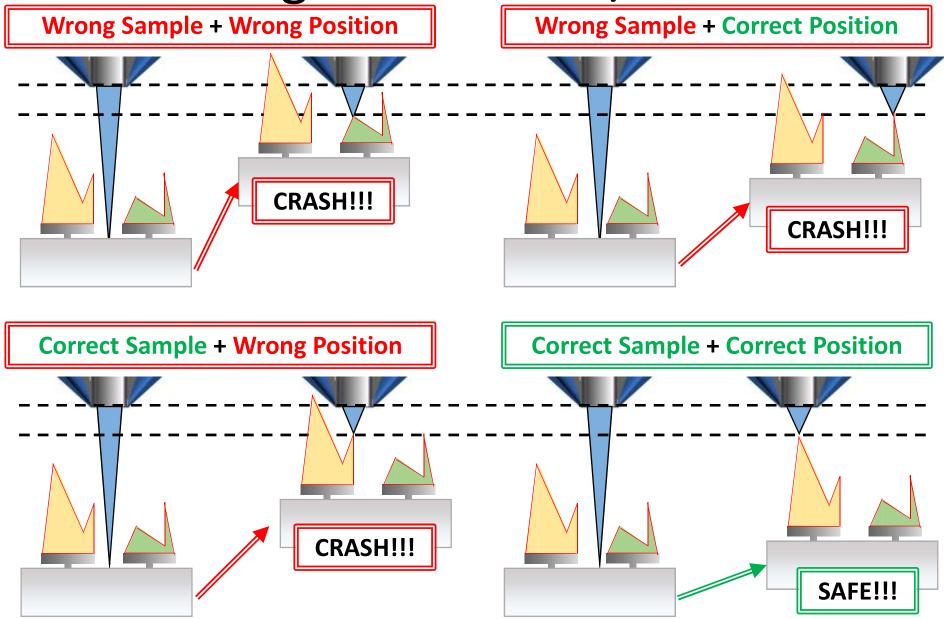
- a. Identify and bring the **tallest position** of your **tallest sample** to the **center** of SEM scanning window
- **b.** Increase MAG until distinct features make up majority of window
- c. Check if mode = **Resolution** or **Depth** (if not, keep increasing **MAG**)
- d. If you can't see transition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



XI. Working Distance – 2/3



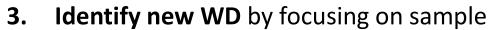
XI. Working Distance – 3/3

PROCEED WITH CAUTION AS CHANGING THE WORKING DISTANCE CAN RESULT IN DAMAGE TO THE SEM!

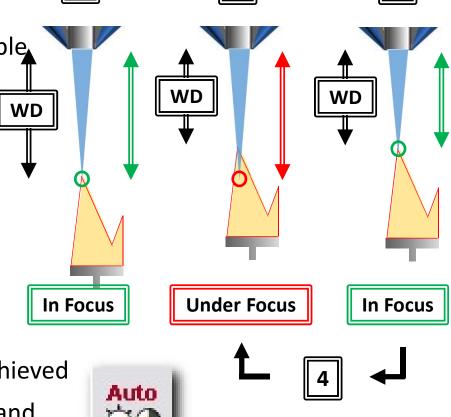


2. Raise the specimen stage by SLOWLY turning the Z-knob counter-clockwise





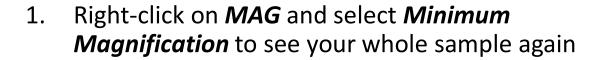
- 4. Repeat steps 2 3 until desired WD is achieved
- 5. Click **Auto** to auto adjust the brightness and contrast if too dark when necessary



6. SLOW DOWN WHEN YOU REACH ~ 10 mm AND DO NOT GET LESS THAN 5 mm

XII. Image Preparation – 1/2

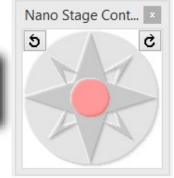
Imaging at *MAG* ≥ 10 kX requires optimization steps XIII. Column Centering and XIV. Stigmation Correction after completion of XII. Image Preparation, else skip and proceed next to XV. Image Acquisition directly



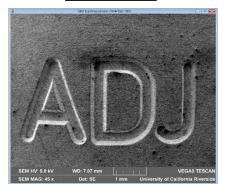
 Identify an area of interest on your sample to image by using a combination of *MAG*, *Stage Control*, focusing (*WD*), and *BI*

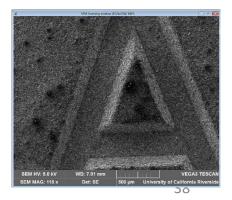






Example





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

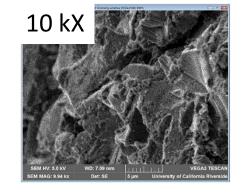


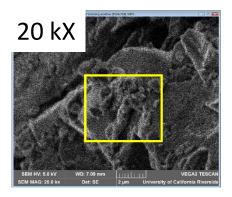






Example



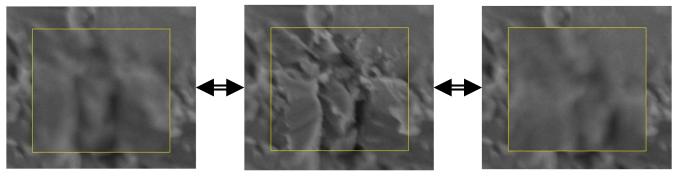


Recommended Initial **BI** values

Magnification	Beam Intensity
Min – 200	13 – 18
200 – 2000	8 – 12
2000 – 10k	7 – 10
>10k	4 – 7

XIII. Column Centering – 1/3

- 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

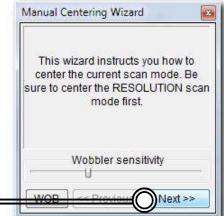




- 5. Click *Manual Column Centering* button
- 6. The Manual Centering Wizard will appear



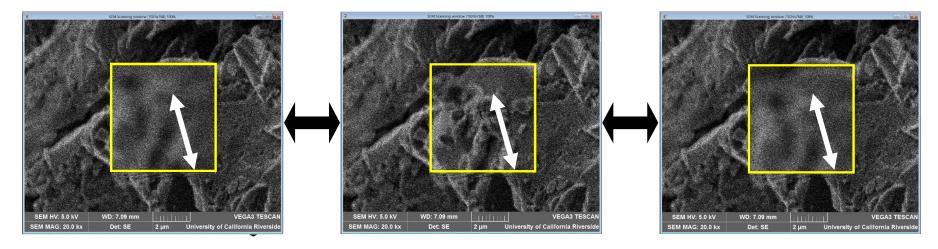




XIII. Column Centering – 2/3

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



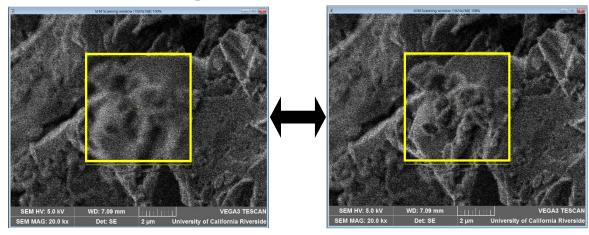
- 9. Minimize image movement by adjusting the OBJ Centering using the trackball
 - X: Hold F12 + trackball
 - Y: Hold F11 + trackball

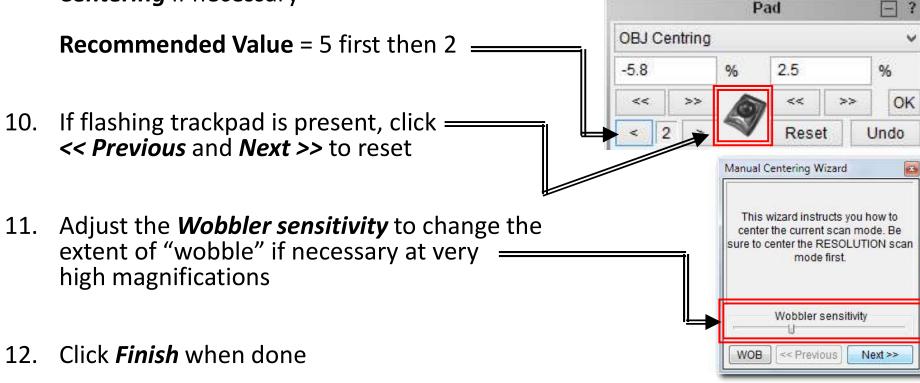


- = Change only X-movement
- = Change only Y-movement

XIII. Column Centering – 3/3

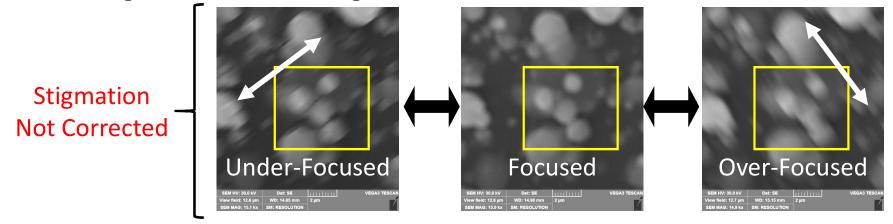
- 8. The image should remain stationary with no X or Y translation but only oscillate in/out of focus
- 9. Adjust the sensitivity to finely control the *OBJ Centering* if necessary



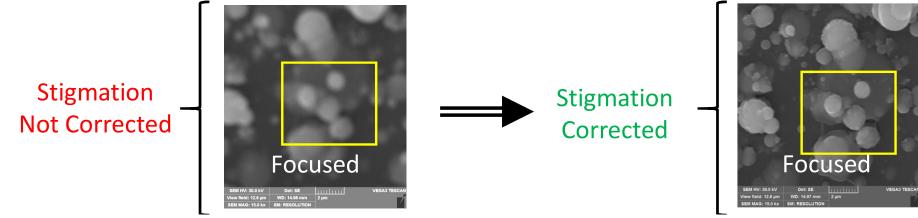


XIV. Stigmation Correction – 1/4

- 1. Create a *Focus Window* on a feature of interest
- 2. Click **WD** and bring the feature **in and out-of-focu**s (both sides) to check if any **streaking** occurs on **non-straight features**



- 3. Any streaks are evidence that *Stigmation Correction* is necessary
- 4. When Stigmation corrected, a focused image will become **significantly sharper**



XIV. Stigmation Correction – 2/4

- 5. Set **SPEED = 4** + appropriate **BI** (see table)
- 6. Click **WD** and create a **Focus Window**
- 7. Focus on a feature (**WD** Sensitivity = 2) as **BEST AS YOU CAN**





Recommended Initial **BI** values

Magnification	Beam Intensity
Min – 200	13 – 18
200 – 2000	8 – 12
2000 – 10k	7 – 10
>10k	4 – 7

- 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)

X: Hold F12 + trackball



= Change the X-component

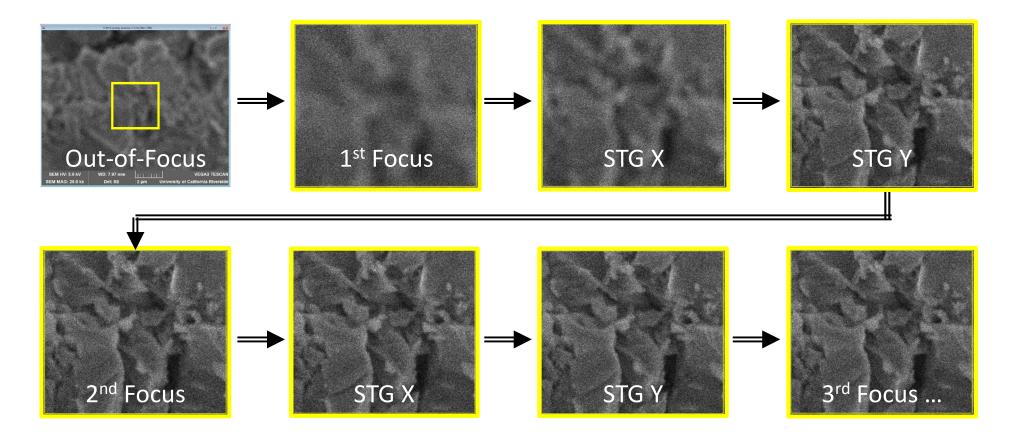
Y: Hold F11 + trackball

= 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

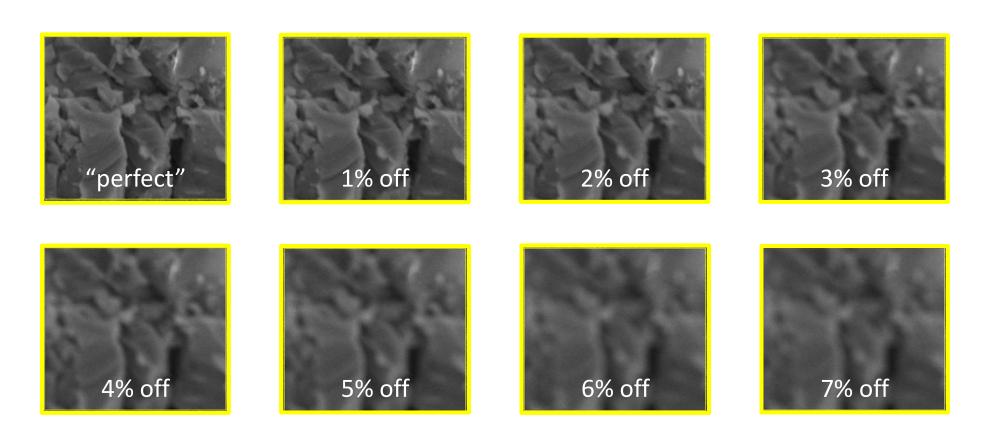
- 13. If your image still doesn't look "good", 99% it's because of poor STG Correction
- 14. The sequence of *STG Correction* should resemble the following:



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

XIV. Stigmation Correction – 3/4

- 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

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



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

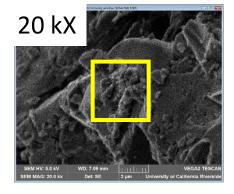
Click *MAG* and set back to desired magnification
 (e.g. Desired Mag = 10 kX)

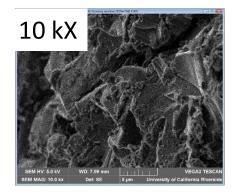


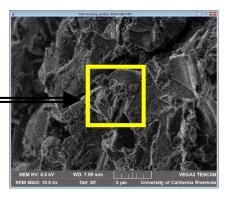
3. Activate the *Focus Window* over a desired feature

Smaller window = requires less time to refresh

Example







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*



SPEED	Acquisition Time
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

NOTE: Remove focus window first else it will only adjust pixels found within focus window + change speed back to 1 for faster auto correction

- 7. If high resolution is desired but excessive graininess is present, increase the **Acquisition Time** (e.g. **SPEED** = 7 -> 8)
- 8. Repeat Steps 5 6 until desired balance between resolution and graininess and is achieved (e.g. see next slide for examples)

XV. Image Acquisition -3/3

Low Resolution **High Resolution Low Graininess High Graininess** = 8 **==== BI** ==== 6 == 10 ======= = 4 === === Speed 8

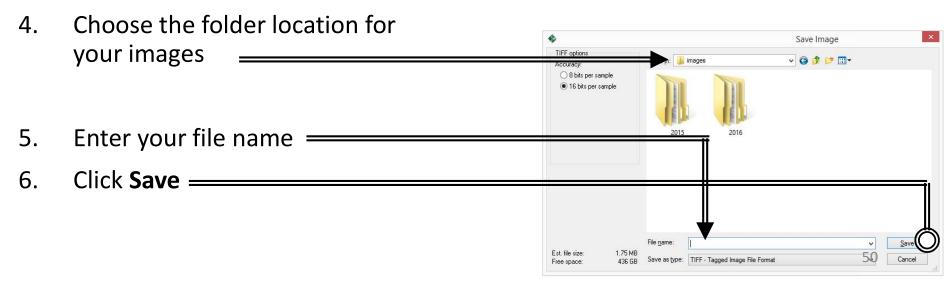
XVI. Saving -1/1

1. Click *Acquire* to capture image

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**



Header of Save Window

VEGA3 SBH

115-0069

0.0 / 0.0 µm

000

000

37.51 pA

MSE161

Description

Project:

Scan Speed:

Image Parameters
Date: 2

2016-11-18

17:00:37

5.00 kV

15.00

0.0 / 0.0 %

00/00%

3.3 / 30.0 %

100 ns

Device:

Serial No:

Rotation:

BI Fine:

Absorb. Curr.

Gain/Black

Detector

Tilt

Add

10 14 x

1 frame

101.9 µA

4 3e-002 Pa

26.671 µm

Source Mag:

Accumulation

Stage (x,y,z):

Stage (r.t):

0.0, 0.0 deg

0.00, 0.00, 0.00 mm

Pixel Size:

Delete

XVII. Sample Unloading – 1/3

1. Right-click on **MAG** and select **Minimum Magnification**



Auto Signal
Auto Speed
Auto WD
Auto Stigmation
Auto BI OptiMag

Minimum Magnification
Fullscreen

2. Click **SPEED** and select **SPEED 1**



3. Carefully lower the sample stage to the lowest position by turning the Z-knob clockwise





4. Set *BI* to **15**



5. Click **Auto**



XVII. Sample Unloading – 2/3

WD 6. Set **WD** to 30 mm WD 30.00 0.000 mm mm OK 2 Reset Undo 7. Click **HV** to turn off the high voltage **Electron Beam** 21 µA 30.00 kV Emission: Live Time: 36 h Heating: 3.558 A 8. Click **VENT** to vent the microscope = HV Heat 30.00 (20...30) kV Adjustment >>> Click **Yes** to confirm venting 9. Vacuum Vent the chamber? 1.0e+005 Pa Column pressure: VENT PUMP No Venting finished. 10. Wait until "Venting finished" appears 11. Set the tilt of the specimen stage back to 0° if not already set to 0° (Advanced Users only)

XVII. Sample Unloading – 3/3

Gently pull the chamber corners toward you to open the chamber 13. Loosen the screw holding the TURN CLOCKWISE = LOWER STAGE specimen stub on the specimen stage 14. Rotate the stage if necessary to access screw port = Nano Stage Cont... Using the provided tweezers, carefully remove the specimen stub out of the specimen stage

XVIII. Cleanup – 1/1

- 1. Ensure that the sample stage is at the lowest position by full clockwise turns
- 2. Carefully close the chamber door by pushing it towards the chamber
- 3. Place finger against chamber door =
- 4. Click **PUMP** to start pumping down schamber
- 5. Wait until bar graph shows red to release finger
- 6. Wait until the bar graph turns green ____ or "Vacuum ready" appears (~ 3 min) =
- 7. Open the **File** menu and select **Logoff**, click **Yes**
- 8. Record your total time used in the Sign-in sheet

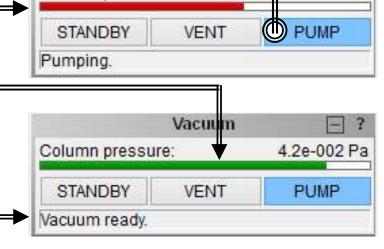


Column pressure:





2.9e+000 Pa



Vacuum