Standard Operation Procedures (SOP) for MAIA3 SEM

(This is a simplified version that focuses mainly on routine operation. For advanced operations, please first consult Prof. Jinyao Tang, <u>jinyao@hku.hk</u>)

This piece of instrument is delicate and expensive, please handle it with extra care. Do NOT use it if you are not trained.

1. Scope

1.1 This document provides the basic SOP and requirements to MAIA3 SEM.

1.2 Booking System: <u>http://store.chemistry.hku.hk:8085/</u>. You must book the instrument before using it. Your right of access will be suspended if you are caught not doing so.

Location: CYM LG 216

Primary Contact: Ms. Crystal CHEUNG. Email: sfcheung@hku.hk

Please immediately report any problems you find when operating the instrument.

2. Boot Order and Sample Loading

2.1 Check the Nitrogen cylinder and make sure it is enough during the experiment. If there is no Nitrogen anymore, please contact Ms. Crystal to change a new cylinder.



2.2 Click on the "**STANDBY**" button in the Vacuum panel to initialize the microscope, then "**VENT**" the microscope until the pressure is at atmospheric level. (Before venting, make sure the stage is in "home" position.)

BEAM ON	1	5.00 (410) kV 👘 🗸						
Adjustment >>>								
	uum	E	?					
Gun Pressure:		< 4.0e-00	8 Pa					
Column Press		2.6e+00	1 Pa					
Chamber Pres		2.6e+00	1 Pa					
Vacuum off. Standby mode!								
STANDBY	VENT		PUMF	>				

2.3 Open the door gently. Left click the mouse on the position carousel to select the sample position, loose the screw to place your specimens, and then tighten the

screw to hold the sample (Wear gloves when you load or unload your samples. Do not touch any of the stage parts if it is moving)



2.4 Close the door gently and click "PUMP" button in the Vacuum panel. Press the door during pumping until no gap between the door and the chamber. Before continuing or going, please check three pressure turning to green.



- 3. Focusing and Image Collection
- 3.1 Set the SEM Image Parameters from the menu SEM→Image Parameters.

EM Image Pai	rameters				?	×	
Windows							
Live:	~	512 x 5	\sim				
Save:		512 x 5	12	\sim			
Averaging					-		
					Ena	ble	
Live:	Live: Frame accumulation ~				10	-	
Save:	Frame accumulation ~			10	-		
Acquisition							
	Acquis	ition	time: 0.	05 s			
Keep actua	al speed					\sim	
Keep view	field / print	magi	nificatio	n		\sim	
Infobar texts	a Iobar						
Beam energy \sim Working distance				\sim			
View field		~	Detec	lor		\sim	
Magnificati	on	~	Date ~				
Burn Note & Sign texts into image							
					Ар	ply	

3.2 Select proper "**HV**" (e.g. generally 5-10 kV, 2kV for glass sample, 1kV or even lower for MOF or other polymer without electric conduction.) and click "**BEAM ON**" to turn on the electron beam. In the SEM Detectors & Mixer panel select the appropriate detector (Generally SE. In beam detector is for UH mode with 5-6mm WD&Z). Then select scan mode (Generally **ANALYSIS** or **OVERVIEW**. Please note

5 C Rot -0.00 deg	Home	SEM Detectors & Mixer 📃 ?	Info Panel	- ?
-) -) Tilt -0.00 • deg	Calibrate			
WD & Z 93.357 + mm	OK <<	Channel A Channel B	Continual Single	Acquire
Use UV coordinates	Define UV	SE V In-Beam BSE V	g ··· ·	
MEM CLR A B C D E F	GHIJ	31.8 % / 91.3 % 54.4 % / 95.5 % Channel C Channel D	UH RESOLUTION	1
Standard Tescan Carousel		SE 🗸 SE 🗸	N DEPTH	
		31.8 % / 91.3 % 31.8 % / 91.3 %	V	1
5 4		100 % A + B, A - B 0 %	OVERVIEW	NVI)
		Electron Beam - ?		5,1)
		Emission: 101 µA HV: 5.00 kV		1
6 4	3	BEAM ON 5.00 (410) kV ~	UHR CrossFree	1
		Adjustm 0.20 (0.20.7) kV	Sugmator0.1 /07 -0.	5%
		Vact 2.00 (1.52.5) kV	Image Shift: 0.0 µm / 0.0	0 µm
		Gun Pressure: 3.05 (2.54.0) kV 5.00 (410) kV	Rotation: 0.00 deg	
		Column Pressure: 15.00 (1030) kV	Deem latereite 40.00	
		Chamber Pressure: < 1.0e-002 Pa	Beam Intensity. 10.00	
		Vacuum ready.	Absorb. Curr: 136 pA	
		STANDBY VENT PUMP	Spot Size: 30.0 nm	

that when your samples include magnetic materials, do not use UH mode.).

3.3 Click the position carousel (1-7) to select aimed specimen, move the sample with the joystick (X Y Stage Movement) in the control panel. Adjust the Magnification, Focus, Contrast, Brightness in the control panel as required. Some easy buttons are set at the right hand of the scanning window for automatic adjustment.



3.4 After focusing the sample well, adjust the **WD&Z** in the "**Stage Control**" panel gradually (during that process you should watch the "Chamber View" at any time and place the mouse pointer at the **Stop** button, when the stage seems to collide with the detector, click stop. Generally 5-10mm for SE, 12mm for BSE detector, 5mm for in beam detector, and in beam BSE detector is complementary to the retractable (in-chamber) BSE detector. If you use EDX software "AZtec", you should select 10mm WD&Z. Please note that "Chamber View" panel should not be closed at any time). You can rotate the stage by writing in the value of "Rot" in the **Stage Control** panel by clicking on the " **⊠Keep view field**". (Please note that "Rotation" button in "Info Panel" just rotates the image rather than the stage.)



Warning on moving stage:

- a. EVERYTIME before moving WD&Z, you have to refocus on sample and make sure the sample is at the focus. This tell the system, where your sample really is. Very Important!! Otherwise, you will crash the objective and the system is permanently damaged.
- b. Raising stage have to be down in several steps, do not move to 10mm or lower directly. Otherwise, you will crash the sample and the system is permanently damaged.
- c. When change samples, you have to lower the stage before clicking on different location on the stage control, because the height of sample could be very different. The WD&Z for one sample could be 10mm but for next one could be already 0, it is very dangerous to change directly. The system may crash the objective and permanently damage itself.
- 3.5 Click the button and then select "next" to adjust the wobbling. At this time,

you can see "**OBJ centering**" in the control panel. Adjust X and Y bulb to minimize the large movement of the swaying image, then "**finish**" the wobbling regulation.



- 3.6 Click the button it to adjust the stigmation. At this time, you can see "Stigmator" in the control panel. Adjust X and Y bulb together with the focus to reduce the astigmatism. Repeat 3.4-3.6 until you get a clear scanning view.
- 3.7 Choose proper scanning speed to get a clear live image by mouse middle wheel or the easy button 🔜, click 🧟 to capture and save your image. Your data should be

saved in **Z drive**. (the software "**AZtec**" On the middle computer is for EDX test.)



Note that you should only insert your USB drive in the computer shown below with

the label of "SEM Data"

, and get your data in the SEM Data file:





4. Shutdown

4.1 Click "BEAM ON" to turn off the electron beam. Vent the chamber, take out your samples and remove all the tapes. Pump the chamber until all the three pressure progress bars turning to green, click "STANDBY" to go into the standy mode and then you can leave. Do not turn off the software. If you turn off the software carelessly, you can log in with guide mode without password.

Please refer to the follwing link for more detailed introduction and

manipulation.

- 1. PDF of MAIA3 instructions.
- 2. Training video by Dr. Jinyao TANG.
- 3. EDX training---teacher viewing angle.

EDX training---computer viewing angle.