

Standard Operating Procedure

| | |
|-------------|----------------------------|
| Title: | Running TESCAN MIRA3 FESEM |
| Issue Date: | 1-May-2015 |
| SOP# | SOP-EMC-YANG-030 |
| Revision # | 0 |

This is a controlled document, authored and maintained under the Faculty of Science. All information contained in this document is the property of Saint Mary's University Faculty of Science and shall not be made available to the public in any format or distributed outside the institution without prior approval.

Author of this Revision:

Date:

First Name, Last Name, Title (i.e Ph.D)

Role (ie. Professor, Department of Biology)

Reviewed by:

Date:

First Name, Last Name, Title

Role

Approved by:

Date:

First Name, Last Name, Title

Role

Authorized by:

Date:

First Name, Last Name, Title

Role

Standard Operating Procedure

1.0 Purpose:

The aim of this guideline is to inform all personnel who use the TESCAN MIRA3 FE-SEM about the proper procedures, safety concerns and to maximize the degree of efficiency.

2.0 Scope and Applicability:

| | |
|---|---|
| The purpose of TESCAN MIRA3 FE-SEM is to analyze samples and specimens in high magnitude. This document applies to any personnel who will be using the TESCAN MIRA3 FE-SEM. | |
| | Department, Lab or Center: Electron Microscopy Centre |
| | Research Group: |
| | Lab Bldg., Room(s): Science Building, Suite 001C |
| | Operation/Experiment: |

3.0 Responsibilities:

The user shall perform the following procedure within the EM Centre (S001C) regarding the use of the TESCAN MIRA3 FESEM. Only trained personnel should operate this equipment.

4.0 Health, Safety and Environmental Considerations:

4.1 Materials and Hazards

| Principal Materials Used | Flammable | Corrosive | Sensitizer | Mutagen | Teratogen | Biological Toxin | Acutely Toxic | Pyrophoric | Water-Reactive | Shock Sensitive | Carcinogen | Unstable | Other Comments |
|--------------------------|-----------|-----------|------------|--|-----------|------------------|---------------|------------|----------------|-----------------|------------|----------|----------------|
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| MSDS attached | Yes | | | If not, please explain: <u>Not Applicable</u> | | | | | | | | | |
| | No | | | | | | | | | | | | |

Standard Operating Procedure

Describe equipment/instrumentation used to monitor/control hazards:

| | |
|--|--|
| | Permits: |
| | Mgmt. Approval: |
| | Training: |
| | Medical Surveillance: |
| | Other: <ol style="list-style-type: none"> 1. IN AN EMERGENCY, LEAVE THE SCENE AS SOON AS POSSIBLE. 2. The EHT voltages present in this instrument can be lethal. 3. Do not use electrical equipment in: <ul style="list-style-type: none"> • Rain or excessive moisture environment • The presence of flammable or explosive gases The equipment is not designed to be water or splash proof, or to be used in area where there are flammable or explosive gases or fumes. |

4.3 Special Emergency Procedures

| | |
|---------------------------|--|
| Fire/Evacuation: | |
| Chemical Spill: | |
| Medical Emergency: | Call security office at 420-5000 immediately, or any of the nearby first aiders. The names, room# and phone# of the nearby first aiders are posted in the lab. |
| Personal Exposure: | |

5.0 Equipment and Supplies:

| | |
|----------------------|--------------------|
| Equipment(s): | |
| X | TESCAN MIRA3 FESEM |

Special PPE Required:

Standard Operating Procedure

| | | |
|--|---|--|
| | | Goggles |
| | | Face Shield |
| | | Chemical Resistant Apron |
| | | Protective Clothing |
| X | | Gloves |
| | | Butyl |
| | | Nitrile |
| | | PVC |
| | | Latex |
| | | Neoprene |
| | | Silver Shield brand |
| | | Kevlar |
| | X | Other: Powder free gloves, lint free sleeves |
| | Respirator (If yes, contact EHS Office for additional assistance) | |
| <p>Note: If special PPE and/or protective clothing is not required, standard PPE and protective clothing required in Part II of the Department Chemical Hygiene Plan must be utilized.</p> | | |

6.0 Terms and Definitions:

Not Applicable

7.0 Procedure:

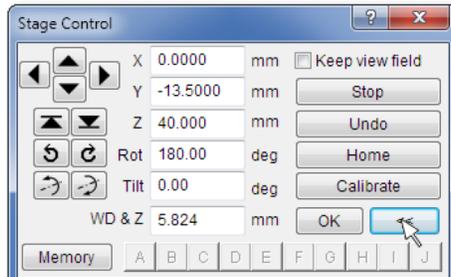
Basic Instructions

1. Logon MiraTC software by using the username you are assigned to. If the software is not open, double clicking  icon on the desktop to start the software.

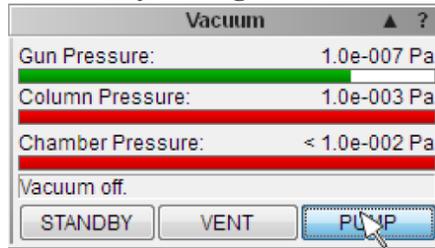


Standard Operating Procedure

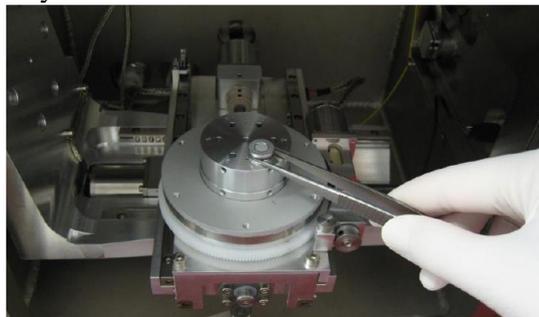
- Click *Home* button on the *Stage Control* panel to set the stage to its default position. This will avoid the specimens in the chamber from crashing into pole piece, detectors or chamber wall.



- Click *Vent* button on the *Vacuum* panel to start venting the specimen chamber and allow you to open the sample chamber door. It will take approximately 3 minutes for the system to reach atmosphere level. **Never pull the door venting period – the EDS detector has a fragile and expensive window; a sudden pressure increase in the chamber pressure would likely damage the EDS window.**



- Load your specimen and standards (please wear powder free gloves). Gently tighten the screw to hold specimen stubs on the stage. **Never reach into the chamber without gloves on!** Always use the sample exchange tool (tweezers and special designed screw-driver) to load/unload your samples. Mark down the position ID you've just loaded. You will find this to be very useful after the chamber door is closed. Ask for help if you are not sure what to do.



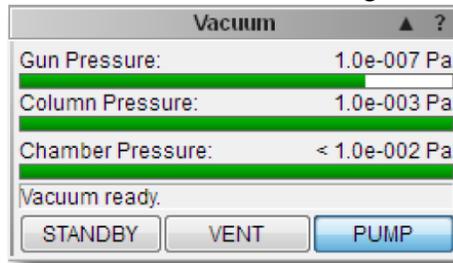
WARNING:

- Any bare hand operation during the sample loading/unloading will result in the loss of privilege of using the system.
- If the stage is moving, do not touch any of its parts. Fail to follow the safety procedure may result in physical injury and the loss of privilege of using the system.

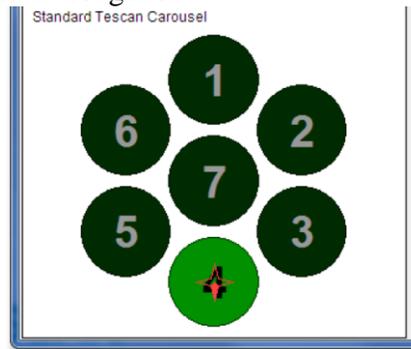
- Close the chamber door and click *Pump* button on *Vacuum* panel to start pumping down the chamber. It would take approximate 2 minutes to reach the workable

Standard Operating Procedure

vacuum. Wait till all the vacuum indication bars turn green.

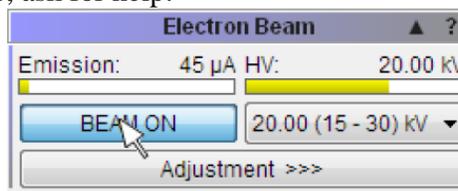


6. Move the first sample to the viewing position by clicking the corresponding number on the stage carousel. The star sign on the stage map indicates the sample position of the stage is currently under investigation.

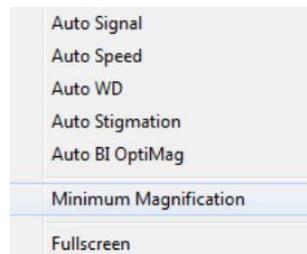


Hint: If you have samples with different height, it is always a good idea to observe the tallest sample first and keep the stage remaining at the same height for the rest of the samples. This will avoid potential collision of your specimen with the SEM chamber components.

7. Turn on the beam by clicking *BEAM ON* button on the *Electron Beam* panel. The voltage suitable for your sample type should have been pre-set by lab personnel based on the information you provided. Double check if the accelerating voltage is good for your sample. If not sure, ask for help.

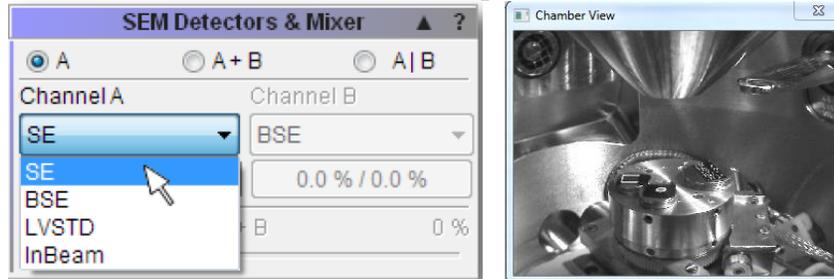


Hint: Always start from lowest magnification possible. This can be achieved easily by right-clicking in the viewing window to open menu and select the *minimum magnification*.



Standard Operating Procedure

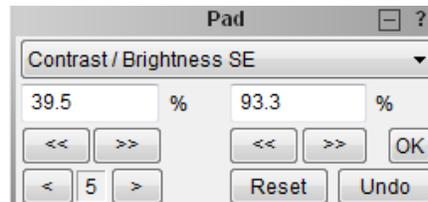
8. The default detector has been set to collect secondary electrons. You can switch to BSE detector if necessary (make sure the BSE detector is in place) by clicking the *Channel A* box on the *SEM Detectors & Mixer* panel to select BSE detector.



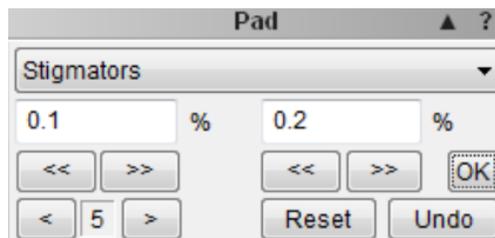
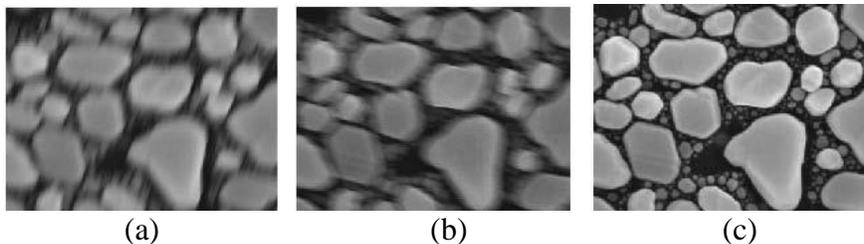
9. You can now work on obtaining a sharp image by adjusting focus (working distance), stigmators, image brightness and contrast controls.

- Click on the *WD (Focus)* icon  on the *Toolbar* (to the right of the viewing window) and turning the Trackball from left to right (or vice versa) to focus the image.

- Click on the *Contrast/Brightness* icon  on the *Toolbar* and turning the Trackball from left to right (or vice versa) to set the contrast, up to down (or vice versa) to set the brightness.



- Click on the *Stigmator* icon  on the toolbar and turning the trackball. The following pictures (a & b) show the existence of astigmatism and picture c is corrected.

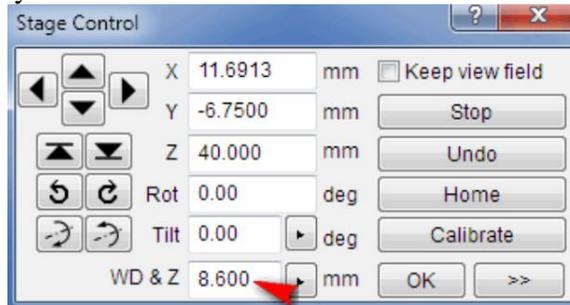


Hint: For higher magnifications (>10 kx) viewing, it is necessary to check if astigmatism is corrected.

Standard Operating Procedure

- Click on the *Degausses* button  or simply type *D* on the keyboard couple times to degauss the column of SEM. The degausses function removes residual magnetism from the column and thus ensures that displayed magnification and working distance is correct and calibrated.

10. Set the working distance to desired value by using the *WD&Z* function on the Stage Control panel, the stage will adjust its height accordingly. Refocus by using the trackball if necessary.

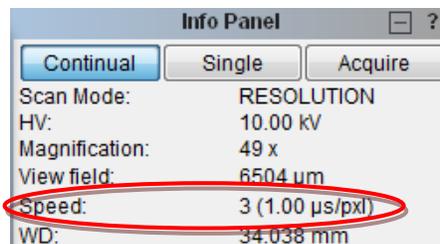


Caution: Be extra careful if you plan to work on a WD less than 10mm, especially with samples of different height, you may potentially damage you sample, detectors or other chamber components if you sample crash onto the pole piece or detectors.

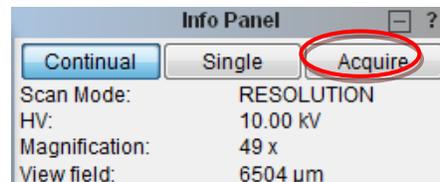
11. Readjust the brightness and contrast if necessary.

Hint: If you find it is difficult to adjust both B&C at the same time, you can adjust B/C one at a time: press F11 + trackball to adjust brightness only, F12 + trackball to adjust contrast.

12. Set the proper scan speed and save the desired image. A scan speed of 5-7 is recommended to obtain a good quality image. You can turn the mouse wheel to adjust the scan speed.

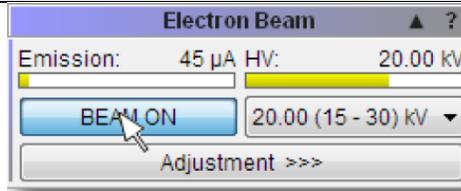


13. To save an image, click on the *Acquire* icon  on the toolbar or the *Acquire* button on the Info Panel.

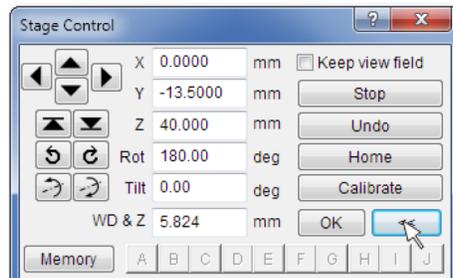


14. When done with collecting data, turn off the beam by clicking the *BEAM ON* button. The color of the button should now turn to grey.

Standard Operating Procedure



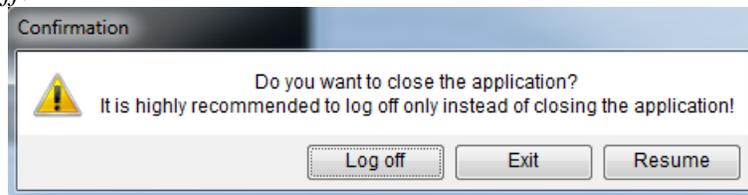
15. Click *Home* button from the stage control panel to reset the stage back to its default position.



16. Remove specimen from chamber, close the door and pump the chamber.

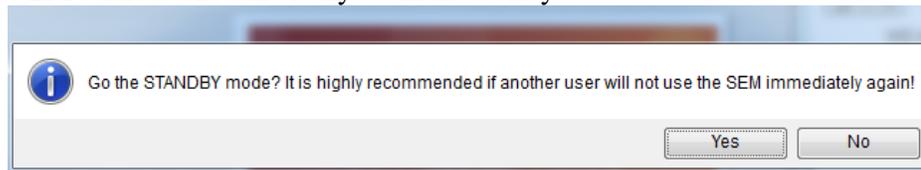
WARNING: Fail to pump the specimen chamber after you retrieve your samples will result in the loss of your privilege of using the system.

17. Close the MiraTC window to log off the software. In the *Confirmation* window, click *Log Off*.



WARNING: If you forget to log off the MiraTC software, you may be charged for the SEM use if someone else is using the SEM with your account. You will also be responsible for any SEM damages caused by other people using your account.

18. Click *No* on the following window. This will ensure the next user is able to use the SEM without encounter any beam instability.



WARNING: Only the lab personnel has the right to decide when to set the SEM to STANDBY mode. If you have accidentally clicked the *Yes*, you are responsible to inform lab personnel immediately. Fail to do so will result in the loss of your privilege of using the system.

Standard Operating Procedure

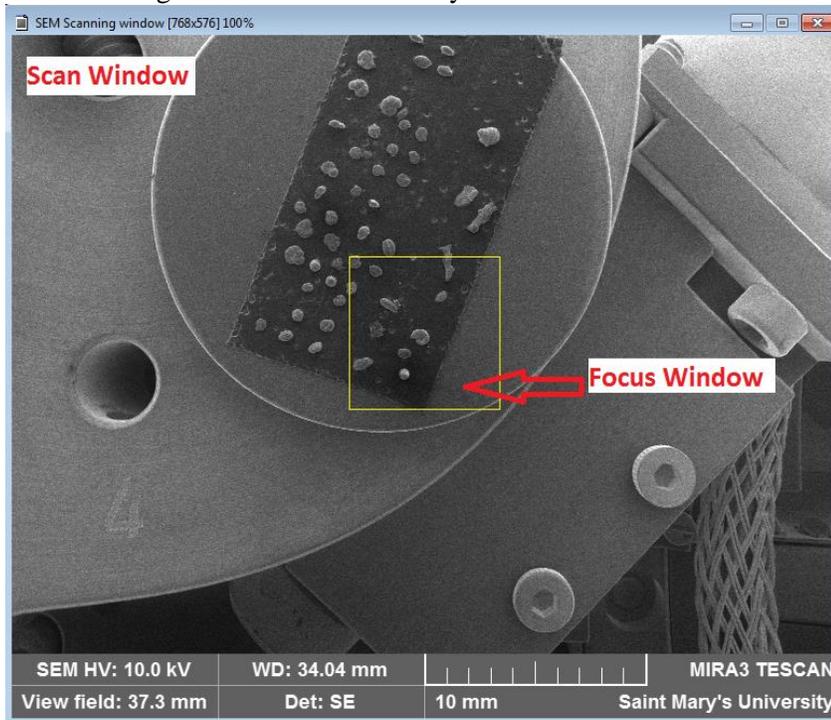
19. Fill the log book.

WARNING: Fail to fill the log book will result in the loss of your privilege of using the system.

Appendix:

Mouse functions within Scan Window

- Turning the mouse wheel changes the *Scan Speed*.
- Clicking the mouse wheel on a selected object in the scanning window moves the stage so that the object lies in the centre of the scanning window.
- Holding down the mouse wheel on a selected object and dragging the cursor to any position in the scanning window moves the object to the selected position in the scanning window.
- Holding down the mouse wheel on a selected object in the scanning window for longer than 0.8 seconds moves the stage so that the object lies in the centre of the scanning window and the magnification is increased by the factor set in the menu SEM-> Options.



Mouse functions within Focus Window

- Double click with the left mouse button on the scanning window switches the Focus Window on and off.
- Holding down the right mouse button and dragging the mouse cursor in the scanning window changes the dimensions of the Focus Window.
- Holding down the left mouse button and dragging the mouse cursor in the scanning

Standard Operating Procedure

window moves the Focus Window.

- Double clicking of the **right mouse button** on the Focus Window moves this window to the centre of the image.

| Task | Hazards | Precautions |
|------|---------|-------------|
| | | |

8.0 References:

1. Mira3 FEG-SEM Instruction for use, TESCAN, Brno, Czech Republic, 2011
2. MiraTC Help Manual, TESCAN, Brno, Czech Republic, 2011

9.0 Applicable regulations and/or legislation:

10.0 Revision History:

| Rev # | Revision Date | Review Date | SOP Section(s) | Revision Description | Revised By |
|-------|---------------|-------------|----------------|--------------------------|------------|
| 0 | May 1, 2015 | | | SOP-EMC-YANG-030 created | Xiang Yang |
| | | | | - | |
| | | | | - | |