ZEISS SUPRA-55 VP FIELD EMISSION SCANNING MICROSCOPE (LV FE-SEM)

Short form user procedure

USER NOTES:

- Must be certified to use instrument by laboratory staff. User form must be completed and signed by staff and user.
- Please use the sample height gauge for sample loading for EBSD using. If a sample breaks or becomes stuck in the specimen chamber, notify staff. DO NOT ATTEMPT TO REMOVE.
- If the red OFF light or the yellow STANDBY light is illuminated, contact staff. DO NOT ATTEMPT TO START THE INSTRUMENT.
- **ALWAYS TURN ON THE CHAMBER CAMERA BEFORE ATTEMPTING TO RAISE THE STAGE.** Failure to observe the interior of the specimen chamber may result in damage to the SEM and the sample. Never adjust x, y, rotate, or tilt if there is a chance that the sample may be touching the lens cap or detectors inside the chamber.
- If you need more information, please contact staff at lab.

- Primary contacts is Yingda Yu, (yingda.yu@material.ntnu.no) and Tor Nilsen, (tor.nilsen@material.ntnu.no)
- **Users that not following rule may be excluded from use of electron microscopes.**
- **The Zeiss PC should be restarted twice per week to keep the memory working correctly.**
- **Before Sample Loading, the TV camera should be turned on first for internal reference.**

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SAMPLE LOADING:

1. Check the FE-SEM vacuum/electronics status panel. If GREEN, OK to proceed. If YELLOW or RED, contact us.

2. Log into the SEM-PC fill in the log with your personal and project information. (both as "SEM")

3. Click on the Smart-SEM icon on the bottom of the left hand LCD. Enter user name "Guest" and no password and activate the SEM interface.

4. Choose sample holder and mount sample.

5. Press the CAMERA button on the keyboard. The interior of the specimen chamber will appear.

6. Press the EXCHANGE button on the keyboard.

7. Wait for the chamber to reach atmosphere pressure, and pull out the stage. Insert the specimen holder.

8. Press the EXCHANGE button on the keyboard.

9. Wait for the VAC: marker in lower part of smart SEM window to become a green mark. Also in the SEM ctrl window it's possible to see column pumping status. When it's ready, continue.

OPERATION:

1. If chamber camera is off, turn it ON. Ctrl-G brings up the SEM control panel.

2. Using the left hand joystick, carefully raise the stage Z position until useful position. Keep one eye on the specimen chamber with the camera.

3. Click on **EHT** on the bottom of the left hand LCD and click **EHT ON**. (Use the **GUN** tab on the SEM control panel to change the accelerating voltage.) The screen should brighten, but the image will probably be out of focus.

4. Ctrl-D brings up the "data zone" at the bottom of the image if it's not on. This will be part of your saved images when it is ON. Ctrl-D toggles this feature off and on.

5. This section is normally not used: If necessary to change Aperture: Select the **APERTURE** tab on the SEM control panel to choose an aperture. The 30 um aperture is a general purpose aperture and a good place to start. Seven apertures are available ranging in size from 7.5 um to 120 um. You may change the aperture at any time, but adjustments for astigmatism and aperture centring may be necessary to achieve an optimum image.

6. Select the **DETECTOR** tab on the SEM control and choose the secondary detector.

7. Toggle the coarse/fine bar to coarse with the mouse and focus the sample. Increase magnification, toggle to fine, and focus again.

8. Check the working distance on the data zone. To adjust, turn the camera on and raise or lower the stage with the joystick, avoiding any unintentional tilt. Then focus again. Repeat until desired working distance is achieved.

9. The right hand joystick controls lateral motion (x/y) and rotation (twist). All stage motion may also be controlled from within the **STAGE** tab of the SEM control panel. Find the desired features and manipulate the stage to position the sample.

10. The **SECONDARY** detector is usually satisfactory for moderate to long working distances and the entire range of accelerating voltages.

11. For superior SE image quality at low accelerating voltages (3 kV or lower) and short working distances (2 to 5 mm), the **IN-LENS** detector is generally preferred. The **IN-LENS** detector may be used UP TO 20 kV, but image quality may degrade as working distance increases. Do not use the **IN-LENS** detector above 20 kV; use the **SECONDARY detector** instead.

12. **QBSD** is a 15 mm 4-quadrant backscatter detector.

13. The **IN-LENS** and **SECONDARY** detectors are the most commonly used.

14. With the chosen detector and appropriate aperture in place, the accelerating voltage selected, and the feature of interest on the screen, adjust contrast and brightness. Normally brightness will be around 49%

15. If there is a large amount of astigmatism present, perform a preliminary correction with the x/y astigmatism controls on the keyboard: Centre the aperture by pressing the **WOBBLE** button on the keyboard. You may adjust the amplitude in the **APERTURE** tab of the SEM control panel. Focus and correct again for astigmatism, using either the keyboard or mouse controls.

16. Shift-F2 activates lens clear. Use this if you are unable to correct the astigmatism or have an otherwise unsatisfactory image. If there is hysteresis in the lens, the image will shift and go out of focus. Focus again. Repeat lens clear/focus two or three times if necessary until you can obtain a satisfactory image. If there is still a problem, contact staff.

17. To change HT: double click EHT in data zone or select Gun in SEM control. Type in new setting.

SAVE IMAGE:

1. When the image is optimized, you may choose to save it. Within the SEM control panel, you can choose from several types of scans (line/frame average, line/frame integration, pixel average) and speeds.

2. When you are ready to save an image, click on **FREEZE** in the SEM control panel. On the left hand LCD; click on **FILE** and **SAVE IMAGE.** Click on **CHANGE DIRECTORY** then choose drive D and open your image folder. Create a sub-folder if you wish, type in a file name and press **SAVE** or **ENTER.** In the SEM control panel, click on **UNFREEZE**, then return to **PIXEL AVERAGE** and fast speed (perhaps 3) to return to live image. Next image can be saved by clicking at TIFF button in upper right corner in the left hand LCD.

3. All of images should be saved into User Folder with your name as sub-folder name.

4. **Please observe that it will not be taken any backup of image files. Images older then 1 month may be deleted without any notice. If problem with PC, the complete system may be reinstalled and all image files deleted.**

EXCHANGE / UNLOAD SAMPLE:

1. Click on **EHT** on the bottom of the left hand LCD and click **EHT OFF**.
2. Press the **CAMERA** button on the keyboard. The interior of the specimen chamber will appear.
3. Press the **EXCHANGE** button on the keyboard.
4. Wait for the chamber to reach atmosphere pressure.
5. Take out sample, close the door.
6. Press the **EXCHANGE** button on the keyboard (or other that starts pumping of chamber).
7. Log out of Smart SEM to disable the SEM interface and make it available for the next user.
8. Log out the SEM PC, a floating window with summary of your using time etc will appear.
9. Click OK, and then turn off the SEM-PC screen.
10. Take your stuff with you, any left articles may be considered as garbage.