Operation of FEI SIRION SEM

Start Up:
  (1) Start "Microscope control" and "XL Docu" software (from shortcuts on desktop). Click cancel to the question of selecting database when starting the XL Docu.
  (2) Record the vacuum and emitter parameters on the log sheet:
        (a) Chamber Vacuum OK ($\sim 10^{-6}$ mBar),
        (b) Upper IGP better than 5×10^{-9} mBar,
        (c) Lower IGP in the order of 10^{-7} mBar.
        (d) These three can be found in the enlarged vacuum page.
        (e) Operate:
        (f) The value can be found in the enlarged beam page.
  (3) Vent the specimen chamber by pressing the "Vent" button in the control program. You may wish to activate the CCD detector at this time (via the software). When the chamber has reached ambient pressure the door will open freely.
  (4) Mount the specimen in the specimen holder. Be gentle with the screw.
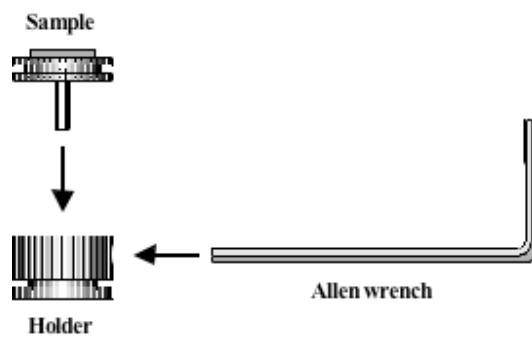
Adjust the height of the sample until it is no closer than 5 mm from the objective lens. Use the scale as shown below.
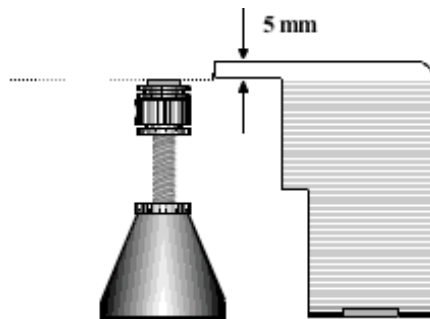
ALWAYS check that the specimen height is low enough to avoid touching the pole piece of objective lens.
  (5) Close the door and hold it with your hand. Press the pump button to evacuate the chamber. Wait until the vacuum reaches 5×10^{-5} mBar.

(6) Switch to SE or TLD detector from CCD. Click High Tension (kV) Button to activate beam acceleration voltage. If the beam is blanked, unblank the beam.

(7) You may (May not) get an error message that saying "for safety reason, it is necessary to focus the image…" Leave this image there and perform the following steps:

(8)

    (a) In the very beginning, you must focus the image at a mag. higher than 2k. Then link the working distance to stage Z value by clicking "ok" to the above message or click the Z<>WD button. Whenever you want to move the specimen in Z direction, you should link the working distance to the stage Z value again.

    (b) Be aware of the morphology of your specimen surface and *make sure that your specimen does not touch the pole piece of objective lens: you may look at the lowest point of your specimen which is far enough from the pole piece while the other points are high and may touch the pole piece.*

    (c) Operating at a working distance of 5 mm is strongly suggested*. Operating at a working distance closer than 1 mm is prohibited.*

Shut Down:

(1) Turn off the beam by clicking the high tension button. Wait (about 30s) until you hear the closing sounds of the column valve.

(2) Vent the chamber.

(3) Remove your specimen.

(4) Pump the chamber, and wait until the chamber vacuum is OK. The chamber must ALWAYS be left in vacuum state after each use of the SEM.

(5) Close the software programs.

Hints for Operation:

(1) In general, 10 kV, spot size 3, lowest mag., TV rate, SE detector are good for start.

(2) Press F2 to get a still image. Click the camera button on the "always on top manual" to transfer image from microscope control to XL Docu.

(3) For high density image, press F5 and click the HD button on the "always on top manual" to transfer image from microscope control to XL Docu.

(4) If the image moves when you focus, you need to perform column alignment. On the beam page, check the lens modulator and drag the cross of column alignment to do it.

(5) If you feel you do not have enough signal, you may want to check the gun tilt: on the beam page, check the gun tilt and drag the cross of gun tilt to get maximum intensity. The image intensity may be saturated during this process. If so, stop there. Click ACB to adjust image contrast and brightness. Then, do the gun tilt adjustment again.

(6) You have a choice between SED and TLD for electron detector. You have a choice of HR and UHR mode. UHR has some special requirements, e.g., working distance and accelerating voltage. The software will inform you if you do not fulfill this requirement.

(7) Save your data to the folder of Offline PC/SEM files/Your name. Tiff format has been tested in this Lab. While other formats are possible, there are no solid reports regarding the effectiveness of these formats.

(8) Transfer your data from the offline PC ASAP. There is nowhere to complain if you lose data. CD and Zip disk drivers are equipped on this computer.

Key Mapping:
(1) "+" key---increase mag.
(2) "-" key—decrease mag.
(3) Press the right mouse button and move the mouse to left or right for focusing.
(4) Press the shift key and right mouse button and move the mouse for adjusting astigmatism.

Key Points to Remember:
(1) Always check to make sure the sample is mounted at a height low enough to prevent touching the objective lens.
(2) Once the sample is loaded, always relink the z stage value to the working distance before attempting to move the sample in the z direction.
(3) Do NOT use samples containing magnetic material when you intend to run the SEM in UHR mode.
(4) Do NOT make any adjustments in the "Configuration Control" menu under the "In/Out" drop-down menu or the "Change" menu under the "Stage" drop-down menu.

Failure to remember these points may damage the SEM!!!

EDS operation

Part 1, General idea regarding Oxford INCA EDS system

    (1)   Log in to Oxford EDS control computer with "user name: INCA" and "Password: INCA"

    (2)   When you vent the specimen chamber, you must wait long enough to let the system vent automatically. This is not a new requirement but should be stressed again here.

    (3)   Make sure SCS software is running in the SEM side to allow the communication between EDS and microscope.

    (4)   EDS must be done when the imaging mode of microscope is EDX mode.

    (5)   Working distance should be 5 mm.

    (6)   The data is saved in a project. Project acts like a basket where everything you input and/or do during the experiment is saved.

    (7)   Oxford computer is not hooked up to the Internet. You can save you data temporarily to this computer and you are responsible to down load all of your data after your session. The Center reserves the right to delete user's data without notice. Center is not responsible to data loss by computer crash or any other reasons. Data can be removed by CD or USB memory stick.

    (8)   Stand alone "INCA" system can be installed on your computer. Ask Chunfei Li for the disk. Rough procedure: install "Flush????" on your computer if it is not installed; install INCA; create "keys" folder under "INCA"; copy key files from floppy disk to "keys" folder.

    (9)   Elemental analysis can be done in "Analyzer", "Point and ID", and "Mapping" mode.

    (10) Printer usage for EDS analysis only is allowed and is ***not*** encouraged.

Part 2, Analyzer

    (1)   Analyzer is the most basic mode. It collects whatever X-ray signal coming to the detector and do not control the microscope. The ideas for other mode, "Points and ID" and "Mapping", are very similar except some extra functions.

    (2)   Start "INCA" program if it is not activated.

    (3)   Highlight Navigator and highlight "Analyzer". There are three flow charts for each mode. The middle one is the most important one and the ones on the two sides are for parameter setting. Follow the middle one step by step first to get the idea for and come back to the two columns on left and right sides later.

    (4)   Highlight "Project" and fill in necessary parameters.

    (5)   Repeat the same procedure on "Sample".

    (6)   Highlight "Microscope setup". Choose suitable accelerating voltage, spot size on the microscope control side. Choose suitable processor time in INCA.

    (7)   Highlight "Acquire spectra". Choose suitable method for "Termination of acquisition" and spectrum conditions. Painted circle is used to start while the square is used to stop, which is true for all the other windows.

(8) "Confirm Elements" allows you to confirm the elements based on your knowledge on your specimen manually.

(9) You can do quantification by pressing "Quant" button.

(10) "Report" allows you to generate a report automatically. You can export this report to MS word by clicking "W" button.

(11) Explanations about functions of buttons on the left and right columns:

"Quant optimization" is used to monitor detector temperature and beam current change. If you are not interested in quantitative results, you can completely ignore this one. In this case, you need to press "retry" every time the computer remind you about "Quant Optimization".

 "Compare" is used to compare two or more spectra.

"Standardize" is a useless button until you are so confident and dedicated that you want to check Oxford's standard specimen.

Quant Setup" is for quantification setting up.

(12) Right click usually gives you many good options, e.g., export image in different formats.

(13) Click on the question mark give you very good hints about the meaning of buttons and the meaning of terminology.

Part 3, Point and ID

(1) The "image setup" and "Site of interest" allow you to grab an image.

(2) "Acquire Spectra" enables you to collect a spectrum from an area specified by you on the image collected at "Site of interest".

Part 4, Mapping

(1) "SmartMap Setup" and "SmartMap" allow you to setup and perform "SmartMap".

(2) "Element Setup" and "Element Maps" allow you to setup and view the elemental mapping results.

(3) For element line scan: draw a line on SmartMap; Click start; Goto element line scan and check the results.

Part 5

The style of report can be edited by simply going to Options/Edit report template.