The Philips FEG SEM Handbook

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(This section adapted from the Philips User Manual -chapter 4)

This chapter describes the basic operation of the microscope, involving several steps to achieve hard-copy results or image transfers to disk. It is assumed that the instrument has been started up and brought to a 'waiting' condition ready for inserting a specimen.

INSERTING A SPECIMEN

- Put on some **gloves**.
- **Turn off the HT**. (the high voltage). The button should be gray.

  If the HT is on (button yellow) turn it off by clicking on the button.
- **Open the chamber**. Vent the chamber with dry nitrogen.

  Click on the VENT button. You will hear the Nitrogen gas turn on, after a short delay needed to verify that the high tension is off (security HT interlock). After a couple of minutes you will be able to open the chamber. Eventually the microscope will say “idle”.
- **Load a specimen. Set Height.**

  Using gloves and/or tweezers place the sample stub into the stage specimen holder. Secure the specimen stub with an appropriate Allen key. Set the sample height to 10mm (eucentric height) with the height guide. First, loosen the cone at the bottom (counter clockwise), then adjust the upper part until the top of your sample is level with the 10mm mark on the height guide. Tighten the cone. Adjust X, Y, Z, Rotation and tilt if necessary.
- **Close the chamber door.**

  WATCH your sample does not hit anything on the way in. Be especially careful if it is tilted. Hold the door closed and click on the PUMP button. When you hear the vacuum turn on, you can let the door go.

Wait for the vacuum status message **“Vac OK”** before proceeding!

GETTING AN IMAGE OF THE SPECIMEN

Make sure you **wait** for the vacuum status message **"Vac OK"** before proceeding!

- **Choose SE detector**. (Secondary electron detector)
- **Pre -check**

  Several items should be checked so as to ensure correct operation, especially for new users of the XL microscope. The following provides a guideline to users:

kV (Accelerating Voltage)	Low kV for non conductors, High kV for conductors
Spot Size	3
Scan	TV rate
Filter	Live or Average 4
Magnification	Lowest (~24x)
Brightness, Contrast	Set to 48 and 24 respectively

- **Turn on the beam (after "Vac OK").**

  On the right side of the screen, there should be a small window with 'Beam" at the top. Inside will be a HT button that is labeled with the current voltage, e.g., "15kV". Click on this gray button to turn on the beam. An image will appear after a few seconds, possibly out of focus and with incorrect contrast and brightness settings.

- **Calibrate the stage.**

  A window will appear with the message: " Confirm the specimen is in focus and click OK to link Z height to FWD (free working distance)". **Do NOT click anything yet!!** You can move the window out of the way but **do not** hit 'ok' or 'cancel'. You can use all the other microscope controls like contrast/brightness, x/y stage motion, and focus while this window is open.

  This window is asking you to calibrate the stage z-height. After you load a sample, the stage does not know how far away the sample top is from the bottom of the pole piece. If you tell the computer to change z-height, it is VERY likely you will CRASH into the pole piece (or the BSE detector if it is installed). You must **focus** the sample **first**. The microscope 'knows' how far away the objective lens focal point is from the pole piece. When the sample surface is in focus, the focal point is on the surface of your sample. Therefore the 'WD' (in the databar) is a measurement of the true working distance.

  So move the window out of the way, find the tallest part of your sample (the bit that will crash first), and focus it at around 200x magnification or better. Then you can click on 'OK'. You should see the information from the Objective lens ('WD" in the databar) be loaded into the z-height over in the stage window. You cannot 'tell' the stage where it is in any other way! Do not attempt to type in your estimate of the working distance while the stage is not calibrated, because the stage will simply try to move there from it's present assumed position, i.e. CRASH! There is no cancel button and there is no "are you sure?" window. Be very careful when you use the computer to change z-height!

- **Correct the contrast and brightness if necessary**

  Use either the controls found at the bottom of the Beam Control group, or on the Video control group of the Control Area Imaging.

  To set Contrast and Brightness, set contrast to zero and brightness to zero. Increase brightness to the point at which the screen just turns from black to gray. Then set the contrast to approximately half the brightness value.

- **Focus the sample (right mouse button)**

  Correct the focus using the right mouse button until the image is sharp. Hold the right mouse button down and move it from side to side to find the best focus.

- **Move the specimen to the area of interest:**

  Manually with the X, Y knobs on the front of the chamber door.

  TRACK: two circles on screen, cursor is target. You set the direction and speed of stage movement with the left mouse button

  GET: cursor is a cross and you double click on a region to center it. At low mag. this is done with stage movement. At high mag. this uses beam shift

  SHIFT: only used at high magnification. Cursor is a hand and you change the region of view by dragging the beam shift with the left mouse button.

  ARROW KEYS: the arrow keys on the keyboard will also move the specimen stage.

- **Change the magnification**

  Set an appropriate magnification using either the magnification menu or using the plus and minus keys (double and halve respectively). You may need to refocus as you increase the mag.

OPTIMIZING THE IMAGE

Once the image is obtained it might need further optimization before it is stored on disk or printed.

- **Magnification and Spot size**

  Optimizing the image involves choosing a suitable area of the specimen and also the relative magnification to suit the structure observed. Spot size should be adjusted to a smaller size for high mag. and larger at low mag.. Judging which spot size is correct for a particular magnification relies on the ability to focus well and correct astigmatism easily at the chosen magnification.

- **Focusing** (right mouse button)

  Use the focus control with the mouse to correct the sharpness of the image. Focusing at 2x-3x the magnification needed for the final result will make the lower magnification sharper, e.g. photo mag = 2000x, therefore focus at mag = 4000x-8000x.

  Focus using either total screen in TV scan, or the selected area window at a slower scan rate (for a sharper image). You can change both the size and position of the window with the left mouse button. Click and hold inside the window to drag it to a new position. Click and hold outside the window to open a new window - drag to the desired size before releasing the mouse button.

- **Aperture Centering**

  If the image moves when changing focus, the aperture needs centering. Make sure you have the settings group of controls at the right of the screen. Maximize the beam window. Find the checkbox labeled “lens modulator” and tick the box. The image will appear to go in and out of focus, and will shift if the aperture is not centered. Center the aperture using the x and y knobs in turn to minimize the movement of the image. Uncheck the box to turn off the lens modulator and minimize the beam window.

- **Astigmatism Correction** (shift + right mouse button)

  Astigmatism usually needs to be corrected initially for most specimens, and then again after changing kV, spot size or working distance. The astigmatism in the image is usually only visible at higher magnifications (as a guide 3000x or greater).

  Focus as well as possible at the focus magnification. Now move the focus control through focus to the other side of focus to observe any astigmatic distortion. If astigmatism is present, the result observed is a directional distortion change of 90° between the two out of focus conditions.

  To correct the astigmatism, hold the shift key and use the right mouse button, or select the 2-dimensional Stigmator box in the Image Control Area. Clicking anywhere in this box and holding the left-hand mouse button in will display cross-hairs over the image.

  By moving the cross-hairs over the image, the image will improve in focus and sharpness at one point. It is easier to do one direction at a time.

  If the astigmatism is severe and the cross-hairs are close to the edge of the screen when nearing correction, releasing the mouse button temporarily will reposition the cross-hairs in the center of the screen to enable further movement.

RECORDING THE IMAGE

There are three options for recording the image: videoprint, save file, Polaroid. The first two are the most commonly used. There are some things you can change before you record the image such as the information shown on the databar, the scan speed for recording, and whether the image is standard definition or high definition. You should also make sure that the magnification shown on the databar is the correct magnification for the image you are recording (Magnification menu, device....videoprint/photo/screen).

· **Videoprint**

Choose photo scan speed or another slow scan rate to give a still, sharp image. Clicking on Freeze under the Filter menu or the Freeze icon button on the Icon Button Bar will freeze the image. Choosing "integrate 1" under the Filter menu will freeze the image automatically after one scan.

With the video printer switched on, click on the In/Out menu then on the Videoprint! option. A print of the screen display should emerge. The databar may be printed on the videoprint if required, the magnification relating to the format of the videoprint.

The video printer will always 'grab' what is seen on the screen; therefore all scan rates including TV can be copied. This means that care should be taken if a high quality image is needed, e.g. slow scan 3 and freeze, or integrate 1 at photo scan speed.

· **Photograph**

The camera should be loaded with film and the correct exposure parameters selected. These can be found by selecting the menu item Change under the In/Out menu. There is a choice of five film types, with user selectable values of contrast and brightness. Once a selection has been made, these values are used by default until changed by entering new values or by selection of a new film type. The image must be properly focused and stigmated before proceeding.

· **Save File**

Click on "Image..." under the In/Out menu. A list box will appear showing the drives and directories available. You should save your files on the E: drive in the userdata directory. Create a directory under your own name. YOU MUST COPY YOUR FILES AND STORE THEM ELSEWHERE. Files will be deleted periodically (without warning) from the hard disk. You may copy files to a zip disk (PC format) or you can ftp them from the computer to your AFS space.

Choose standard or high definition image under the Filter menu. Save files as .img (microscope specific format) or .tiff (standard graphics TIFF format). Unfortunately, Philips chose to use rectangular pixels in their images, and most graphics programs expect square pixels. You will need to resize your TIFF images to the correct aspect ratio to correct the resulting distortion.

Standard Definition: 344kB (TIFF), 702 x 484 pixels

High Definition: 1.45 MB (TIFF), 1404 x 968 pixels

· **Databar**

Click on Databar under the In/Out menu and enter or delete items by clicking on the appropriate check boxes. There is also a 28-character user box, at the top of the dialogue box, which can be filled with relevant information. When completed click on OK.

ELEMENTAL ANALYSIS

There is an EDX detector attached to this microscope. You can use it to determine what elements are in your sample. The software will allow you to collect spectra and do semi-quantitative composition analysis from particular places on your sample. You can also do elemental mapping to show the distribution of particular elements.

The kV used will determine what elements you will see. Heavier elements will generally require higher kV. The software will show you at what energy (eV) peaks will be for different elements. The incoming kV must be higher than any peak you hope to see, preferably almost 2 times higher.

The interaction volume for x-rays is MUCH larger than the spot size. It depends on the energy of the incoming electrons (i.e., the beam voltage) and the atomic weight of your sample. Use the program "Electron Flight Simulator" to run a Monte Carlo simulation and find out the interaction volume (and therefore the possible resolution) of any elemental analysis you will do.

- You will need to be at eucentric height, i.e. Working distance = 10mm.
- Go to Program Manager. Open Folder EDAX.
- Use program EDX control for collecting spectra, EDX Mapping for determining elemental distribution.
- Leave the microscope in full screen mode for information about the whole scanning area. Use small screen, or spot mode to limit the area.
- Use the stopwatch button just above the spectra window to start and stop a scan. Use the wiper/roller button to clear the scan. From EDX control, spectra can be saved as comma delimited text for export to other plotting programs.
- In EDX Mapping you may have to choose "collect : Spectrum" to bring up the spectrum window, which will then appear to be very similar to the EDX control program.

Ask for a demonstration.

GENERAL GUIDELINES FOR BOOKING THE EMAL MICROSCOPES

1. Do not book time unless you are a qualified user for that particular time period or you will be accompanied by such a user (whose name should appear in the Booking file). This user will be operating the microscope FOR you, NOT training you.

2. Book only the time that you think you will need. Note that the basic Prime Time (weekdays) sessions are four hours long. If you require less time please indicate, in the relevant booking box, the amount of time you desire and try to confine these "split sessions" to the afternoon periods only.

3. Sessions not used or canceled with less than 24 hours notice, may be charged as if used.

4. Booked sessions that are unclaimed 1/2 an hour after the scheduled starting time will be made available for any other qualified user.

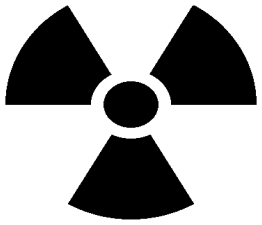
5. User Levels There are four levels of user for each particular microscope and they may be defined as follows:

   1. Unrestricted use, includes days, nights, weekends and holidays.
   2. Use restricted to times when a more qualified user is present in the microscope suite to deal with questions and problems.
   3. Use restricted to times when a more qualified user is present in the microscope room.
   4. Non-users who require electron microscope studies should negotiate with a qualified user to perform the microscopy.

6. **Users may be qualified** by John Mansfield, 936-3352 (ESEM,FEG SEM,2000,4000, AFM) and Corinna Wauchope, 936-3353 (FEG SEM, 2000, 4000, AFM, XPS, Auger) ONLY.

7. Problems with booking the microscopes should be reported to John Mansfield or Corinna Wauchope.

NOTE Users will be expected to be competent enough to operate the microscope without having to refer to the user handbook or manuals before they will be allowed unrestricted use.



Radiation Safety Instructions
for
The North Campus Electron Microbeam Analysis Laboratory
University of Michigan
Space Research Building
2455 Hayward, Ann Arbor MI 48109-2143

By: Alan Jackson, MS
Radiation Safety Service
Occupational Safety and Environmental Health
936-1587

PROCEDURAL

UNIVERSITY OF MICHIGAN USERS

Internal university users should receive this notice when they first apply to use Electron Microbeam Analysis Laboratory (EMAL) instruments, if that instrument is a radiation source or if they will be working in a room that contains an instrument that is a radiation source. This radiation safety information is appended to the instruction handbooks for all radiation producing EMAL instruments.

A copy of the handbook for the instrument that the user wishes to use will be given to each new user together with an EMAL Authorization form. This Authorization Form has two purposes:

1. The users advisor, immediate supervisor, or the owner of the account against which EMAL use will be charged, must sign the form to authorize the said charges.

2. The user should familiarize him/herself with the saftey information in the Instrument User Handbook, including the radiation safety information if the instrument to be used is a radiation source.

Users should read this safety information before returning the signed EMAL Authorization form. Returning the signed EMAL authorization form is an indication that the user has read and understood all of the safety information in the handbook. If there are questions and/or concerns the user should not sign the form until they have discussed these concerns with John Mansfield or Corinna Wauchope.

NON-UNIVERSITY OF MICHIGAN USERS

Non-University of Michigan Users, i.e. those from other universities, industry, national and government labs are given a handbook for the instrument that they are to use before they commence use. They should appraise themselves of the information in the handbooks, paying particular attention to the safety information and then sign the safety section of an EMAL Authorization form.

OBJECTIVES

Promote safety and regulatory compliance

REGULATORY ORGANIZATIONS

Michigan Department of Public Health

The division of Radiological Health regulates the use of x-ray producing machines

PRESENCE OF RESTRICTED AREAS/RADIATION SOURCES

- The electron microscopes produce x-ray radiation.
- The x-ray producing machines may be identified by the posting on the door of the laboratory, the identification markings and stickers on each machine and the registration certificates for

each unit. If you have any question about the location of the machines contact your facility supervisor.

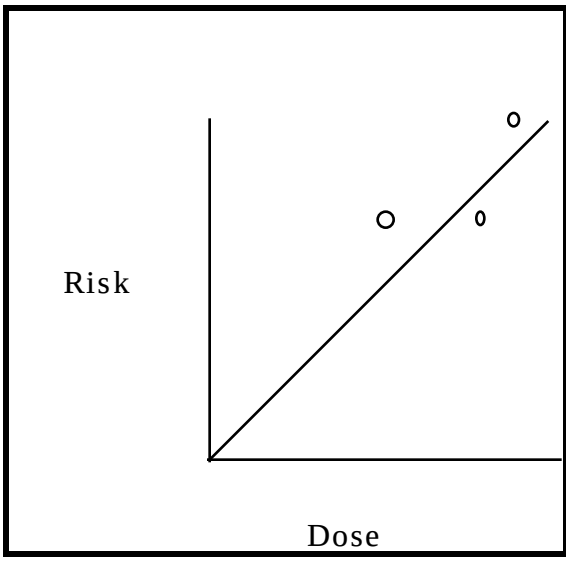
- General procedures describing the use of these units are located on the on the door of each laboratory.
- Each unit has been surveyed by Radiation Safety Service to ensure that the units do not "leak" unacceptable amounts of radiation.

RADIATION BIOEFFECTS

Acute versus Chronic effects

- **Acute**-(Short term effects) Skin Erythema (Like a sunburn), Epilation (loss of hair), Hemopoeitic, Death etc. These acute radiation effects can be eliminated if radiation doses are kept below the occupational dose limits.
- **Chronic**-(Short term effects) Cancer and Genetic effects. These chronic radiation effects can not be eliminated if radiation doses are kept below the occupational dose limits. These effects are stochastic which means that the probability of the effect is a function of your total radiation dose. That is, you are more likely have a radiation induced effect if you have high radiation doses. This is a result of the non threshold dose response relationship.

THE NON THRESHOLD DOSE RESPONSE RELATIONSHIP



This relationship is an assumption (based on some good science) that is used to estimate risks that can not be directly measured. This graph implies that all radiation doses have some risk. The amount of this risk is very low for very low doses but is not zero! Some would say that there is no "safe" radiation dose. This is a misrepresentation. There are radiation doses with negligible risk. I would say that something with a very low risk is "safe."

STOCHASTIC (PROBABILISTIC) RADIATION EFFECTS

Cancer [a]3/10,000 chance of cancer per rem

Normal cancer rate [a]25-33%

Genetic Effects [a]5-75/1,000,000 genetic disorders per rem Normal genetic disorder rate of [a]10%. No radiation genetic effects have ever been demonstrated in human populations.

Latent period-Radiation induced cancers occur after a latent period has elapsed. This latent period is >30 years for most cancers (2 years for leukemia)

Fetal Radiation Effects The fetus and children are 5-10 times more sensitive to radiation induced cancers than an adult. Teratogenic effects are also possible at very high doses.

The magnitude of your radiation risk is dependent on your total radiation dose.

HISTORICAL RADIATION DOSES FOR ELECTRON MICROSCOPISTS

The typical measured radiation dose at the Material Science/ Space Physics Research Laboratory is "M." This "M" refers to less than the minimum detectable amount of the badge. These badges are capable of measuring 0.01 rem per month. The annual dose limit for normal adults is 5 rem per year. Clearly, your radiation dose is expected to be very low. Why is training required for this low risk? The State of Michigan requires this training of electron microscopists and stringently reviews the training records. The regulations which govern the use of electron microscopes were developed in the past (prior to 1970) when poorly designed units could cause significant radiation fields. Obviously, old machines and significantly modified machines could have the safety deficiencies that these regulations are intended to protect against.

PROTECTION METHODS

External versus Internal Radiation Exposure-The radiation exposure comes from an x-ray machine. No radiation lingers after the machine is powered down. There is no risk of radioactive contamination from an x-ray machine**.** Thus, there is no need for special procedures are required for wearing personal protective equipment.

Protection Techniques

Time-Minimize time near sources. Work efficiently.

Distance-Maximize distance from sources. The radiation dose rate is reduced by the inverse square of the distance from the source. When you double the distance that you are standing from the machine your radiation dose goes down by a factor of 4.

Shielding Modern electron microscopes are well shielded. This Engineered control is probably the most important radiation safety feature of modern machines.

Dose limits-Many radiation effects (acute/Nonstochastic) are eliminated if these dose limits are observed

Whole Body-5 rem per year

Extremity-50 rem per year

Skin-50 rem per year

Eye-15 rem per year

Member of Public- 100 mrem per year

Minor-500 mrem per year

Declared Pregnant Women-500 mrem per pregnancy

ALARA Policy-notifications at ª10% and 30% of limits

Safety Systems-Electron Microscopes have a number of safety feature designed to minimize radiation doses. For example, the sample chamber is typically interlocked with the beam on light. This protects the filament and the user. Another important safety system is the warning lights. These warn you when the machine is operating. Beam alignment is an important safety feature. Safety systems must be in perfect condition to operate the unit. Notify the facility supervisor this system malfunction.

Surveys-Surveying the machine after significant alterations is an important safety technique. Radiation Safety Service will survey equipment when requested. The manufacturer repairman should also be able to competently survey the machine after significant maintenance.

Knowledge-You need to fully understand your system if you expect good results and safe operation. Reading the manual for the unit and general texts about EM techniques are important means of eliminating safety and experimental problems.

Personnel Monitoring Techniques

Requirement: if you will receive 10% of dose limits you must wear a radiation dosimeter-no one here is required to be badged. If you insist, a radiation dosimeter will be issued.

Note: Each machine has a badge. These machine badges typically never receive a measurable dose.

Wearing-wear with badge facing outward, rings toward source.

Storing-Store in low background area away from extreme heat.

You have a right to review the results!

You must be informed if you receive an overexposure!

NATURAL BACKGROUND RADIATION EXPOSURES

This is a highly variable value.

Total ª200-300 mrem per year

Radon: ª150 mrem per year

EMPLOYER RESPONSIBILITIES

An employer must provide dosimetry, train employees, provide postings, notify employee of overexposures, provide procedures.

WORKERS RIGHTS

- Read the *Notice to Employees* in your laboratory!-summary
- You have the right to contact MDPH
- You can not be discriminated against for calling regulators

WORKER RESPONSIBILITIES

- Abide by all of the rules
- Keep all the required records

  Do not falsify any record or willfully violate the rules
- Use Protective Techniques: Time, Distance, Shielding and Decay
- Report violations of MDPH rules, licenses or registration certificates
- Report unnecessary exposure of radiation to MDPH

CONTAMINATION VERSUS EXPOSURE

radioactive "dirt"

SURVEYS

Survey results

X-RAY PRODUCTION

Bremmstrahlung

OPERATING INSTRUCTIONS-OPERATOR MUST:

- be authorized by radiation safety supervisor
- be trained to use the unit
- notify Radiation Protection Supervisor of any machine malfunctions, alterations, safety issues
- read, understand follow procedure for unit
- never insert any part of your body into the unit
- never repair unit without authorization
- never alter any interlock for the unit
- never compromise the shielding of the unit
- maintain maximum possible distance from the unit during use
- limit your time near the unit.
- read the notice to employees
- Radiation producing machines are labeled
- Interlocks must function at all times!
- Surveys are performed by a Health Physicist
- Radiation Safety Supervisor- **John Mansfield**

EMERGENCY PROCEDURES

Administer lifesaving first aid without regard to radiation!