

**Camscan Series II
Scanning Electron Microscope
Users Guide**

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**Version 2.0 (1999)
by
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Reference List

General Reviews of Geochemical Analytical Techniques

These texts discuss various analytical techniques, including electron microprobe analysis. They are appropriate for the novice.

AUTHOR Potts, P. J.
TITLE A handbook of silicate rock analysis / P.J. Potts.
IMPRINT Glasgow : Blackie ; New York : Chapman and Hall, 1987.
COLLATION ix, 622 p. : ill. ; 29 cm.
NOTE Bibliography: p. 587-610. Includes index.
SUBJECT Rocks, Siliceous --Analysis.
Geology QE438 .P68 1987

AUTHOR Zussman, J.
TITLE Physical methods in determinative mineralogy / edited by J. Zussman.
EDITION 2d ed.
IMPRINT London ; New York : Academic Press, 1977.
COLLATION xiv, 720 p. : ill. ; 24 cm.
NOTE Includes bibliographies and indexes.
SUBJECT Mineralogy, Determinative --Methodology.
Geology QE367 .Z8 1977

Scanning Electron Microscopy and Microanalysis

Scanning Electron Microscopy and X-ray Microanalysis (A Text for Biologists, Materials Scientists, and Geologists) is the single best reference for scanning electron microscopy and x-ray microanalysis. All users of the scanning electron microscope and electron microprobe are expected to read the pertinent chapters from this text. Your research group should purchase a copy that can be shared among users of the facility. The second edition is generally better than the first edition, but some material did not make the transition; see the first edition if you want the whole picture.

Goldstein, et al., *Scanning Electron Microscopy and X-ray Microanalysis (A Text for Biologists, Materials Scientists, and Geologists)*, 2nd ed. New York: Plenum Press, 1992. ISBN 0-306-44175-6. Geology library QH 212.S3 S29 1992 (The first edition of this book is very good too -- Geology QH212.S3 S29 1981)

Chapter 2 Electron Optics
Chapter 3 Electron - Specimen Interactions
Chapter 4 Image Formation and Interpretation
Chapter 5 X-ray Spectral Measurement: WDS and EDS
Chapter 6 Qualitative X-ray Analysis
Chapter 7 X-ray Peak and Background Measurements
Chapter 8 Quantitative X-ray Analysis: The Basics
Chapter 9 Quantitative X-ray Analysis: Theory and Practice
Chapter 10 Compositional Imaging
Chapter 11 Specimen Preparation for Inorganic Materials: Microstructural and Microchemical Analysis
Chapter 12 Sample Preparation for Biological, Organic, Polymeric, and Hydrated Materials
Chapter 13 Coating and Conductivity Techniques for SEM and Microanalysis
Chapter 14 Database

The workbook **Scanning electron microscopy, X-ray microanalysis, and analytical electron microscopy : a laboratory workbook** is a companion of sorts for the Goldstein textbook (it is used by the Lehigh short courses). It has exercises that may be used in the lab during more advanced demonstrations, and the answers are in the back of the book!

TITLE Scanning electron microscopy, X-ray microanalysis, and analytical electron microscopy : a laboratory workbook / Charles E. Lyman [et al.]
IMPRINT New York : Plenum Press, c1990.

COLLATION xi, 407 p. : ill. ; 26 cm.
NOTE Includes bibliographical references and index.
SUBJECT Scanning electron microscopes --Laboratory manuals.
X-ray microanalysis --Laboratory manuals.
Electron microscopy --Laboratory manuals.
ALT AUTHOR Lyman, Charles E.
Millikan 9 F1 QH212.S3 S335 1990

Scanning and transmission electron microscopy: an introduction is another general reference for electron microscopy. It is written with the TEM in mind and does not go into the detail that Goldstein et al does. It is appropriate for those who do not understand anything in the Goldstein text.

Flegler, et al., Scanning and transmission electron microscopy: an introduction. Oxford: Oxford University Press, 1994. Geology library QH212.S3 F58 1993

AUTHOR Flegler, Stanley L.
TITLE Scanning and transmission electron microscopy : an introduction / Stanley L. Flegler, John W. Heckman, Jr., Karen L. Klomparens.
IMPRINT New York : W.H. Freeman, c1993.
COLLATION viii, 225 p. : ill. ; 27 cm.
NOTE Includes bibliographical references and index.
SUBJECT Scanning electron microscopy.
Transmission electron microscopy.
ALT AUTHOR Heckman, John William.
Klomparens, Karen L.
Geology QH212.S3 F58 1993

The Reimer text **Scanning electron microscopy : physics of image formation and microanalysis** discusses the physics of imaging and microanalysis, and approaches the subject in a elegant but terse manner. It is at the same or more advanced level than the Goldstein text.

AUTHOR Reimer, Ludwig, 1928-
TITLE Scanning electron microscopy : physics of image formation and microanalysis / Ludwig Reimer.
IMPRINT Berlin ; New York : Springer-Verlag, c1985.
COLLATION xviii, 457 p. : ill. ; 24 cm.
SERIES Springer series in optical sciences ; v. 45.
NOTE Bibliography: p. [405]-446. Includes index.
SUBJECT Scanning electron microscopes.
Millikan 9 F1 QH212.S3 R452 1985

Images of Materials offers examples of images collected by various techniques.

TITLE Images of materials / edited by David B. Williams, Alan R. Pelton, Ronald Gronsky ; with a foreword by Peter B. Hirsch.
IMPRINT New York : Oxford University Press, 1991.
COLLATION xiv, 379 p., [32] p. of plates : ill. (some col.) ; 29 cm.
NOTE Includes bibliographical references and index.
CONTENTS Imaging by light optical microscopy / G.F. Vander Voort --
Scanning electron microscopy / D.C. Joy and J.I. Goldstein --
Analytical imaging with a scanning ion microprobe / R. Levi-Setti
Acoustic microscopy / G.A.D. Briggs and M. Hoppe
Transmission electron microscopy / G. Thomas --
Electron diffraction images / D.B. Williams and K.S. Vecchio --
Atomic-resolution microscopy / R. Gronsky
Surface imaging by
Millikan 6 F1 TA418.7 .I44 1991

Electron Microprobe Analysis

Quantitative electron-probe microanalysis is a good text on aspects of quantitative microanalysis, and goes into more detail than Goldstein et al. It covers ZAF and PRZ correction schemes.

TITLE Quantitative electron-probe microanalysis / editors, V.D. Scott

and G. Love.
IMPRINT Chichester, West Sussex, England : E. Horwood ; New York :
Halsted Press, 1983.
COLLATION 345 p. : ill. ; 24 cm.
SERIES Ellis Horwood series in physics.
NOTE Bibliography: p. [320]-336. Includes index.
SUBJECT Electron probe microanalysis.
Microchemistry.
Chemistry, Analytic --Quantitative.
ALT AUTHOR Scott, V. D. (Victor D.), 1928-
Geology QD117.E42 Q36 1983

An introduction to X-ray spectrometry : X-ray fluorescence and electron microprobe analysis is a relatively good text which discusses both instrumentation and quantitation aspects of microanalysis (and related aspects of x-ray fluorescence).

AUTHOR Williams, K. L.
TITLE An introduction to X-ray spectrometry : X-ray fluorescence and
electron microprobe analysis / K.L. Williams.
IMPRINT London ; Boston : Allen & Unwin, 1987.
COLLATION xiii, 370 p. : ill. ; 25 cm.
NOTE Bibliography: p. 364-366.
Includes index.
SUBJECT Rocks --Analysis.
Mineralogy, Determinative.
X-ray spectroscopy.
Fluorescence spectroscopy.
Microprobe analysis.
Geology QE435 .W55 1987

Electron probe quantitation is a collection of papers that discuss the state-of-the-art of microprobe analysis. It is relatively new. It is not an introductory text.

TITLE Electron probe quantitation / edited by K.F.J. Heinrich and Dale
E. Newbury.
IMPRINT New York : Plenum Press, c1991.
COLLATION viii, 400 p. : ill. ; 26 cm.
NOTE "Result of a gathering of international experts in 1988 at ...
National Institute of Standards and Technology"--Pref.
Includes bibliographical references and index.
SUBJECT Electron probe microanalysis --Congresses.
Microchemistry --Congresses.
Spectroscopy.
ALT AUTHOR Heinrich, Kurt F. J.
Newbury, Dale E.
Geology QD 117 .E42 E44 1991

Electron Microprobe Analysis is another text which discusses the technique as an overview. There is a new edition which we do not have a copy of.

AUTHOR Reed, S. J. B.
TITLE Electron microprobe analysis / S. J. B. Reed.
IMPRINT Cambridge [Eng] ; New York : Cambridge University Press, 1975.
COLLATION xvi, 400 p. : ill. ; 23 cm.
SERIES Cambridge monographs on physics.
NOTE Bibliography: p. 381-395. Includes index.
SUBJECT Microprobe analysis.
Millikan 8 F1 QD117.E42 R43

Fundamentals of Energy Dispersive X-ray Analysis is an introduction to EDS and offers an in-depth discussion of the instrument and its pros and cons. It is enlightening for the novice and should be read for those sem users who do EDS analysis.

AUTHOR Russ, John C.

TITLE Fundamentals of energy dispersive x-ray analysis / John C. Russ.
 IMPRINT London ; Boston : Butterworths, 1984.
 COLLATION 308 p. : ill. ; 24 cm.
 SERIES Butterworths monographs in materials.
 NOTE Bibliography: p. 295-304. Includes index.
 SUBJECT X-ray spectroscopy.
 Millikan 8 Fl QD96.X2 R87 1984

Microbeam analysis are the proceedings volumes of the annual meeting of the Microbeam Analysis Society. These volumes contain almost all research papers (as extended abstracts) concerning various aspects of electron microprobe analysis. A bibliographic database of papers from these proceedings is under construction.

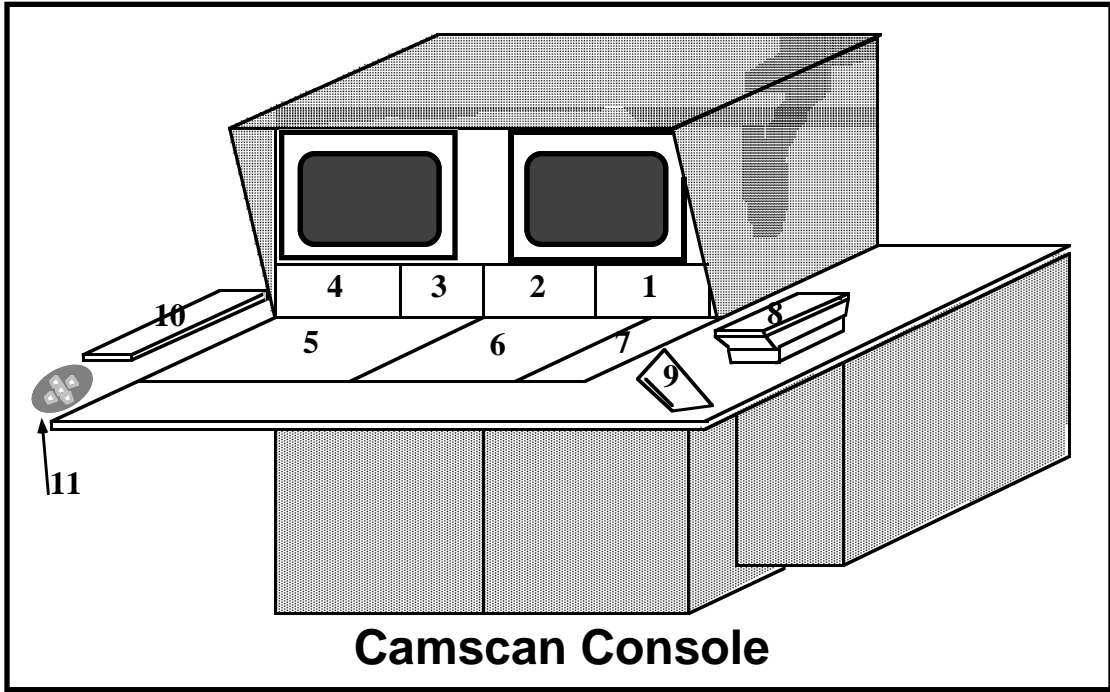
TITLE Microbeam analysis.
 COVERAGE 14th (1979)-
 IMPRINT San Francisco, Calif. : San Francisco Press, c1979-
 FREQUENCY Annual.
 COLLATION v. : ill. ; 29 cm.
 LIB. HAS GEOL has: 14th (1979)+
 Millikan 9th floor has: 16th (1981), 17th (1982), 19th (1984)+
 NOTE Proceedings of the 14th- Annual Conference of the Microbeam
 Geology QH212.E4 N3 1981

X-ray fluorescence analysis in the geological sciences : advances in methodology

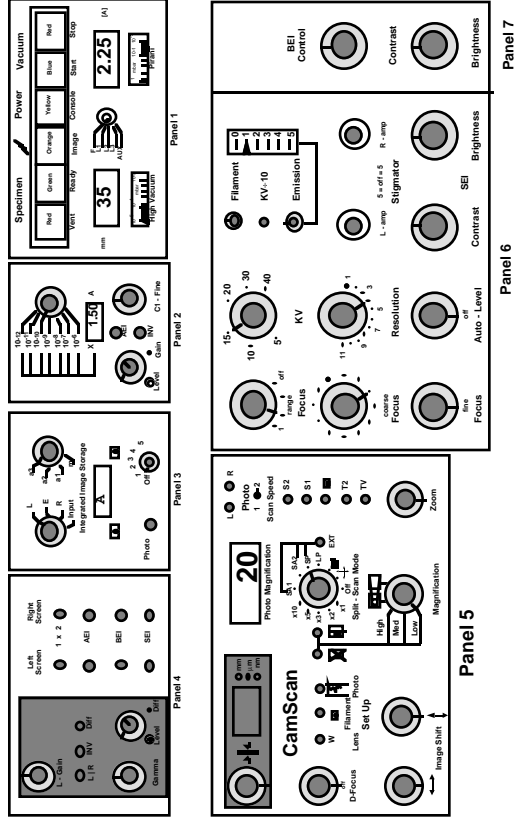
TITLE X-ray fluorescence analysis in the geological sciences : advances
 in methodology / S.T. Ahmedali, editor ; contributors, S. Abbey [et al.]
 IMPRINT St. John's, Nfld. : Geological Association of Canada =
 Association geologique du Canada, 1989.
 COLLATION xii, 297 p. : ill. ; 28 cm.
 SERIES Short course notes (Geological Association of Canada) ; v. 7.
 NOTE Includes bibliographical references.
 SUBJECT X-ray spectroscopy.
 Analytical geochemistry.
 ALT AUTHOR Ahmedali, S. T. (Syed Tariq), 1941-
 Geological Association of Canada.
 Geology QD96.X2 X729 1982

Advances in x-ray analysis : proceedings of the annual Conference on Application of X-ray Analysis are the proceedings of an annual conference on x-ray analysis. These volumes contain older papers on x-ray analysis.

AUTHOR Conference on Application of X-ray Analysis.
 TITLE Advances in x-ray analysis : proceedings of the annual Conference
 on Application of X-ray Analysis.
 COVERAGE Vol. 1 (6th/1957)-
 IMPRINT New York, London, Plenum Press, 1960-
 FREQUENCY Annual.
 COLLATION v. ill. 24-28cm.
 LIB. HAS GEOL has: v.1 (1957)-v.24 (1980), v.26 (1982)+
 Geology TA417.65 .C6 v.1 (1957)



Camscan Console Master Diagram



SEM Startup Begins Here

Initial Startup Settings and Final Shutdown Settings

This section covers where the controls are and what the correct startup and shutdown settings are for the SEM. Figure 1 shows the location of the controls. Go through the following sequence and verify that each control is in its correct default position before you start your work. You will also use this section to set the controls back to the startup settings at the end of your run.

You will be charged a penalty if you fail to put the controls back to their startup settings at the end of a run. If you come to use the SEM and the controls have not been put back to their startup settings, make a note in the logbook.

For advanced users: Note that the appropriate action is indicated by a bullet • next to the required setting. If you have used the instrument many times, you may scan down the page to the next bullet without the need to read all the explanatory text. However, do not skip any steps.

Panel 1:

The system status lights are as follows, from left to right:

Specimen Vent (red): The Specimen Vent push button is used to vent the entire column including the sample chamber. This is normally done **ONLY** for large samples. Do not use this without supervision.

NOTE: do not confuse this with the **vent** button on the column which is used to vent the sample exchange chamber when you put samples in!

- Verify **Specimen Vent** is **NOT** pressed in and not illuminated.

Specimen Ready (green): The Specimen Ready push button is illuminated when the vacuum is below 10^{-4} torr. We run the instrument at 10^{-5} torr, so this light being on is a necessary but not sufficient condition for turning on the high voltage. (The **high vacuum** gauge below the system status lights should be at 10^{-5} torr before high voltage is turned on.) If the light is not on, there is a vacuum leak or the machine was just vented and is still pumping down. Either wait a few minutes or get help.

NOTE: The Specimen Ready push button is used to turn off the high voltage when you are about to do a sample exchange. The high voltage is turned on by pressing the Image push button, and it is turned off by pressing the Specimen Ready push button.

- Verify that the **Specimen Ready** is illuminated.

Image (orange): The Image push button turns on the high voltage when it is pushed in (the

Specimen Ready push button is pressed to turn off the high voltage). The instrument vacuum level must be lower than 10^{-4} torr in order for the Specimen Ready light to come on (and high voltage to be enabled), and we prefer to run the instrument at 10^{-5} torr. It is also necessary for the **viewport cover** on the column to be in place and rotated fully clockwise (this cover protects the secondary electron collector from being swamped by room light).

- The **Image** button should not be illuminated at this time. You will turn it on prior to filament saturation.

Console Power (yellow): The Console Power push button is used to supply power to the console, display screens, and stage motors.

- Turn on **Console Power** power.

Vacuum Start (blue): The Vacuum Start push button turns on the vacuum system (roughing and diffusion pumps) and attempts to reach high vacuum. This is always left on.

- Verify that the **Vacuum Start** push button is illuminated.

Vacuum Stop (red): Turns off the vacuum system for full instrument shut down. **DO NOT PRESS** this button! We leave the instrument under vacuum at all times. If the vacuum logic of the camscan detects a problem, the instrument will automatically "retreat" back to vacuum stop after shutting down the diffusion pump. If you observe the **Vacuum Stop** light on, get help immediately.

- Verify that the **Vacuum Stop** push button is **NOT** illuminated.

High Vacuum gauge: Measures pressure inside the column. This should be at 10^{-5} torr before high voltage is turned on.

- Verify that the **High Vacuum** gauge is reading close to 10^{-5} torr (far left of scale).

Digital readout monitor: The Digital readout monitor (between WD mm and Filament Current displays) is used to read the filament current when the selector is set to F.

- Verify that the **Digital readout monitor** is set to F.

Panel 2:

Absorbed current range selector: the absorbed current range selector is used to set the range for digital readout of the absorbed current. It is set to 10^{-6} range except for filament saturation and EDS analysis.

- Verify that the **Absorbed current range selector** is set to 10^{-6} range (fully counterclockwise).

C1 Fine condenser lens: the C1 fine condenser lens is used to set the fine adjustment of the probe current via excitation of the condenser lens system. This control is set fully counterclockwise except for EDS analysis and variable current backscattered-electron imaging.

- Verify that the **C1 Fine condenser lens** is fully counterclockwise.

Panel 3:

Integrated Image Storage: The Integrated Image Storage unit (or "frame store") is controlled on this panel and the frame store controller pad (number 11 on console perspective diagram). This

unit is used to average subsequent frames of the scanning image, which serves to reduce the noise and increase the signal to noise ratio of the image being viewed.

- Verify that the **Integrated Image Storage** selector switches are set to the following:
top left switch (input) set to L
top right switch set to a3
- Verify that the **Integrated Image Storage** power switch (lower right corner) is off

NOTE: The orange Photo push button on the Integrated Image Storage unit is used to dump the contents of the frame store buffer to the polaroid camera. This buffer has only 500 line resolution and will result in a grainy picture. For this reason, do not push the orange Photo button for photography, use instead the blue Photo button on panel 5.

Panel 4:

Image Selector: The Image Selector has two banks which are used to select which image is viewed on the left and right viewing screens of the SEM. The instrument is normally run with the secondary electron image on the left screen (bank 1 controls), and the backscattered-electron image on the right screen (bank 2 controls).

- Verify that the **Image Selector** bank 1 (left screen) is set to SEI (i.e. secondary electron image) with the SEI button pressed in, and no other buttons on this bank are pressed in.
- Verify that the **Image Selector** bank 2 (right screen) is set to BEI (i.e. backscattered-electron image) with the BEI button pressed in, and no other buttons on this bank are pressed in.

Panel 5:

Linewidth measurement >||<: This feature is not activated on our instrument; these measurements can be made using the scale bar in imaging mode.

D-Focus: This is the dynamic focus control, which is used to correct for depth of field problems when imaging on a tilted surface. It modifies the scan of the SEM to correct for tilt, and results in a compromise in image distortion and depth of field. It only corrects for tilting in the direction toward the top of the image screen. It must be turned off for normal imaging, or distortion will result.

- Verify that the **D-Focus** knob is fully counterclockwise.

W Lens: The 'wobbler' lens is used to perturb the imaging in a way that allows one to center the objective aperture (if necessary) as the final step in column alignment. This is normally done by the lab personnel, but we can show you how to check the alignment. It is normally left off during imaging.

- Verify that the **W Lens** is off (not pushed in).

[•] Filament: The [•] Filament button (when pushed in) allows us to actually image the electron beam crossover point below the electron gun. This is used for the filament saturation procedure, and is turned off during normal imaging. The sample cannot be imaged normally when filament imaging is enabled.

NOTE: The absorbed current cannot be read with the absorbed current meter when the [•] filament button is pushed in.

- Verify that the **[•] Filament** button is off (not pushed in).

The **Photo linescan** button is used to display contrast and brightness information from the image on the left screen as a line profile display on the right screen. The linescan is a horizontal slice of the contrast and brightness function the left screen image taken halfway up the screen in the x-axis direction. On the right screen, the profile is displayed vertically, with the left side displayed at the top, and the right side displayed at the bottom. The left vertical line of the line profile display represents black, and the right vertical line of the display represents 80% brightness. The position of the signal in this line profile display is its brightness, and the amplitude of the signal is its contrast.

- Verify that the **Photo linescan** button is **NOT** pressed in.

Image Shift: The **Image Shift** controls have two functions:

1. When the **Split-Scan Mode** selector is set to SP (Spot mode), the **Image Shift** knobs control the screen position of the spot marker. Note that the **orange [X] spot mode** button must be pressed in for spot mode to be activated.
2. In normal imaging, when the **Magnification** selector knob is set to selected-area display (the setting just above high magnification), these controls move the selected area scan box on the scanning image (rather than a cursor); and at highest magnification (most clockwise of the two box settings), the image on the screen is the area scanned inside this selected area box.

- The **Image shift** knobs should be left where they are.

Photo Magnification: This is where the magnification is displayed. This number is not actually recorded on a photograph, but the magnification can be calculated from the length of the scale bar on a photograph. The magnification is set using the **Magnification** knob.

[X] Orange spot mode: When the **[X] Orange spot mode** button is pushed in, the electron beam is no longer rastered (and therefore no image is displayed on the screen), and the electron beam is now fixed at the position of the cursor. The **Split-scan selector** knob must be set to SP for this to work properly (in this mode the cursor is displayed on the screen). The cursor can be positioned using the **Image Shift** knobs while in the normal imaging mode (i.e. [X] is not pushed in). The cursor should be positioned while at relatively high magnification (i.e. the scale bar is on the order tens of microns); this minimizes the placement error in positioning the cursor. The [X] button is used to select spot mode for the acquisition of an EDS x-ray spectrum or a semi-quantitative analysis.

- Verify that the **[X] Orange spot mode** is off (not pressed in).

Split-Scan Mode: When set to SP (Spot mode), the cursor represents the position of the beam when the instrument is placed in spot mode (using the orange [X] switch). The selected area is positioned using the Image Shift controls. Several of the comparison scan modes are activated via split-screen imaging, where two halves of the screen are used to display the image at different magnifications.

- Verify that the **Split-Scan Mode** selector is set to SP.

EXT External beam control: Control of the scanning image can be handled externally by another computer. We have several programs that allow us to acquire digital images using this feature.

- Verify that the **EXT** button is **NOT** pressed in.

Magnification: This is the coarse magnification control -- it has the settings Low, Medium, High, Selected Area Positioning, and Selected Area High Mag. Continuous zoom is provided by the Zoom control for all modes except Selected Area Positioning.

- Verify that the **Magnification** knob is fully counterclockwise.

Zoom: The Zoom control provides a continuous range of magnification within current coarse magnification selected.

- Verify that the **Zoom** control is fully counterclockwise.

Photo: The left blue Photo button is used to take a polaroid photograph of the selected screen. We always use the left screen for photography because it has less noise than the right screen.

Note: Do not confuse the blue Photo control with the orange Photo button of the frame store. The orange Photo push button on the Integrated Image Storage unit is used to dump the contents of the frame store buffer to the polaroid camera; this buffer has only 500 line resolution and will result in a grainy picture. The blue Photo push button initiates a slow photo scan that has 2500 line resolution and will result in a much higher resolution polaroid photograph.

- Verify that the left **Photo** button is selected.

Photo scan speed: The Photo scan speed switch selects the scan rate for Polaroid photography, 1 is for a 30 sec exposure, and 2 is for a 120 sec exposure. We always use 2.

- Verify that the **Photo scan speed** switch is set to 2.

Scan speed: The Scan speed push buttons are used to select different scan formats and scan rates. The available scan formats are:

S2 Slow 2: This is the slowest scan rate (you will need to turn out the lights to see this clearly)

S1 Slow 1: Faster than S2 and same scan mode

[] Rapid Scan: A reduced area rapid scan mode that is used for focus and stigmation

T2 Tv 2: A more rapid scan mode using the actual scan area sampled by the polaroid camera

TV TV: The most rapid TV-rate scan for the full screen image format. This mode is used by the frame store and can be viewed easily with the room lights turned on.

Normally TV-rate is used for most rapid scanning. T2 is a TV-rate image at the format used for photography (scale marker is not displayed). The small box mode is Rapid Scan mode, used for high resolution focus and stigmation adjustment. S1 and S2 are slow scan modes used for higher resolution and low noise imaging (which can also be accomplished by using the frame store, however).

- Verify that the **Scan speed** is set to TV-rate (the bottom push button).

Panel 6:

Focus Range: The Focus Range control is a 4 position selector switch that is used to set the working distance range for the objective lens. The working distance ranges are:

1 = 0 to 22 mm

2 = 15 to 55 mm (normally used)

3 = 40 to 144 mm

Off = lens current zero for hysteresis purging.

- Verify that the **Focus Range** is set to 2 (this is the second dot from the left).

Coarse Focus: The **Coarse Focus** knob is a 23-position step control for the selection of working distance for imaging (selected within the range set using the Focus Range control). The value shown in the working distance display is obtained when the **Fine Focus** control is set precisely at 5 turns from either limit.

Note: On our instrument the **Coarse Focus** control is normally set to 35 mm working distance as a standard procedure. This means that the sample will be focused using the z-axis crank on the electron column for coarse focusing (i.e. the sample is positioned to the z-axis position where the electron beam is focused). When the 35 mm working distance is selected in this way, the x-ray takeoff angle relative to the Tracor and Link EDS detectors is exactly 35 degrees. This condition must be met in order to perform quantitative analysis on the instrument, since the standard EDS spectra were collected under the same standard condition of 35 mm working distance.

- Verify that the **Coarse Focus** is set to 35 mm working distance (displayed on mm display of Panel 1). **Special Procedure:** Set the Coarse Focus by turning the control counter-clockwise to a lower value of the working distance, and then turning it up to 35 mm (this is to counteract hysteresis of the magnification system).

Fine Focus: The Fine Focus knob is a continuous variation fine focusing control that is used to focus the image at high magnification. It is a ten-turn knob that must be set to **precisely** five turns from either limit for the standard condition (i.e. if one is doing quantitative EDS analysis). The setting of this control for imaging applications is not important.

- Verify that the **Fine Focus** knob is set **precisely** 5 turns from either limit (DO NOT force the knob past the limit!).

KV: The KV selector knob is used to select the accelerating voltage. Normally we run the instrument at 15 Kev since this is the best accelerating voltage for x-ray analysis (and the standard spectra were collected at this voltage). For some high resolution imaging a higher accelerating voltage may be used.

- Verify that the **KV** selector knob is set to 15 KV.

KV / 10: The KV / 10 push button is used to obtain low accelerating voltage conditions by dividing the accelerating voltage selected using the KV knob by 10. For example, when the KV is set to 15 and KV / 10 is pressed in, 1.5 KeV is obtained. Note: image acquisition at low KV is much more difficult compared to higher voltages. Surface contamination becomes a predominant feature in the image.

- Verify that the **KV / 10** push button is **not** pushed in.

Resolution: The Resolution selector knob is used to control the magnitude of the condenser lens current, which in turn controls the range of probe current available on the instrument. There are two ranges available for use:

High current range (4 to 1, where 1 provides the highest probe current)

Low current range (11 to 5, where 5 provides the highest probe current in the low current range)

The highest probe current available on the instrument is at a setting of 1, with the C1 Fine Condenser lens turned fully clockwise (this provides a probe current of 10 - 15 nA depending on which objective aperture is in place). The lowest probe current available on the instrument is at a setting of 11 (which provides a probe current of just a few pA). Because the minimum achievable beam diameter is directly related to the probe current used (low current = narrow beam, high current = wider beam), this control is labelled 'resolution' when it really is controlling the probe current directly, and the beam diameter indirectly. It should really be labelled "Probe Current".

- Verify that the **Resolution** control is set to 4.

Auto Level: This function is not enabled on our instrument. The control should be turned fully counterclockwise and left there.

Filament current: The Filament current control is used to set the current (in microamps) passing through the tungsten filament of the electron gun. When the current is sufficiently high,

the tungsten filament emits electrons and the electron beam can be generated. The Filament current value is read off the digital display marked by [A] on Panel 1.

Note: The **Filament current** knob **MUST** be fully counterclockwise at the beginning of a run (when the electron gun is cold).

- Verify that the **Filament current** knob is fully counterclockwise.

Emission current: The Emission current control is used to set the emission current (in microamps) for the electron gun. The emission current meter (connected with a line to the Emission current knob -- see Panel 6) shows the emission current (where 1 is 100 microamps). The emission current is used to set the electron gun bias, which is the electric field that focusses the electrons at the initial crossover point above the anode plate.

- Verify that the **Emission current** knob is fully counterclockwise.

Stigmators: The Stigmators are two 10-turn potentiometers that are used to correct beam astigmatism when imaging. This corrects for non-circular beam cross-section of the electron beam, which is mapped to the circular beam of the display screens. The stigmators should be set at high magnification after the fine focus has been set as well as can be done (using rapid scan mode). The stigmators are then individually adjusted for the sharpest image.

- The **Stigmator** knobs should be left as they were last set -- there is no standard condition.

SEI contrast: The SEI contrast knob is used to adjust the contrast of the secondary electron image.

- Leave as is initially -- there is no standard condition.

SEI brightness: The SEI brightness knob is used to adjust the brightness of the secondary electron image.

- Leave as is initially -- there is no standard condition.

Panel 7:

BEI contrast: The BEI contrast knob is used to adjust the contrast of the backscattered-electron image.

- Leave as is initially -- there is no standard condition.

BEI brightness: The BEI brightness knob is used to adjust the brightness of the backscattered-electron image.

- Leave as is initially -- there is no standard condition.

Camera (8):

The polaroid camera can be used for either Type 53 or Polapan 400 4 x 5 sheet film. The f-stop needs to be set for the type of polaroid film that you plan to use during your session. Do this as follows:

- Go around to the right side of the SEM table and stand by the camera. On the right side of the camera (as you view it) is a small silver knob that says "open" and "lock" with a silver dot next to "lock". Turn it counterclockwise to "open" (i.e. the word "open" is next to the silver dot).
- Carefully lift the whole camera out of the hole it rests in. Do not knock the camera against the table, and also do not let dust or debris fall inside the hole. The screen that is used for photography

is at the bottom of this chamber, and any dust that falls in there will land on top of the photo screen.

- Check the f-stop setting. It should be set as follows:

f-11 for Polapan 400 or Type 52 film

f-18 for Type 53 film (f-18 is the dot between f-11 and f-22 on the camera aperture)

Note that the instrument is calibrated for these film types only

Note: Do Not coat Type 52 prints in the lab. Take the prints back to your office and coat them there.

- Carefully replace the camera. Turn the silver knob clockwise back to "lock" (i.e. the word "lock" is next to the silver dot).

Console Keyboard (9):

The **Console Keyboard** is used to type text on the screen and/or data strip of the image. The data strip at the bottom of the image shows a scale bar that is keyed to the magnification that is currently selected. The data strip is turned on when the red button on the Console Keyboard is pressed in. A second step is necessary to remove a garbage character that is present when the instrument is first started up (i.e. when console power is first turned on)

- Verify that the red button on the **Console Keyboard** is pressed in.
- To get rid of the garbage character on the screen (at upper left), press the Clear button on the Console keyboard, and then press the "down arrow" button until the cursor is at the bottom left of the display screen. This places the cursor in the data strip area, which is where it is recommended that you put a short text identifier.

Stage Controller (11):

The **Stage Controller** has four buttons with arrows to drive the x-stage and y-stage motors.

Note: The motor speed depends on the magnification used, and drives fastest at 20x magnification, and slowest at high magnification. The red button, normally out, is pressed in to enable one to position the stage one frame at a time, rather than driving the stage only when the arrow buttons are pushed. This is used for search-mode operations.

- Verify that the red button of the **Stage Controller** is out (i.e. **not** pushed in).

Column:

Allen-head Screwdriver: The Allen-head Screwdriver is supposed to be kept in a socket on top of the sample chamber on the Camscan.

- Locate the Allen-head Screwdriver. You will need it to attach your sample holder to the stage assembly for sample exchange.

Z-axis crank ("Z"): The Z-axis crank is used to raise or lower the sample relative to the plane of focus of the electron beam. This control moves the entire stage assembly at whatever setting of tilt and rotation are currently dialed in.

Note: The sample exchange position is 35.5 mm, which is read as 355 on the z-axis odometer.

- Verify that the **Z-axis crank** is set to the sample exchange position of 35.5 mm.

Rotation ("ROT"): The Rotation control is used to rotate the sample from 0 - 360° within the currently selected plane of tilt.

Note: The sample exchange rotation setting is 0°.

- Verify that the **Rotation** is set to 0°.

Tilt ("TILT"): The Tilt control is used to tilt the sample toward the secondary and EDS detectors (i.e. to the left as viewed from the front of the column, and toward the top of the screen as viewed on the console). You must check the clearance of the sample while setting the tilt angle by looking through the viewport. **Do not** set the tilt greater than 45° without supervision; tilt at high angles places great stress on the stage!

Note: The sample exchange tilt setting is 0°, i.e. no tilt.

- Verify that the **Tilt** control is set to 0°.

z' axis (eucentric z): The z' axis is the eucentric component of z-axis positioning for the sample. Eucentric z is perpendicular to the plane of tilt and rotation. If tilt is set to 0° then z' is parallel to z. At any angle of tilt, z' is the offset perpendicular to this tilt plane, while changes in z affect the vertical height of the entire stage assembly. Eucentric positioning is used mainly for stereo photography.

Note: The sample exchange setting for z' is 5.6 mm.

- Verify that the **z' axis** control is set to 5.6 mm.

X and Y stage odometers: The X and Y stage odometers display the stage position in millimeters for both the x-stage and y-stage coordinates.

How to read the stage odometers: The stage odometers are read as nn.n millimeters so the last digit is the fractional part of the stage position, for example:

902 is 90.2 mm
125 is 12.5 mm

Range of X and Y: The usable range of the stage is about +50.0 mm to -50.0 mm for both X and Y. **Warning** -- the odometers do not display the negative sign in their position, so you must pay attention to where you are on the stage. The range of the X and Y odometers can be thought of as follows:

Position	X odometer	Y odometer
Center of stage	000 (=100)	000 (=100)

For sample exchange the motors are positioned to the closer of 000 or 100 mm, for example:

if at 10.2 drive **down** to 000 (=100)
if at 90.5 drive **up** to 100 (=000)

- Verify that the **X and Y stage odometers** are both at 000.

Link EDS Detector: There are currently two EDS detectors on the SEM but only the Link EDS detector is used:

The white **Link EDS** detector has both Be window and thin window modes, and can be used for elements Be and higher, although it's intended use is for better sensitivity of elements such as Na (and the detection of Be, B, C, and N are very difficult). The **Link EDS** detector is withdrawn to

10 cm when not in use. The **Link EDS** detector is placed at 6 cm for quantitative analysis.

- Verify that the **Link EDS detector** is withdrawn to the 10 cm position. However, if you plan to do EDS analysis, insert the detector to the analysis position of 6 cm. If you have not done this before, get someone to help you.

Viewport cover: The window to the sample chamber is covered by a metal viewport cover. All positioning of the sample, and settings of the tilt control and z-axis positioning must be verified visually by removing the viewport cover and checking the clearance between the sample and the detectors. The viewport cover must be replaced and **rotated clockwise** or the high voltage will not turn on.

- Verify that the **Viewport cover** is on and rotated fully clockwise -- **Do Not force it**.

Sample Exchange chamber: When introducing a sample from outside the SEM and placing it into the high-vacuum environment inside the instrument, which is at 10^{-5} torr, it is necessary to use a sample exchange chamber that is pumped down to vacuum for sample exchange. Later, the sample must be removed from this high vacuum to atmospheric pressure outside the instrument, and again this airlock is used.

Because the column of the SEM normally is not vented during this procedure, there is a sample exchange chamber that is evacuated in order to open up the inner chamber for sample exchange. There are three buttons which control the vacuum system during sample exchange, a gate valve, and the sample exchange rod. The vacuum controls are:

Pump (blue): The Pump push button evacuates the sample exchange chamber prior to sample exchange. This does not guarantee that sufficient vacuum will be reached (especially if you try to pump down a sample that has wet carbon paint on it -- this must be dried out in the gold coater first)

Ready (green): The Ready light (on the column, not on the console) indicates that an adequate vacuum has been reached, and the gate valve can be opened for sample exchange. If one tries to open the gate valve before the sample exchange chamber has been evacuated, a beeping alarm will sound. Note that this is an indicator light that tells you when adequate vacuum is reached; you **do not press this button**.

Vent (red): The Vent push button (again, on the column not on the console) allows one to vent the sample exchange chamber after the sample has been put into, or taken out of, the chamber of the SEM. The gate valve must be closed, or this will vent the instrument.

Note: The Vent button must be pressed twice to fully vent the exchange chamber.

- Press the **Vent** button twice to make sure the exchange chamber has been vented.
- Verify that none of these three buttons is still illuminated.

Now proceed to Sample Exchange

Sample Exchange

**Use 1 to Insert a Sample
Use 2 to Remove a Sample**

In this section you will insert your sample into the chamber at the base of the column. If you will be doing quantitative analyses, you should start by inserting the block "Standard 1." and locating the fayalite standard which is used to check and/or calibrate the gain of the EDS detector.

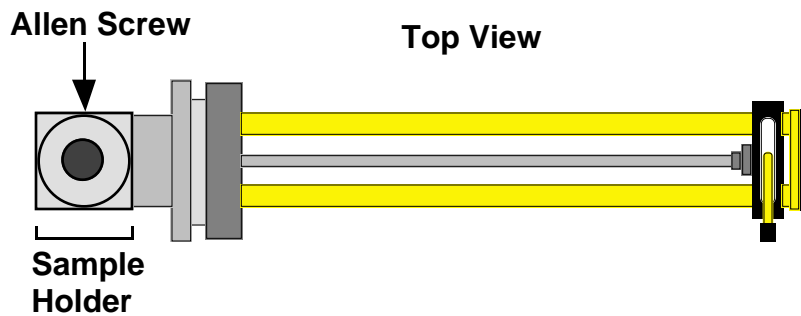
You should draw a map of your sample(s) before putting them in the SEM to help you navigate later.

NOTE: You may be banned from the lab if you refuse to wear gloves while handling sample holders or any other component that is used in a high-vacuum environment. The acid from fingerprints permanently etches and damages the metal surface of these holders. You may be charged for the replacement of sample holders as well. You have been warned.

1 -- Inserting a Sample into the SEM

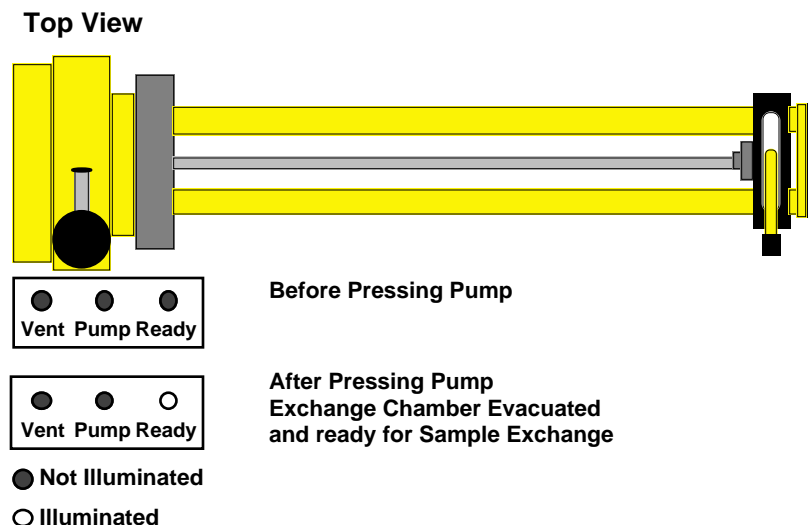
1. The instrument must be set to the sample exchange coordinates before you can perform the sample exchange. You should do or verify the following:
 - **Z axis** set to 35.5 mm working distance using the Z-axis crank
 - **Rotation** set to 0° using the Rotation control
 - **Tilt** set to 0° using the Tilt control
 - **z' axis** (eucentric z) set to 5.6 mm using the z' axis control
 - **Magnification** set to 20 or whatever the minimum setting is
 - **X and Y stage odometers:** Drive the X and Y stage motors to 000 mm (same as 100) as follows:
If the odometer reads less than 50 drive **down** to 000 (i.e. from 20.5 drive to 000)
If the odometer reads more than 50 drive **up** to 000 (i.e. from 85.2 drive to 100 = 000)
2. To insert or remove samples during sample exchange, the high voltage must normally be turned off. Look at the orange "Image" button on Panel 1. If it is illuminated, high voltage is on. Turn it off by pressing the green "Ready" button next to it.
 - Press the green **Ready** button on Panel 1.
3. **Put on gloves.** You must wear gloves whenever handling samples or the exchange rod.
4. Mount your sample in the appropriate holder and insure electrical ground by using carbon paint. **Do Not put samples with wet carbon paint into the machine!** First outgas the sample in the gold coater to achieve a vacuum in the black gauge range (0.2-0.1 torr), or simply wait 1-2 hours until the paint dries.

5. Rotate the **viewport cover** counterclockwise and take it off. Look inside. If there is a sample inside, you have to take it out before you can put another one in. If so, jump ahead to Sample Removal.
6. Push the red **Vent** button twice to completely vent the exchange chamber.
7. Hold the sample exchange device by the large metal collar next to the instrument and remove the sample exchange device from the Camscan by turning counterclockwise -- do not twist the exchange rod itself!
8. Lay the exchange rod on the console desk of the SEM.
9. Place your sample in the stage sample holder and gently tighten with the allen wrench / screwdriver located on top of the Camscan chamber (see figure below). You may want to sketch a map of your sample(s) before inserting them into the chamber. The edge you put farthest to your left in the chamber will be at the top of the TV screen ("north") when you image it.

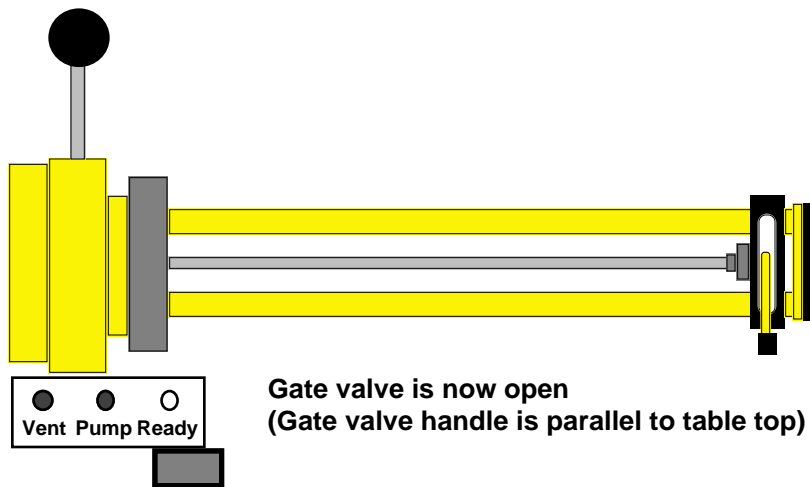
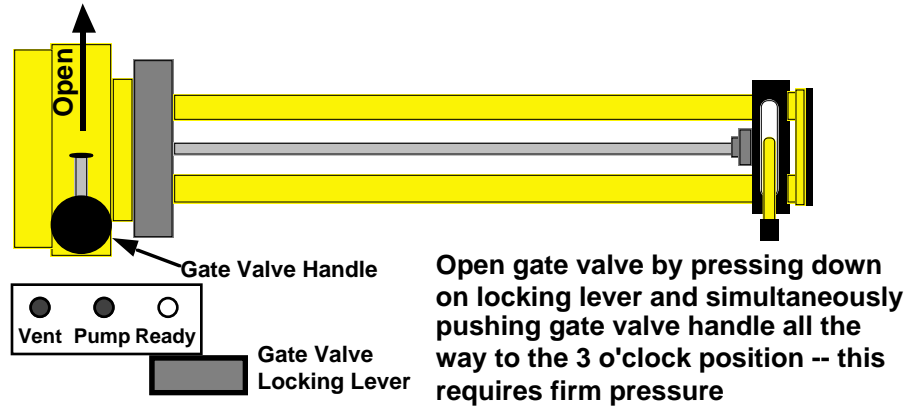


10. Replace the sample exchange device on the Camscan (clockwise to seal). Make sure that the exchange rod is locked in the fully extended and locked position.
11. Evacuate the exchange chamber by pressing the blue **Pump** button, located below the gate valve lever. Wait for the green **Ready** light. If the **Ready** light does not come on, then your sample has not outgassed enough to be placed in the machine-- remove it and pump down in the gold coater. If the **Ready** light is on, go to next step.

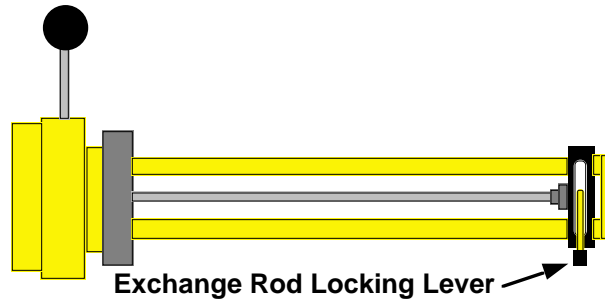
NOTE: This figure and the following ones have an error. The second box (lower) below should show that **both** the Pump and Ready lights are illuminated when the sample exchange chamber has been pumped down.



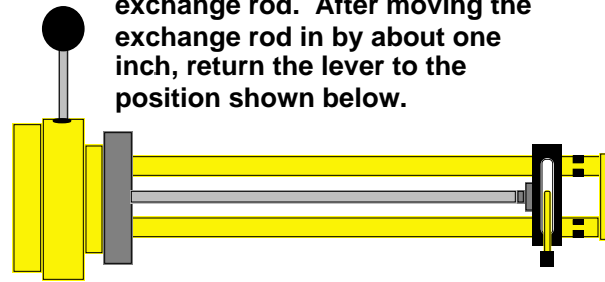
12. While watching the Camscan high vacuum gauge on the control console (Panel 1), slowly open the gate valve. Do this by simultaneously pushing down on the gate valve latch and pushing the gate valve away from you until the gate valve is horizontal. If you hear a beeping noise, you have not pumped down the exchange chamber; go back and do that now. The gate valve does not begin to open until the rod is past the 2 o'clock position, and the gate valve is completely open at the 3 o'clock position.



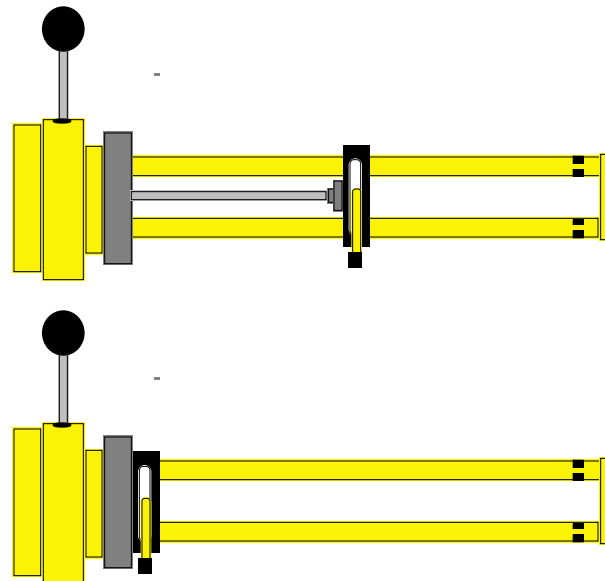
13. Release the exchange rod by lifting the locking lever out of the exchange rod's groove at the end.



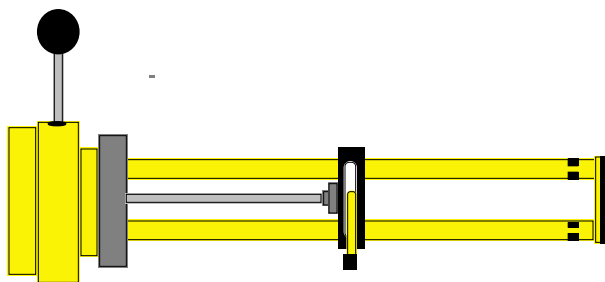
The exchange rod locking lever must be raised up out of the groove in order to unlock the exchange rod. After moving the exchange rod in by about one inch, return the lever to the position shown below.



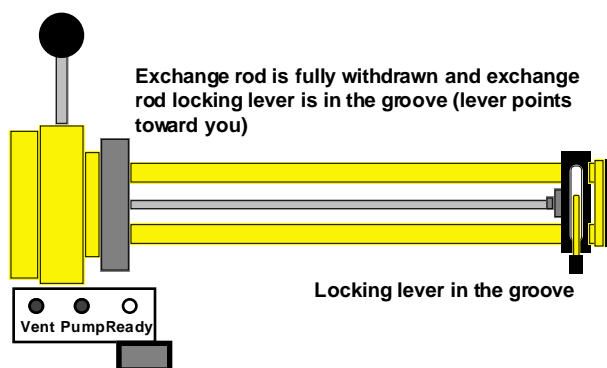
14. Verify that the locking lever is pointed toward you as shown above.
15. Slowly push the exchange rod all the way in while watching through the viewport. The sample/stage assembly should smoothly slide on the holder. If it does not, check the stage settings again. If you still have difficulty, try changing the Z-axis position by a few tenths of a mm, or change the stage rotation slightly.



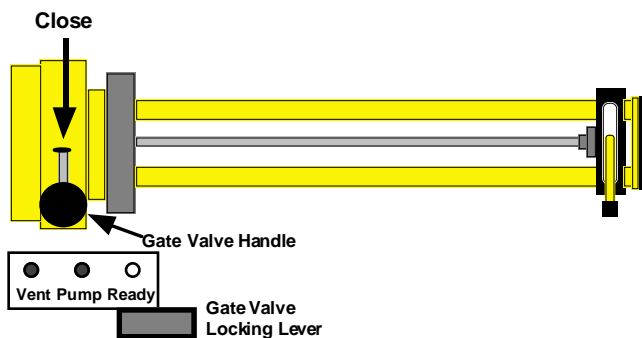
16. Disengage the sample by simply pulling the exchange rod back out, with the locking lever pointed toward you (as shown below). The sample and stage assembly will remain in the machine due to friction (it will generally move a little).



17. When the exchange rod is fully withdrawn, put the locking lever back into the groove at the outermost end of the rod.



18. Close the gate valve by pulling it up and toward you until the gate valve latch locks again. Do not force the gate valve.

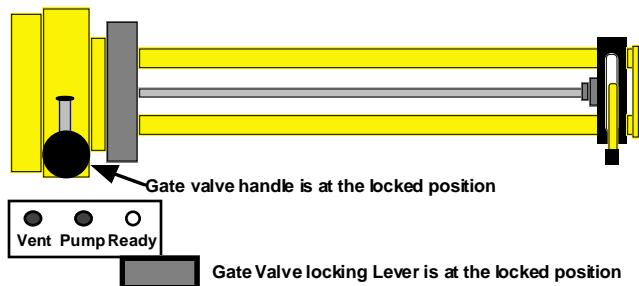


Close the gate valve by pulling the gate valve handle toward you until the gate valve locking lever closes again- this requires firm pressure.

NOTE: If the sample has slipped on the exchange rod during retrieval, it will partially block the gate valve from closing. DO NOT FORCE THE GATE VALVE!

To correct this problem, open the gate valve again, rotate the stage slightly, and push the sample on to the exchange rod fully by lightly bumping the sample holder against the stage. Again retrieve the sample and close the gate valve.

19. Push the red **Vent** button once and wait for the **Ready** light to flash. Now press the **Vent** button twice to completely vent the exchange chamber.



With the gate valve closed, the sample exchange chamber must be vented -- it is not left at vacuum since the vacuum system must return to column evacuation. The illuminated Ready light tells you that the vacuum system is still pumping on the sample exchange chamber.

Press the Vent button once (the Ready light will flash once). This tells the vacuum system that you are done evacuating the sample exchange chamber.

Press the Vent button TWO more times (this is necessary to fully vent the sample exchange chamber). The sample exchange chamber is not left under vacuum.

At the conclusion of the procedure, none of the lights are illuminated.

20. Decide now which working distance to use. If you have not been checked out for this operation DO NOT change the working distance; set it to 35 mm and leave it there. If you need to position the sample to a working distance less than 35 mm, **look through the viewport** and raise the sample using the Z-axis control while watching the proximity to the BSE and SEI detectors in the chamber.

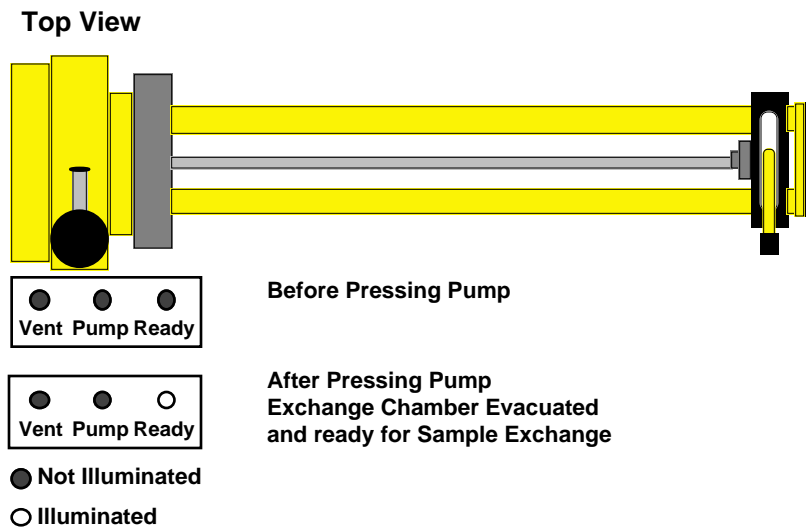
Note: Remember that 35mm is the working distance for X-ray analysis. If you need to do high resolution work (at a lower working distance) and X-ray analysis, set the working distance to a reasonable value and do your high mag work first. Then you can safely increase the working distance to 35mm and continue with X-ray analysis. Above all **Do Not change the working distance to a lower value without visually checking the clearance through the viewport!**

21. If you are doing x-ray work, position the Tracor X-ray detector to 4.0 mm (this is the correct position for the Tracor detector if you are doing quantitative analysis). If you just want to get x-ray spectra from your sample, you can set the detector to 5.0 mm.
22. Replace the chamber viewport cover and (gently!) turn fully clockwise to close the high voltage protection switch.
23. Leave the sample exchange device on the Camscan while you do your work.
24. Proceed now to Filament Saturation.

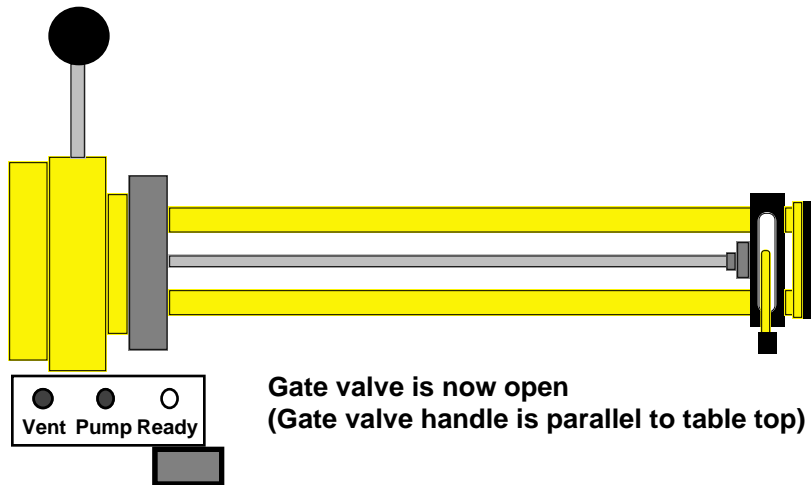
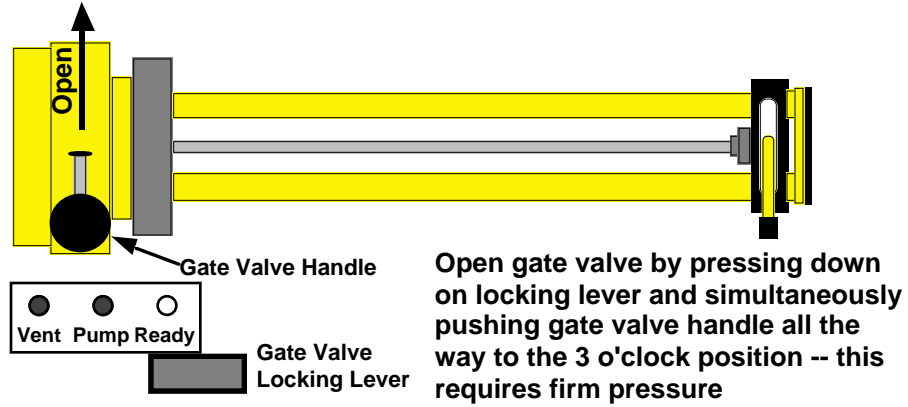
2 -- Removing a Sample from the SEM

1. The instrument must be set to the sample exchange coordinates before you can perform the sample exchange. You should do or verify the following:
 - **Z axis** set to 35.5 mm working distance using the Z-axis crank
 - **Rotation** set to 0° using the Rotation control
 - **Tilt** set to 0° using the Tilt control
 - **z' axis** (eucentric z) set to 5.6 mm using the z' axis control
 - **Magnification** set to 20 or whatever the minimum setting is
 - **X and Y stage odometers:** Drive the X and Y stage motors to 000 mm (same as 100) as follows:
If the odometer reads less than 50 drive **down** to 000 (i.e. from 20.5 drive to 000)
If the odometer reads more than 50 drive **up** to 000 (i.e. from 85.2 drive to 100 = 000)
2. To insert or remove samples, the high voltage must normally be turned off. Look at the orange "Image" button on Panel 1. If it is illuminated, high voltage is on. Turn it off by pressing the green "Ready" button next to it.
 - Press the green **Ready** button on Panel 1.
3. **Put on gloves.** You must wear gloves whenever handling samples or the exchange rod.
4. Rotate the **viewport cover** counterclockwise and take it off.
5. Evacuate the exchange chamber by pressing the blue **Pump** button, located below the gate valve lever. Wait for the green **Ready** light.

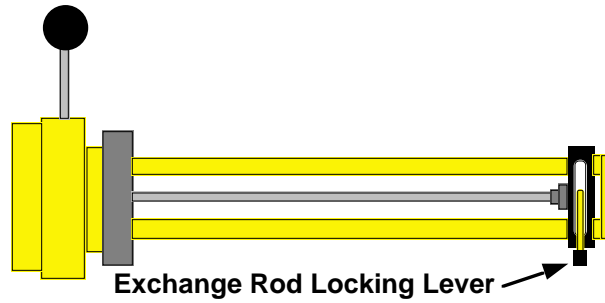
NOTE: This figure and the following ones have an error. The second box (lower) below should show that **both** the Pump and Ready lights are illuminated when the sample exchange chamber has been pumped down.



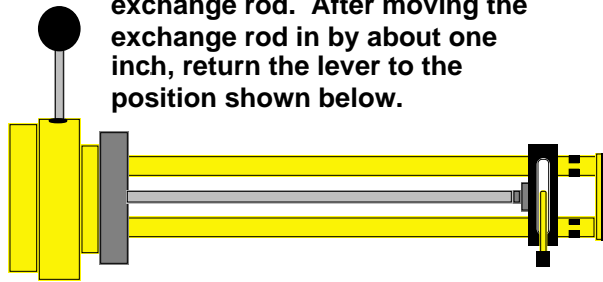
6. While watching the Camscan high vacuum gauge on the control console (Panel 1), slowly open the gate valve. Do this by simultaneously pushing down on the gate valve latch and pushing the gate valve away from you until the gate valve is horizontal. If you hear a beeping noise, you have not pumped down the exchange chamber; go back and do that now. The gate valve does not begin to open until the rod is past the 2 o'clock position, and the gate valve is completely open at the 3 o'clock position.



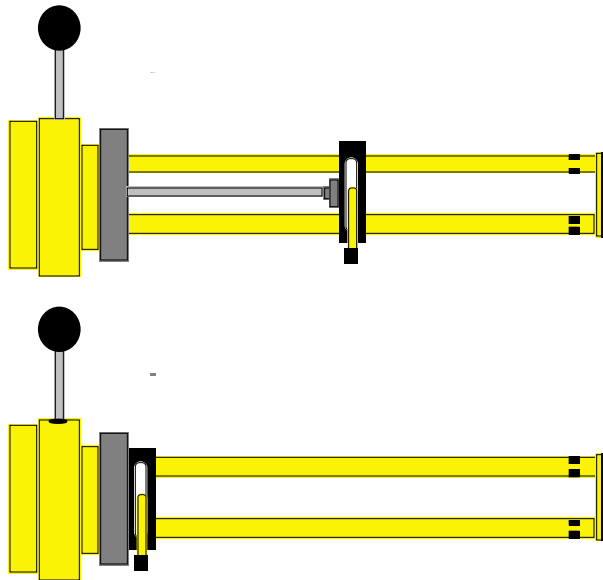
7. Release the exchange rod by lifting the locking lever out of the exchange rod's groove at the end.



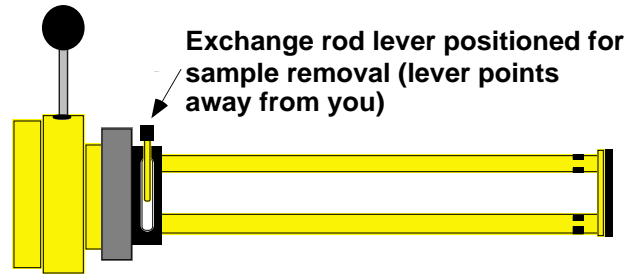
The exchange rod locking lever must be raised up out of the groove in order to unlock the exchange rod. After moving the exchange rod in by about one inch, return the lever to the position shown below.



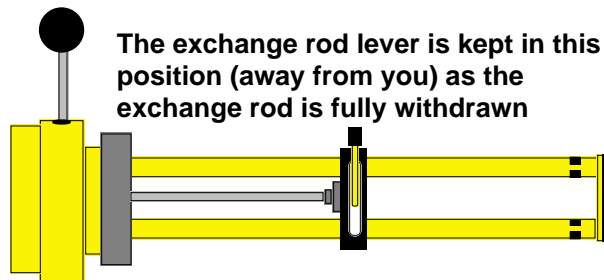
8. Verify that the locking lever is pointed toward you as shown above.
9. Slowly push the exchange rod all the way in while watching through the viewport.



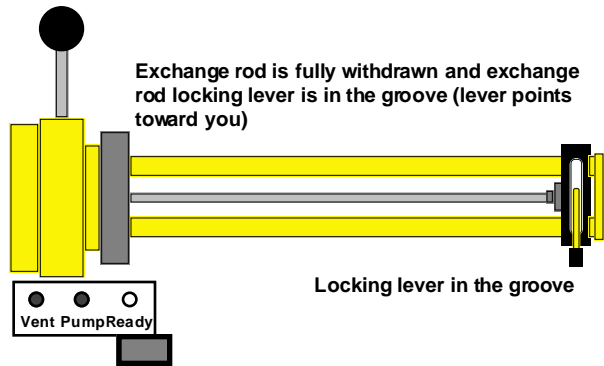
10. Engage the sample block by pushing the rod in until it is in contact with the sample and then push the locking lever away from you.



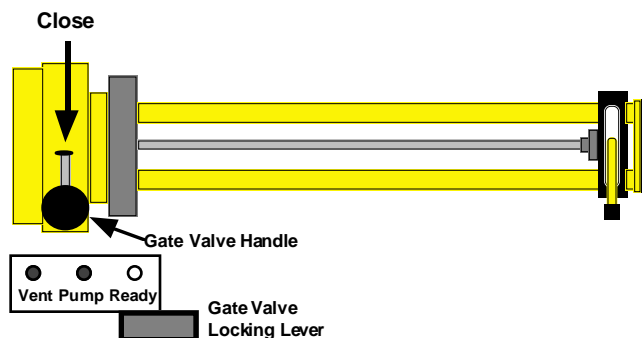
11. Withdraw the rod and sample by pulling the rod out. If the sample doesn't come out with the rod, you haven't properly engaged it. Hold the locking lever towards you while approaching and contacting the sample, and then move it away from you to grab the sample. If the sample appears to be too far on the stage to retrieve, withdraw the exchange rod and drive the Y stage odometer to 95.0 and try withdrawing the sample again.



12. When the exchange rod is fully withdrawn, put the locking lever back into the groove at the outermost end of the rod.



13. Close the gate valve by pulling it up and toward you until the gate valve latch locks again. Do not force the gate valve -- the sample may not be all the way on the exchange rod. If the sample is blocking the gate valve, it needs to be bumped onto the exchange rod all the way. Correct this problem by opening the gate valve again, rotate the stage slightly, and push the sample onto the exchange rod by lightly bumping the sample holder against the stage. Again retrieve the sample and close the gate valve.

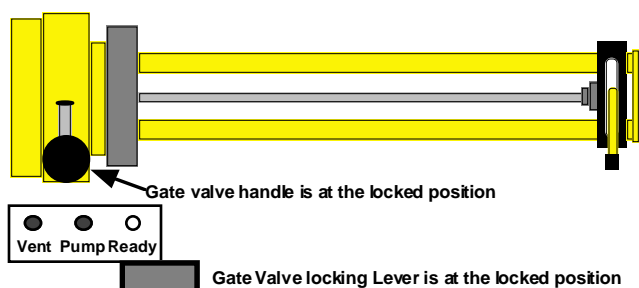


Close the gate valve by pulling the gate valve handle toward you until the gate valve locking lever closes again– this requires firm pressure.

NOTE: If the sample has slipped on the exchange rod during retrieval, it will partially block the gate valve from closing. **DO NOT FORCE THE GATE VALVE!**

To correct this problem, open the gate valve again, rotate the stage slightly, and push the sample on to the exchange rod fully by lightly bumping the sample holder against the stage. Again retrieve the sample and close the gate valve.

14. Push the red **Vent** button once and wait for the **Ready** light to flash. Now press the **Vent** button twice to completely vent the exchange chamber.



With the gate valve closed, the sample exchange chamber must be vented -- it is not left at vacuum since the vacuum system must return to column evacuation. The illuminated Ready light tells you that the vacuum system is still pumping on the sample exchange chamber.

Press the Vent button once (the Ready light will flash once). This tells the vacuum system that you are done evacuating the sample exchange chamber.

Press the Vent button TWO more times (this is necessary to fully vent the sample exchange chamber). The sample exchange chamber is not left under vacuum.

At the conclusion of the procedure, none of the lights are illuminated.

15. Replace the chamber viewport cover and (gently!) turn fully clockwise to close the high voltage protection switch.
16. Remove the sample exchange device from the Camscan and remove your sample from the sample holder.
17. If you have another sample to put in the instrument, follow the procedure for sample exchange, otherwise put the sample exchange device back on the instrument.

Filament Saturation and Column Alignment

This section contains the instructions for filament saturation and column alignment. You should have already put a sample into the instrument using the sample exchange procedure.

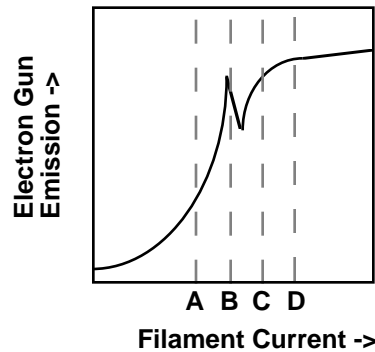
1. Verify that the vacuum has reached 10^{-5} torr on the **High Vacuum gauge** of panel 1. If you just performed a sample exchange and the vacuum is still above this level, wait a few minutes for the instrument to pump down.
2. Press the orange **Image** button on Panel 1 to turn on the high voltage power. If the button does not light up, either the vacuum is not high enough (green **Specimen Ready** light not on), or you have left the viewport cover off of the chamber or have not rotated the cover clockwise to close the high voltage protection switch.
3. The next step is to raise the filament current until the filament is saturated. You don't want to raise this current too high; if you do, you will burn out the filament or shorten its life.
4. The **filament current** setting used by the previous user serves as a guide. Look up this value in the log book and turn the **filament current** up **SLOWLY** to 0.20 microamps below that value (e.g. if the book says 2.41, go to 2.21). **SLOWLY** means turning the filament current knob not much faster than the speed of a second hand on a clock--this takes patience!
5. Raise the **Emission current** to 1 (=100ua).
6. Now you want to image the filament on the screen so you can see it while you saturate it. Turn up the **SEI** and **BEI** brightness and contrast controls until you see noise on the TV screens. Using the buttons on Panel 4, set the left screen to SEI and the right screen to BEI initially. (To change a screen from BEI to SEI or vice versa, you must push both the SEI and BEI buttons, to turn off BEI and turn on SEI; the buttons are toggles.)
7. Push in the **[•] Filament** button. This enables an imaging mode whereby you can see the electron beam crossover point directly below the electron gun. You should see a spot on the screen.
8. Push the **Photo linescan** button (next to the **[•] Filament** button). This allows you to look at a linescan profile through the electron beam crossover point. This profile is a slice through the filament brightness function. Adjust the contrast and brightness on the SEI detector to see most of the filament profile in the linescan.

NOTE #1: you may need to adjust the brightness and contrast several times during the saturation procedure in order to keep the filament profile image within the two vertical lines. If you set the brightness too high or too low, the profile will not be inside the lines. If you set the contrast too low you will not see enough detail of the profile, and if you set the contrast too high, the profile will lie outside of the vertical lines.

NOTE #2: The linescan profile mode will "time out" after awhile, and you will no longer see the signal (you will only see the two vertical bars that represent dark and 80% saturation). To return to the profile mode, first press the TV Scan Speed button (this returns you to a scanning image) and then press the Photo linescan button once more. Repeat as often as necessary.

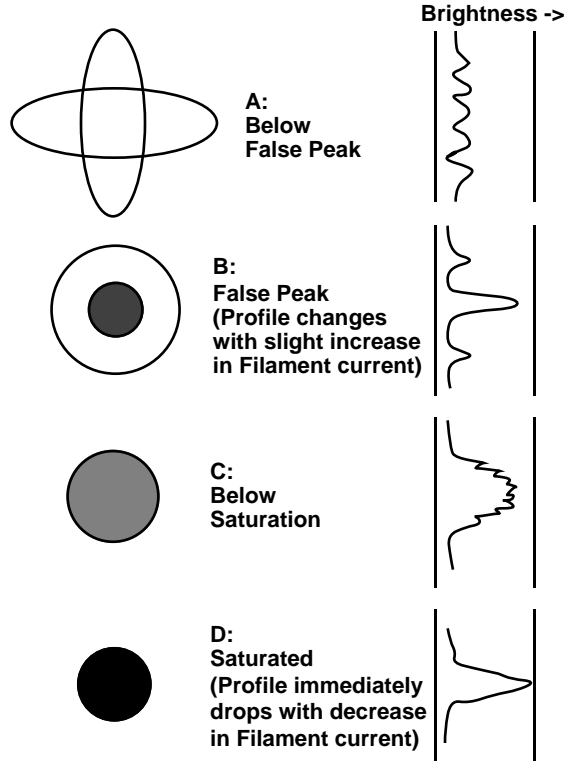
9. Slowly raise the **Filamen current** until you achieve a gaussian curve shape. Refer to the figure below to properly saturate the filament. Note that you can view the saturation sequence in the screen image mode (the fuzzy spot, not recommended) or by the profile image mode (recommended), which allows precise location of the saturation peak. Set the **Filament current** to the minimum value necessary to achieve this gaussian shape, and rock the filament current below and ever-so-

slightly above the saturation point to verify that you are at this minimum current setting for saturation. (When you have saturated properly, turning the **Filament current** down just a little will cause the curve to drop quickly. It is better to be slightly undersaturated than oversaturated.)



Screen Image

Line Profile Image



Filament Saturation Diagram

10. Turn off the [•] **Filament** button. Return to a scanning image by pressing the **Scan Speed TV**-rate raster display (Panel 5, lowest button). Set the magnification to the lowest value for the current working distance by turning the magnification and zoom knobs on Panel 5 fully counterclockwise.
11. Set the **Absorbed Current** meter to a scale range that displays current to x.xx format. Do not allow a number higher than 9.00 to be displayed (i.e., set the scale to a higher number to reduce the displayed value). It will probably be on 10^{-10} .

12. Adjust the **filament centering knobs** located on the front and right-hand side of the electron gun assembly on top of the column. Adjust these knobs one at a time to obtain the highest possible absorbed current on the absorbed current meter. Iterative adjustment is required. You should check this adjustment repeatedly while you do your work, every ten minutes while the machine warms up, and then every half hour or so.
13. Return to the console and check the filament saturation level again using steps 7-9. A slight dip in the filament profile may persist; do not oversaturate to overcome this. Now write in the log book the filament current value displayed next to [A] on the console. This level may need to be checked every 30-45 minutes if the filament was cold at startup.

Imaging, focusing, stigmatation

14. If you are using 35mm working distance, check that the COARSE FOCUS control is set to 35mm and displayed in the WD mm window. Set the coarse focus to the correct working distance if using a different WD. Once set, this control is not changed; all focusing is performed with the Z-axis control.

15. The FINE FOCUS knob is a ten turn adjustment. Rotate the knob until you reach a stop, and then rotate in the opposite direction five turns. This ensures that you are at a standard working distance.

16. Use the arrow buttons on the stage controller to locate a feature that has a sharp detail (cleavage, pits, etc.). Use the magnification and zoom controls to set the magnification high enough to see this feature -- typically 5000-10000x. Adjust the Z-axis control until this feature is as clearly resolved as possible. Coarse focusing is now performed with the Z-axis control for most flat samples.

17. Focus the feature by adjusting the X and Y STIGMATOR controls (iteratively) until you get the sharpest possible image. You will need to turn these knobs fairly quickly to see any effect. If adjustment of these controls does not visibly change the image, you must go to a higher magnification; conversely, if no adjustment works and the image is blurry no matter what you do, you are at too high a magnification.

18. You should now have a clear image. If there are problems, check the filament saturation, the electron gun alignment, the coarse focus (Z-axis), and finally the stigmators.

NOTE: DO NOT raise the sample unless you are absolutely sure that it will not hit the secondary or backscatter detectors. Lowering the sample is OK. Verify the clearance through the viewport at the time of sample exchange.

Digital Frame Store

The frame storage device is used to view the average of several rastered images on the left screen in order to cut down noise and see details more clearly. The frame storage has many different capabilities, but most users just use it for seeing a low-noise image. The instructions that follow tell you how to obtain a low-noise image. If you are interested in other features of the frame storer, consult the separate frame storer manual (1" binder).

1. Turn the frame storer on by turning the (unlabeled) knob on Panel 2 clockwise one notch. An "A" will be displayed right above the knob.
2. Push the "CONT" button in the lower lefthand corner of the frame storage keyboard. You will have to push this button again occasionally if the frame storer times out while you are taking a picture, doing an analysis, etc.
3. Adjust the number of averaged frames from 1 to 10 to get the image quality you like. A higher number results in a clearer image, but when you are moving around the sample with the stage controller arrow buttons, you may want a lower number so that the image will refresh itself fast enough for you to tell where you are on the sample. If you don't understand this, play with changing the number of averaged frames a little bit to see what it's like to drive around the sample with the frame storer on. Of course, the right screen is not an averaged image, so you can always watch there while you move around.

Photography

This section describes how to take polaroid photos of your samples. Not only do photos serve as a permanent record of the images of your samples, but in many cases you can see more detail in photos than you can on the screen because the noise level is lower and the resolution is better.

To take a photograph:

Set the focus and stigmatism at a magnification at least two times higher than the photo magnification.

Set the brightness and contrast while in Photo linescan mode (Photo linescan push button pressed in) so that the signal spans the central 2/3 of the brightness range.

Set the polaroid film holder to L for "load".

Insert the polaroid film with the number side up (and the side that says "This side toward lens" should be facing down) -- If you put the film in upside down it will eject developer jelly all over the inside of the film holder and YOU will clean it up. Push the film in until it is fully inserted.

Pull the film jacket out to expose the film inside the camera.

Press and HOLD the left blue Photo button until the fast scan has finished and the shutter to the polaroid camera has opened.

When the exposure has finished, the sem will return to a normal scanning image.

Push the film jacket back into the polaroid film holder all the way in.

Switch the polaroid handle from L to P for "print".

Pull the film pack all the way out WITHOUT STOPPING.

Use your fingers to help spread the developer all the way down to the corners (next to the metal strip) because the picture will not develop there unless it gets developer jelly over the full area of the print.

After the required time, open the film jacket and peel the print away from the other portion of the jacket.

Remove the print and throw away the used jacket (Do not stack the used jackets on any surface in the lab). Do not do any coating of Type 52 film in the lab!

1. Get a box of film ready. There is usually film sitting next to the camera which you can use, but you have to indicate how many photos you took in the log book. If you prefer, you can bring your own film (cheaper). Film can be purchased from various stockrooms on campus, including the VWR stockroom in Beckman.
2. Next you need to check the f-stop on the camera. Go around to the right side of the SEM table and stand by the camera. On the right side of the camera (as you view it) is a small silver knob that says "open" and "lock" with a silver dot next to "lock". Turn it counterclockwise to "open" (i.e. the word "open" is next to the silver dot).
3. Carefully lift the whole camera out of the hole it rests in. Try not to knock the camera against the table, and try very hard not to let dust or debris fall inside the hole. The screen that is used for photography is at the bottom of the chamber, and any dust that falls in there will land on top of the photo screen.
4. Check the f-stop setting. It should be at f-11 for Polapan 400 or f-18 (the dot between f-11 and f-22) for Type 53 film. Change it if necessary.
5. Carefully replace the camera. Turn the silver knob back to "lock" (i.e. the word "lock" is next to the silver dot).
6. Now you're ready to put film in. Using the arrows on the film as a guide, insert a piece of film into the slot on the front of the camera. Be careful to put the film in straight so that it can be pushed all the way in fairly easily. The film should go in until less than an inch remains visible.

7. Now pull the film back out gently until it stops. The film is now ready for you to take the picture.
8. Check that the screen selection button marked with "L" and "R" is on "L," and get the image you want to photograph framed and focused on the left screen.
9. Check that the photo scan speed selector is on "2" (slow, scan takes ~2 minutes).
10. If you want to type in a label, do so with the keyboard next to the camera.
11. Now you need to set the brightness and contrast for the camera. The camera has a different display tube from the screens, so the brightness and contrast you see on the screen may be a little different from the camera. Push the "photo" button on Panel 5 to get a linescan of the brightness and contrast the camera sees. (This is the same photo button you pushed a while ago to get a linescan of the filament during the saturation procedure.)
12. You should now have a fuzzy linescan on the right screen. Adjust the brightness and contrast knobs until the fuzzy line is just a bit to the right of the center of the two solid lines and is about 1/4" wide on the screen.
13. Now push the "L" button and hold it in for 10 seconds before letting go. The slow linescan will begin after a moment. Wait for the regular image to come back, which will take about 2 minutes.
14. When the slow scan is over, push the film all the way back into the camera again.
15. Push the lever arm on the front of the camera to "P."
16. Pull the film straight out of the camera. You may have to pull firmly at the end to get the spine of the film out.
17. Wait about a minute (or the length of time specified on the film or film box), then grasp the two tabs on the film and pull them apart to reveal the picture. The gooey chemicals inside the film are not particularly good for you, so avoid touching them if possible, and refrain from eating them.
18. If you are unhappy with the result, you can try again with different brightness and contrast settings, better focus, etc. Keep in mind, however, that the photos are not cheap!

Shutdown

You say you've had enough? This section will tell you what to do to finish up and how to leave the instrument when you're done.

1. Slowly turn the filament current down to 0.00.
2. Turn the emission current down to 0.
3. Turn off the high voltage by pressing the green "READY" button on Panel 1.
4. Set the Link EDS detector to 10 cm (withdrawn position) if it is at the analysis position of 6 cm.
 - 4.a. Set the magnification to 20X.
5. Move the sample stage back to the sample exchange position ($Z=35.5$, $X=Y=000$, etc.) and remove your sample using the instructions in the section on sample exchange.
6. **Return all controls to their default positions as listed in Section I.**
7. Turn off the console power by pressing the yellow "CONSOLE" button on Panel 1.
8. Finish filling out the log book and the charge sheet.
9. Clean up the area.