

## Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

### Drexel Zeiss SUPRA 50VP

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*These notes are meant to serve as an aid to assist users who have been trained and certified by MCC Staff. If ever you are unsure about the correct operation of the instrument or any of its components, please consult a MCC staff member before continuing. Never use equipment that you are not trained and approved to use.*

*Before using the MCC, please review the MCC User Handbook available through our website.*

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## OPERATING GUIDE FOR SmartSEM SOFTWARE CONTROLLED SEM'S

### INTRODUCTION

Users trained on the ZEISS SUPRA 50VP FESEM are assumed to have read this document prior to beginning their training.

- The SUPRA 50VP has a joystick controlled, motorized stage with motorized tilting up to 90 degrees.
- The SUPRA 50VP has an infrared CCD camera system for visualizing the sample-lens positioning.
- The SUPRA 50VP has a non-mechanically adjustable aperture.
- Aperture centering is accomplished electronically through the SmartSEM software.
- The SUPRA 50VP has a thermally-assisted, Schottky Field Emission Gun that is left heated at all times.
- The EHT is toggled ON/OFF at the selected kV by the user.
- There is no user alignment of the gun. Novice users are prohibited from accessing the gun functions.
- The SUPRA 50VP has automatic isolation valving that protects the gun from exposure to the atmosphere while venting the sample chamber. The gun vacuum should always be in the  $10e-10$  mBar range, at vacuum, the system is typically in the  $10e-5$  mBar range. Chamber isolation valve opens at  $1.6 \times 10^{-5}$  torr.

The SmartSEM controls are displayed on screen in graphical form. These controls can now be easily customized to suit the needs of individual users.

The graphical controls are operated through the actions of a standard computer mouse and keyboard. The role of the computer is to translate these actions into SEM commands or parameter adjustments. (Example: Select some function (Center Point), or adjust Focus / Magnification.)

The image is formed in a separate digital image processor that is also controlled through the computer-graphical interface. The image is merged with the graphical controls. The result is a large screen display of the image with operating controls conveniently located at the top and bottom of the screen.

The following steps to learning the system are recommended:

1. Familiarize yourself with the LOGON, START-UP and CLOSE-DOWN procedures.
2. Familiarize yourself with the SAMPLE EXCHANGE procedure.
3. Practice using the interface and mouse to control parameters.
4. Adjust aperture alignment and correct astigmatism at different kVs and working distances.
5. Familiarize yourself with acquiring images and sending these to a file or hardcopy.

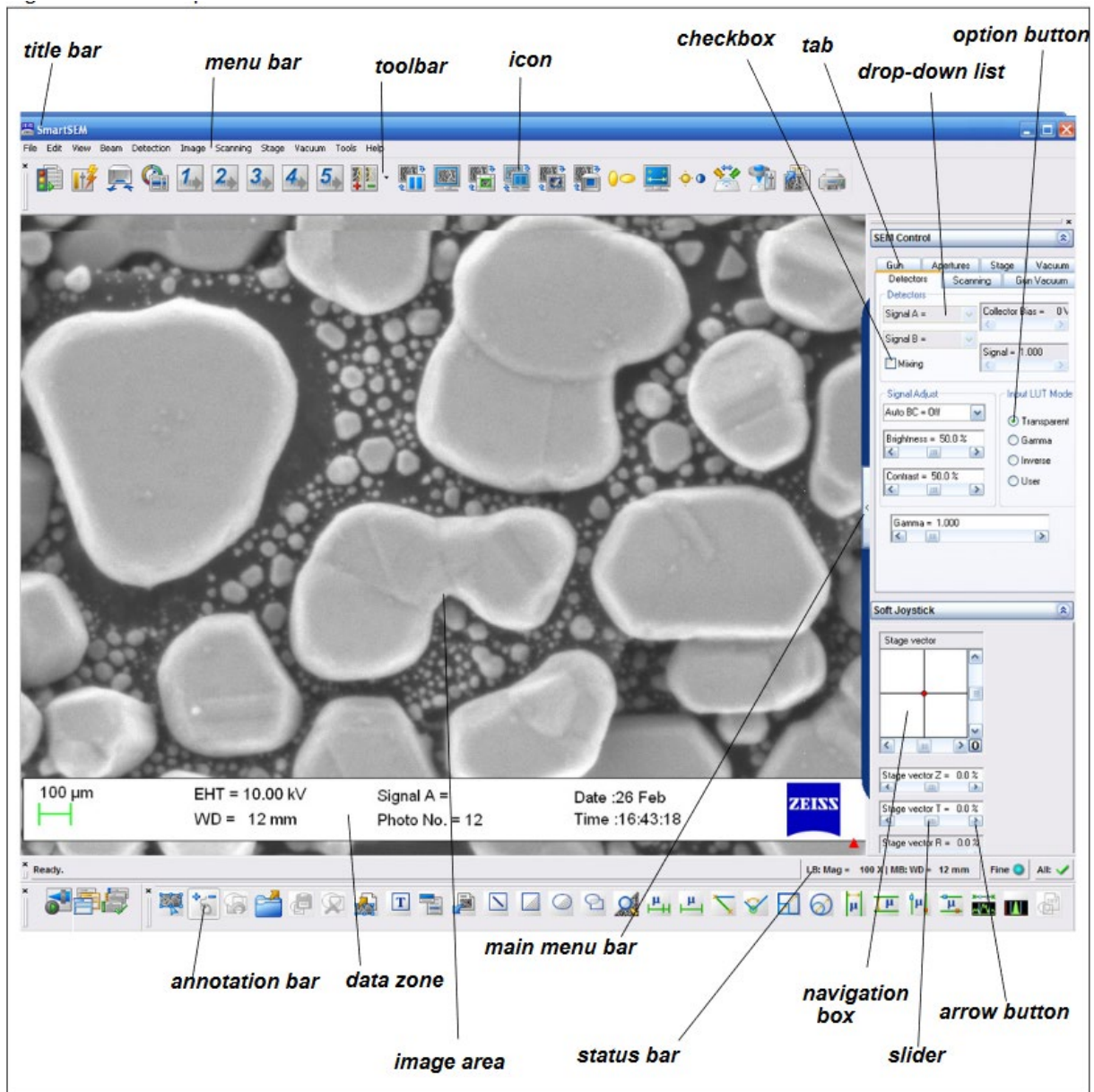
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## Training Library – Standard Operating Procedures

This operating guide will assist you with the familiarization of the user interface, and basic SEM operations. This contains excerpts and images from the comprehensive on-line HELP. The on-line HELP is an excellent reference for the more advanced features. F1 is the keyboard shortcut to SmartSEM HELP.

### THE SCREEN DISPLAY

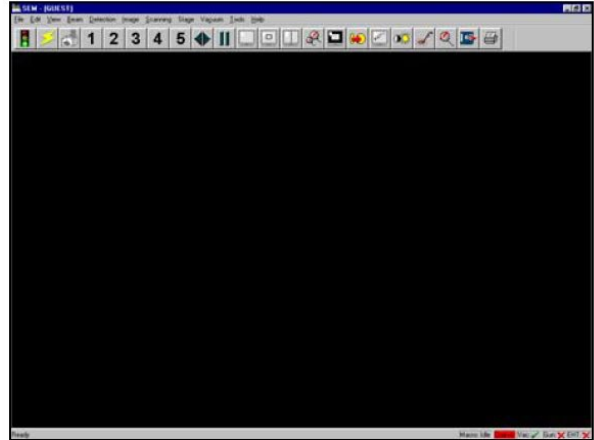
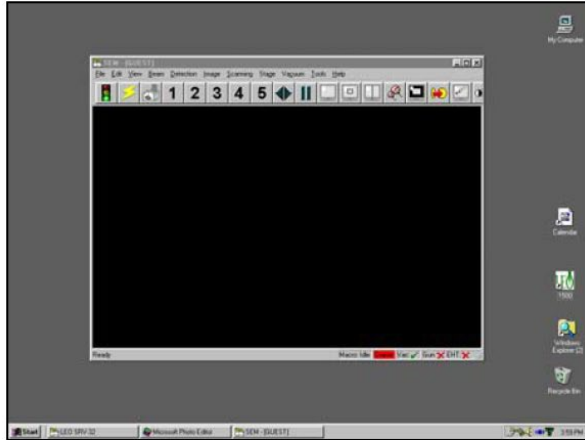
#### SmartSEM user interface



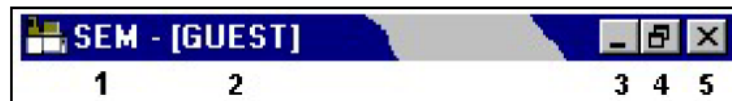
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# Materials Characterization Core at Drexel University

## Training Library – Standard Operating Procedures

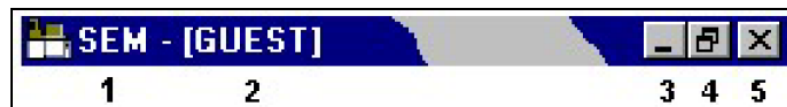


After logon the screen should appear as above. If the user interface is not at full size, select the middle icon (4) in the extreme upper right corner of the top window caption bar.



### COMPONENTS OF THE SCREEN DISPLAY

- o Top Window / Caption Bar
- o Menu Bar
- o Hard Panel Status Bar (Optional, if Hard Panel is fitted)
- o Toolbar
- o Image area
- o Status Bar

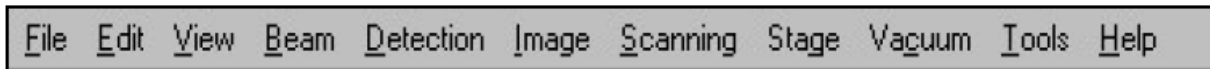


The Caption bar displays on the left side:

1. User Interface Application Name (SmartSEM)
2. Current User Name (e.g. GUEST)  
On the right side of the Caption bar the usual Windows buttons are displayed
3. Minimize button enables the application to be iconised while leaving the SEM operational.
4. Reduce / Maximize display button.
5. The close button (5) to terminate the application.

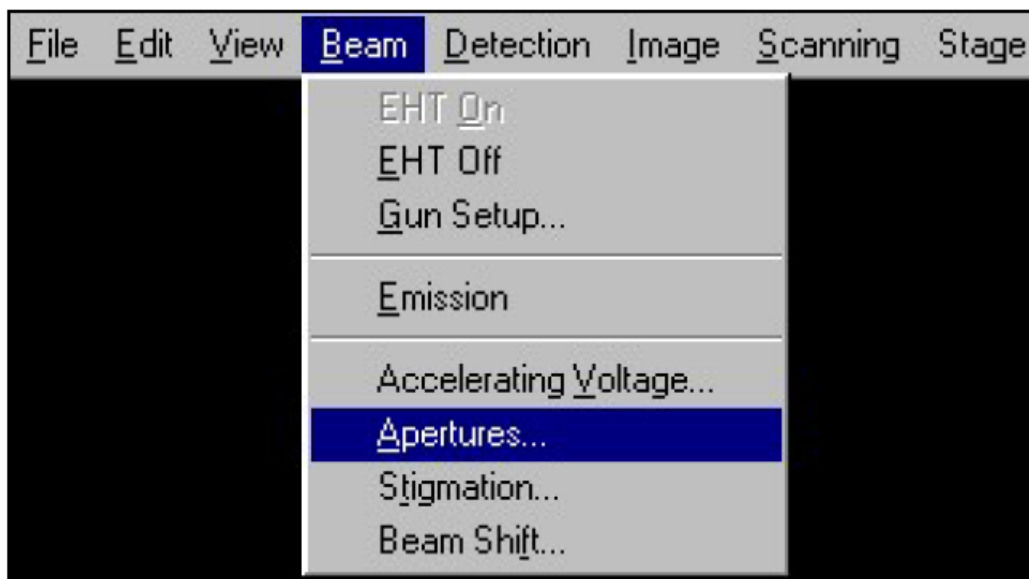
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### MENU BAR

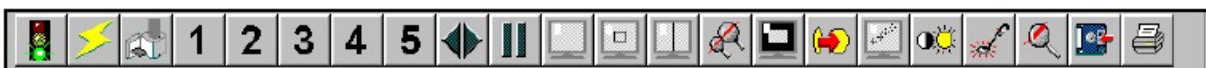


Beneath the Caption bar is the Menu Bar. All accessible functions of the SEM software are displayed in drop-down menus. If functions are not readily available they are dimmed.

Drop-down menus in the Menu Bar can be called by placing the cursor on the menu bar and then clicking with the left mouse button.



### TOOLBAR



The Toolbar is displayed at the top of the Image Window (or elsewhere, if the user so desires) and provides easy access to commonly used, user defined, functions.

Note: This may not be the same toolbar that you will see during your login session.

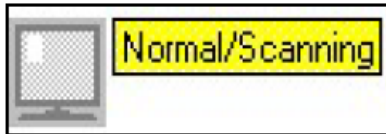
If not displayed, it is switched on from the Menu Bar with View, Toolbars, and then select User Toolbar from the Toolbar Views panel. Other toolbars typically activated are Status Bar, Annotation Bar and Docking Panel.

## Materials Characterization Core at Drexel University

### Training Library – Standard Operating Procedures

The Toolbar contains Icons, which may be selected with the LEFT or MIDDLE mouse buttons to perform required operations. You may find non-functioning toolbar items. These can be overridden by selecting the corresponding functions from the SEM Control panel. Additional functions are available in the pull-out menu to located at the left side of the docking panel.

Tool Tips are available displaying information about the function behind an icon.



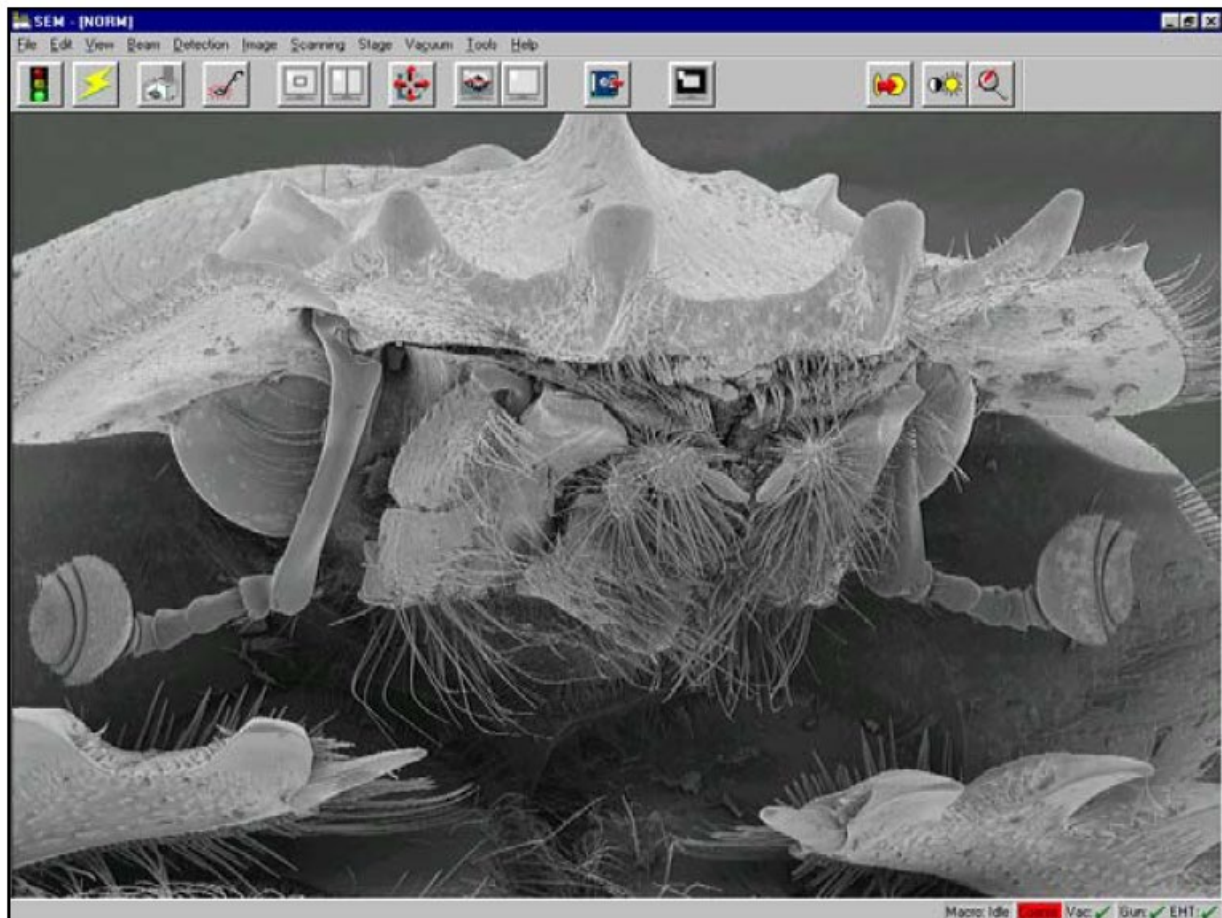
The Icons and corresponding actions may be edited using the Toolbar Editor. This enables users to customize their SEM controls.



The specific operation of these icons may have been customized and may not conform to the suggested uses as detailed in SmartSEM HELP. (F1 keyboard shortcut)

Different sets of icons and actions may be loaded according to the application requirements and operator skill level. Contact Kate Vanderburgh for information on how to customize your toolbar.

## IMAGE AREA



The Image area becomes active when a detector is on.

While the mouse cursor is in this area, and if adjustable parameters are assigned to the mouse LEFT and MIDDLE buttons (Refer to: PARAMETER ADJUSTMENT WITH THE MOUSE), the selected parameter will be adjusted when the appropriate mouse button is held down while dragging the mouse.

- Text annotation, SEM conditions and measurements can be placed in the image area and may be saved with the image if "Save Annotation" is checked. Annotations can also be saved and recalled during future sessions by pulling down the File menu and selecting the Save/Load Annotation functions.

## STATUS BAR

The Status Bar is shown at the bottom of the Image Window if it is enabled.

The Status Bar is switched on as follows:

- Select View - Toolbars with the mouse LEFT button from the Menu Bar
- The Toolbar Views menu pops up and then select the Status Bar.

Or

- Type <CTRL + B> to access the Toolbar View pop up menu and then select the Status Bar
- Click with the left mouse button on the Toolbar if it is activated. Select Status checkbox.
- 

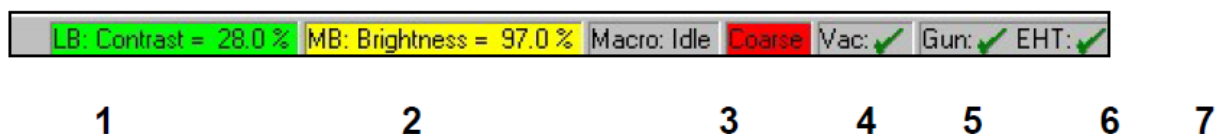
The left side of the Status Bar shows either results or hints:



If a dynamic process is under way a progress bar is displayed:



The right side of the Status Bar displays parameters, (1 & 2), which are controlled by the mouse when the cursor is in the Image Window. (EM Mouse Control must be selected at bottom left. Values displayed may be absolute, (WD=5mm), or relative, (Contrast = 28%). Values expressed in % indicate the amount of the available control used. (Range 0 to 100%)



The status of macros is displayed (3). If no macros are executing, the state is idle. If a macro is running, the macro number is displayed.

Zeiss Supra GETTING STARTED  
ZEISS\_SUPRA 50VP\_2011rev2.doc 7

When adjusting parameters with the mouse or arrow keys the increments may be COARSE or FINE. The current state of the coarse /fine is indicated in the Status Bar (4).





## Materials Characterization Core at Drexel University

### Training Library – Standard Operating Procedures

The state may be toggled using the <TAB> key or by clicking on the status indicator with the cursor and a left mouse button click.

Vacuum status and control (5), provides a visual reference of the current vacuum status and the ability to carry out manual operations of the specimen chamber vacuum.

Gun status and control (6), provides a visual reference of the current electron gun status and the ability to carry out manual operations of the electron gun.

EHT (Extra High Tension), (kV, Gun Voltage, Accelerating Potential, Beam Voltage), status and control (7), provides a visual reference of the current EHT status and the ability to carry out manual operations of the EHT.



A **GREEN** check indicates an ON or READY state.



A **YELLOW** check with a **RED** slash indicates a state between ON or READY, and OFF or NOT READY. (Gun running up or down, Vacuum pumping down).



Crossed **RED** slashes indicate a state of OFF or NOT READY.

Selecting these fields with the LEFT mouse button will present a pop-up dialog with available manual control options. (VENT or PUMP, GUN / EHT ON or OFF (SHUTDOWN)).

NOTE! Gun shutdown (OFF) is a privileged control and is not available to all users. If the entire system is shutdown using the **RED** front panel button, the gun will shut off. It can be re-started once vacuum is reestablished at the correct level for the gun to operate (10e-10 torr range).

### PARAMETER ADJUSTMENT WITH THE MOUSE

There are 2 modes that the mouse operates under:

1. Parameter Adjustment (EM Mouse Control)
  - a. In this control mode the mouse is used for parameter adjustment. The parameters assigned to the LEFT and MIDDLE mouse buttons are shown in the Status Bar at the bottom right of the display.
  - b. Different parameters may be assigned such as:
    - i. Magnification and Focus (WD)
    - ii. Brightness and Contrast
    - iii. Stigmation X and Y
    - iv. Beam (Image) Shift X and Y
    - v. Aperture Alignment X and Y
  - c. Various actions will assign these functions.
  - d. The most common is by selection of an icon. (See Toolbar Operation.)
2. Annotation (Select Annotation).

Select these settings at the far lower left of the bottom toolbar. While the mouse cursor is in the image window, parameter adjustment is available.

### Dual joystick

- Used for stage control and specimen navigation
- 2 joysticks on the dual joystick panel
- Right joystick (larger) primarily controls specimen stage movement in the x & y directions. It can also rotate the specimen stage
- Left joystick controls specimen stage movement in the z direction
- The 'Break' button in the top middle of the joystick panel will stop all movements of the specimen stage.



## Control Panel



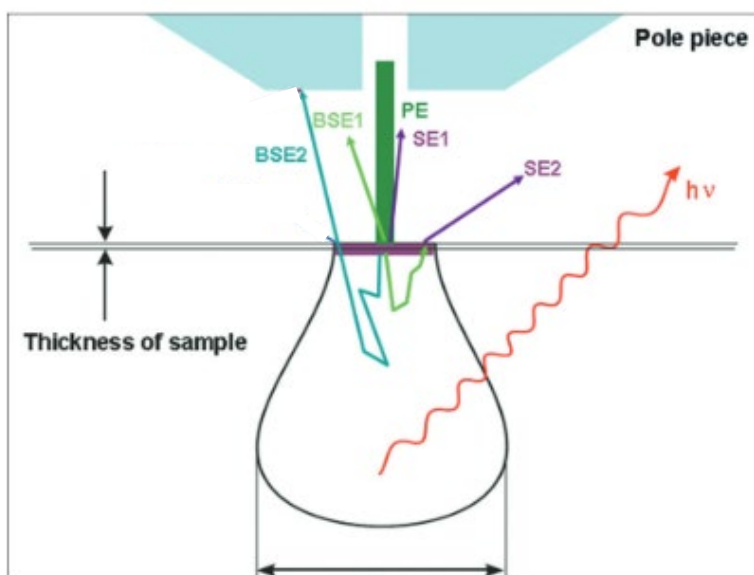
- Magnification
- Stigmator X
- Stigmator Y
- Aperture X
- Aperture Y
- Scan rotate
- Shift X
- Shift Y
- Brightness
- Contrast
- Focus

### Push buttons:

- Reduced
- Wobble
- Freeze
- Exchange
- Resume
- Camera
- Scan speed +
- Scan speed –

## Detectors

Detector	Detected signals	Typical application
In-lens	SE1	Surface structure
SE2	SE2	Topography and surface structure
Backscattered	BSE	Topography and compositional contrast



Huang, Jason, Timothy Cavanaugh, and Boaz Nur, 2013, An introduction to SEM operational principles and geologic applications for shale hydrocarbon reservoirs, in W. Camp, E. Diaz, and B. Wawak, eds., Electron microscopy of shale hydrocarbon reservoirs: AAPG Memoir 102, p. 1–6

PE: primary electrons

SE1: type I secondary electrons – emit at a high angle at close proximity from the impact point with the surface of the specimen

SE2: type II secondary electrons – reflect at a lower angle, generated from deeper and wider volume compared to SE1

BSE: backscattered electrons – emit at a high angle and commonly used to characterize composition and crystalline structures