

Electron Microprobe Users Manual

Windows-Based Software for the Cameca SX50

Part 1. Basic Microprobe Setup and Sample Exchange

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TYPOGRAPHIC CONVENTIONS

The following typographic conventions have been used:

BOLD CAPITALIZED - indicates the name of a window or icon.

UNDERLINE CAPITALIZED - indicates a software button, field, or menu choice that the user needs to select or enter data into.

• Message: **Bold** - indicates an instruction that appears on the computer screen

Action: - The response you should make to an instruction from the computer.

R-Click – Indicates that you should click on the Right mouse button

L-Click - Indicates that you should click on the Left mouse button

GENERAL NOTES ON MICROPROBE SOFTWARE

USING THE MOUSE

R-Click: In the current software the clicking the right mouse button (R-Clicking) on a button, icon, or underlined field usually causes a pop-up menu to appear from which you can choose various options for that field.

L-Click: In the current software the clicking the left mouse button (L-Clicking) on a button, or icon usually selects the default choice for that option. The default choice may or may not be shown on the button or icon. In some cases L-Clicking is also used to toggle a button between two different states.

The middle mouse button is not used in the main SX50 software. However, it is used in some cases in the PGT-IMIX EDS software.

Recommendation: If you are in doubt about which button to press, try R-Clicking first. If there are no menu options available nothing will happen. If there are menu choices available you will be allowed to select the one rather than getting a choice you may not have wanted by default.

STARTING THE SOFTWARE

- 1) Log-in to the sx directory (see me for login name and password).
- 2) Wait for the blue (Openwindows) Sun screen to appear.

Probe setup, WDS, and Imaging

- 1) R-Click on the blue Sun screen background and select SX from the pop-up menu.
- 2) Software loading is complete when the **SETUP** screen displaying a schematic picture of the microprobe appears. This takes about 1 minute.

EDS

- 1) R-Click on the blue Sun screen background and select PGT-IMIX from the pop-up menu.

- 2) The EDS display window will appear after about 20 seconds.
- 3) R-Click on the MENU button to select action.
- 4) To quit, R-Click on the top of the window and select QUIT from the pop-up menu.
- 5) L-Click CONFIRM in the pop-up window.

RESTARTING THE SOFTWARE

It is occasionally necessary to restart the SX50 software if it is not running correctly. The procedure for this is:

- 1) Close any open sub-windows or programs.
- 2) Make Sure the CUP is ON.
- 3) R-Click on the top of the gray SX50 menu bar and select QUIT from the pop-up menu.
- 4) L-Click YES.
- 5) R-Click on the blue Sun screen background and select PROCESS CONTROL from the pop-up menu.

The CAMECA PROCESS UTILITY should appear.

- 6) L-Click on the all processes in the process list. They should turn dark when selected.
- 7) L-Click on DELETE PROCESS to remove the process from the list.
- 8) R-Click on the top of the CAMECA PROCESS UTILITY window and select QUIT from the pop-up menu.
- 9) R-Click on the background of the blue sun screen and select SX from the pop up menu.
- 10) Wait for the SX50 software to appear (takes about 2 minutes).
- 11) See if the program now operates correctly.

TYPES OF WINDOWS

There are 2 types of windows in the software. They need to be opened and closed differently.

- 12) Windows that are opened by clicking on the icons in the gray **SX50** menu bar (i.e. SETUP, QUALI, IMAGE, TASKS, and TOOLS) and any associated sub-windows should always be closed by R-clicking on the original icon button and selecting EXIT APPLICATION. This is true even if the window offers a done option button (like Quantitool). Closing these windows in any other way will leave a new copy of the window application running in memory and will degrade system performance.
- 13) Most windows that are opened by L-Clicking buttons in the SX50 SETUP, STAGE, and ROLLER WHEEL windows can be closed just by L-Clicking on the original button, L-Clicking on the pin in the upper left corner of the window, or R-clicking on the top border and selecting DISMISS from the pop-up menu.

If in doubt about the type of window, R-Click on the top border of the window and look at the pop-up menu. If the first menu selection is not DISMISS, then the window needs to be closed by the method described above).

TYPING IN VALUES FOR SETTINGS

Some settings allow you to also manually enter the values you want (these field usually have a gray underline beneath them. To do this:

- 1) Move the mouse to the field you want to change.
- 2) Delete the value that currently appears there (use the Delete key or highlight the entry and press the Cut button).
- 3) Type in the value you want. Typically you must start typing from the farthest left position on the line for the command to work.
- 4) Press the <return> key.

Note: You have to end all entries with a <return> for the values you type to take effect.

SHUTTLE EXCHANGE PROCEDURES

SAMPLE CHANGE - SHUTTLE EXTRACTION

- 1) L-Click on STANDBY in the main **SETUP** window to turn off the electron beam.
- 2) L-Click on the VACUUM button located in the main **SETUP** window.

The **VACUUM SETUP** window should appear.

- 3) L-Click on the SAMPLE EXCHANGE button in the **VACUUM SETUP** window.
- 4) L-Click YES to confirm.

Follow the instructions in the **VACUUM SETUP** window.

Messages appear in black letters on a white background in the space below the words “Pressure Units: Pa”. When the airlock vacuum is ready, additional, messages appear in the gray area at the top of **VACUUM SETUP** window. Always wait for the appropriate message from the machine before performing any action.

- Message: **Turn the Gun Valve to Position 1**

Action: Manually turn the gun valve knob (large black knob with numbers located behind the column) fully clockwise to position 1 (i.e. the number 1 on the knob pointed to the left).

- Message: **Gun Valve Ready**

Action: Wait.

- Message: **Airlock backup complete**

Action: Wait.

- Messages: **Open Airlock Gate Valve
Move In/Out Shuttle
Close Airlock**

Appear in the gray area at the top of the **VACUUM SETUP** window.

Action: Proceed with Step 5.

Important: DO NOT open the sample chamber door unless these three messages are present in the gray upper portion of the VACUUM SETUP window.

- 5) Rotate the ring located on the transfer rod to the Extraction position (white arrow pointing towards you).
- 6) Open the sample chamber door (the door between the airlock chamber and the sample chamber) by pushing the black lever at the left of the airlock in and up. Note that the lever locks in position and rotates slightly.
- 7) Push the transfer rod all the way in and then pull it out again. The shuttle should appear on the prongs in the airlock chamber.
- 8) Close the sample chamber door (rotate the black lever and swing it down). Note that the lever locks and moves out slightly.
- 9) L-Click CONTINUE. (The airlock will be automatically vented and the sample chamber is pumped down to vacuum again).
- 10) Wait until the airlock housing is vented. Press the black release button on the left side of the airlock and swing open the airlock housing. Remove the shuttle from the transfer rod. Close the airlock housing afterwards.

Note: If you do not perform a sample change within a certain length of time the message will appear:

- **Message: Turn the Gun valve to Position 2.**

Action: Manually turn the gun valve all the way counterclockwise to Position 2. (i.e. the number 2 on the knob pointed to the left).

- 11) If you are not inserting another shuttle, close the **VACUUM SETUP** window by L-Clicking on the VACUUM button in the **SX Setup** window.

SAMPLE CHANGE - SHUTTLE INSERTION

- 1) Press the small black release button on the left side of the airlock and swing open the outer airlock housing and place the shuttle on the prongs of the transfer rod. The shuttle should click into position. Close the outer airlock housing.
- 2) Rotate the ring located on the transfer rod to the Insertion position (white arrow pointing towards the machine).
- 3) If the **VACUUM SETUP** window not open, L-Click on the VACUUM button located in the **SX SETUP** window.

The **VACUUM SETUP** window should appear.

- 4) L-Click on the SAMPLE EXCHANGE button.
- 5) L-Click YES to confirm.
- 6) Follow the instructions displayed in the Vacuum Setup window. Messages appear in black letters on a white background in the space below the words “Pressure Units: Pa”. When the airlock vacuum is ready, additional, messages appear in the gray area at the top of **VACUUM SETUP** window.

- Message: **Turn the Gun Valve to Position 1**

Action: Manually turn the gun valve knob (large black knob with numbers located behind the column) fully clockwise to position 1 (i.e. the number 1 on the knob pointed to the left).

Note: This message only appears if the gun valve is not already at position 1 (i.e. when you are exchanging one shuttle for another).

- Message: **Gun Valve Ready**

Action: Wait.

- Message: **Airlock backup complete**

Action: Wait

- Messages: **Open Airlock Gate Valve**
Move In/Out Shuttle
Close Airlock

Appear in the gray area at the top of the **VACUUM SETUP** window.

Action: Proceed with Step 7.

Important: DO NOT open the sample chamber door unless these three messages are present in the gray upper portion of the VACUUM SETUP window.

- 7) Open the sample chamber door (the door between the airlock chamber and the sample chamber) by pushing the black lever at the left of the airlock in and up. Note that the lever locks in position and rotates slightly.
- 8) Push the transfer rod all the way in and then pull it out again. The shuttle should remain in the sample chamber.
- 9) Close the sample chamber door (rotate the black lever and swing it down). Note that the lever locks and moves out slightly.
- 10) L-Click CONTINUE. (The airlock will be automatically vented and the sample chamber is pumped down to vacuum again).
- 11) Wait for the message:

- Message: **Turn the Gun valve to Position 2.**

Action: : Manually turn the gun valve all the way counterclockwise to Position 2. (i.e. the number 2 on the knob pointed to the left).

Note: The time it takes for the vacuum to get to this point will vary depending on the sample. In general it should take 2 to 5 minutes.

- 12) Wait for the message:

- Message: **Vacuum Ready**

Action: Proceed with step 13.

- 13) Close the **VACUUM SETUP** window by L-Clicking on the VACUUM button in the main **SETUP** window.

MICROPROBE SETUP PROCEDURE

A) TURN ON THE OPTICAL MICROSCOPE LIGHT

- 1) L-Click on the O button located in the **SETUP** window.

The **OPTICS SETUP** window will open.

- 2) L-Click on the Light ON button.
- 3) Close the **OPTICS SETUP** window by L-Clicking the O button in the main **SETUP** window.

B) VERIFY THE STAGE POSITION

- 1) L-Click on the STAGE button located in the main **SETUP** window.

The **STAGE** window will open in the lower left corner of the screen.

- 2) L-Click on the STAGE DISPLAY button in the **STAGE** window.

The **Stage Display** sub-window will appear.

- 3) R-Click on the REFERENCE in the **Stage Display** sub-window and select MOVE STAGE TO REFERENCE from the pop-up menu.

The stage should move to the position $(X = Y = Z) = 0$.

- 4) R-Click on the ROLLER CHOICE button in the **Roller Wheel Control** window (located in the lower right portion of the computer screen) and select STAGE from the pop-up menu to activate the roller wheel stage controls.
- 5) Focus the optical image and move the stage using the X, Y, Z roller wheels so the crosshairs are centered on the shuttle reference mark.
- 6) R-Click on the REFERENCE button in the **Stage Display** sub-window and select and select SET STAGE REFERENCE from the pop-up menu.
- 7) L-Click YES to confirm.

The new stage position will be set to $(X = Y = Z) = 0$.

- 8) L-Click again on DISPLAY in the **STAGE** window to close the **Stage Display** sub-window.

C) SETUP THE STANDARD BLOCK and MOVE TO THE BENITOITE STANDARD

- 1) L-Click on the STAGE CONTROL button in the **STAGE** window.

The **Stage Control** window will appear.

- 2) R-Click on POSITION in the **Stage Control** sub-window and select 0 from the pop-up menu.
- 3) R-Click on BLOCK in the **Stage Control** sub-window and select 0 from the pop-up menu. Continue to hold the right mouse button down and wait for the list of standard names to appear in the pull right menu. R-Click on BENI from the pull right menu (the first entry in the list).
- 4) L-Click on the MOVE button in the **Stage Control** sub-window.

The stage should move to the benitoite standard.

- 5) R-Click on DONE to close the **Stage Control** sub-window. Leave the **STAGE** window open in the lower left hand corner for future use.

D) START THE ELECTRON BEAM

- 1) Make sure the Faraday Cup is on. L-Click on CUP ON in THE **ROLLER WHEEL CONTROL** window.
- 2) L-Click on the SETUP button located in the upper part of the main **SETUP** window.
- 3) L-Click on the desired Setup file (usually HV15) in the pop-up menu to highlight it.
- 4) L-Click on LOAD.
- 5) Close the pop-up menu by L-clicking on the SETUP button in the main **SETUP** window.
- 6) Wait for the message:

• Message: **Setup Ready**

to appear in the pop-up status window, just below the gray SX menu bar.

Note: The HV and Beam Current (15 keV, 20 nA for setup HV15) should appear in the upper part of the SETUP window.

- 7) Make sure the Faraday Cup is on (it can sometimes turn itself off during setup). L-Click on CUP ON in the **Roller Wheel Control** window.
- 8) L-Click on the COLUMN button in the main **SETUP** window.

The **BEAM SETUP** window will appear.

- 9) R-Click on BEAM SIZE and select OFF (if not already selected).
- 10) Close the **BEAM SETUP** window by L-Clicking on the COLUMN button in the main **SETUP** window.

E) SELECT THE DESIRED VIDEO MODE

Note: If the video mode indicated on the TV monitor (i.e. SE or BSE) is already what you want, skip to step 6.

- 1) L-Click on SCAN ON in the **Roller Wheel Control** window.
- 2) L-Click on the VIDEO button located in the main **SETUP** window.

The **VIDEO SETUP** window will open.

- 3) In the Left Hand VIDEO 1 column, L-click on the video mode you want to use (BSE for backscattered electron image; SE for secondary electron image).

Wait for the correct options to appear on the screen.

- 4) Check the settings in the VIDEO 1 column. Normal settings should be:

For BSE:

- CONTRAST ATOMIC
- In Offset AUTO
- Brightness AUTO

- GAIN LOW
- COUPLING DC
- POLAR BIAS

For SE:

- PM AUTO
- DETECTOR 3
- PLATES ON

Notes: 1) Turn the SE detector OFF before changing from SE to BSE.

- 2) The Faraday cup needs to be off in order to change from MANUAL to AUTOMATIC mode.

- 5) If the settings are correct, close the **VIDEO SETUP** window by L-Clicking the VIDEO button located in the main **SETUP** window.
- 6) L-Click on the ROLLER CHOICE button in the **Roller Wheel Control** window to reset the roller wheels to XYZ. (the roller wheels automatically change to video adjustments when any video operations are performed).

F) CHECK THE BEAM AND CENTER THE TV MONITOR.

- 1) R-Click on the ROLLER CHOICE button in the **Roller Wheel Control** window (located in the lower right portion of the computer screen) and select STAGE from the pop-up menu to activate the roller wheel stage controls.

- 2) Move to a clean portion of the benitoite standard and focus using the X, Y, and Z thumbwheels.
- 3) L-Click on CUP OFF in the **Roller Wheel Control** window.
- 4) L-Click on SCAN OFF in the **Roller Wheel Control** window.
- 5) The beam should appear as a florescent blue point on the benitoite in the optical TV screen.
- 6) Adjust the camera so that the beam is centered in the crosshairs of the TV monitor. This is done by turning the two allen wrenches located on either side of the column just above the two front spectrometers.
- 7) After adjustment, L-Click on CUP ON in the **Roller Wheel Control** window.

G) MOVE TO THE ANDRADITE STANDARD.

- 1) L-Click on the STAGE CONTROL button in the **STAGE** window.

The **Stage Control** sub-window will appear.

- 2) R-Click once on STANDARD NAME in the **Stage Control** sub-window. When the standard list appears select ANDRADITE from the pop-up menu (the second name on the list).
- 3) L-Click MOVE in the **Stage Control** sub-window.

The stage should move to the andradite standard.

- 4) Close the **Stage Control** sub-window by L-Clicking on the STAGE CONTROL button in the **STAGE** window.
- 5) R-Click on the MAG button in the **Roller Wheel Control** window and select 1000.
- 6) L-Click on SCAN ON in the **Roller Wheel Control** window.
- 7) L-Click on CUP OFF in the **Roller Wheel Control** window.
- 8) R-Click on the ROLLER CHOICE button in the **Roller Wheel Control** window (located in the lower right portion of the computer screen) and select STAGE from the pop-up menu to activate the roller wheel stage controls.
- 9) Move to a clean portion of the andradite standard and focus using the X, Y, and Z thumbwheels.
- 10) L-Click on CUP ON in the **Roller Wheel Control** window.
- 11) L-Click on SCAN OFF in the **Roller Wheel Control** window.

H) SET AND VERIFY THE SPECTROMETERS.

- 1) L-Click on the WDS button located in the main **SETUP** window.
- 2) The **WDS SETUP** panel will appear.
- 3) L-Click on the XTAL button.
- 4) Check which crystals are currently installed. To change the current setup R-Click on the CRYSTAL button of desired spectrometer and R-Click on the crystal you want from the pop-up menu.
- 5) The crystals will change to the ones you have selected.
- 6) L-Click the PHA button. Then make sure that all of the spectrometers are set as follows.

Mode: Differential

(To Change: R-Click on the MODE button for each spectrometer and select DIFF from the pop-up menu).

Baseline: 500
Window: 4000

(To Change: Type in these values for each spectrometer. Be sure to enter a <return> after each entry or the changes will not take effect).

- 7) L-Click on the XTAL button.
- 8) Select the spectrometers to verify by L-Clicking on the SP1, SP2, SP3, and/or SP4 buttons. (The buttons of the selected spectrometers will turn dark gray).
- 9) **R-Click** on the VERIFY button and select ON CURRENT POSITION.

Important: Do not L-Click on the VERIFY button or the stage will move to a different spot. Always move to the andradite standard first and use the ON CURRENT POSITION selection.

- 10) L-Click YES.

Optional Note: If you wish to check the spectrometer offset values for your records they can be viewed in the SXLOCAL window. L-Click twice on the **SXLocal** icon on the Sun screen to open this window. Be sure to close it again when you are done recording the offsets.

D) ADJUST THE PHA SETTINGS

- 1) L-Click the PHA button.
- 2) Select the spectrometers to adjust by L-Clicking on the SPx (x = 1 – 4) buttons. The buttons of the selected spectrometers will turn dark gray.
- 3) L-Click the ADJUST button.

The gain and bias of the selected spectrometers will be adjusted.

- 4) Check that the tops of the PHA peaks shown in the display window lie in the middle of the energy distribution bar (the upper black line in the display) for each spectrometer. If not, repeat the procedure for the spectrometers that are off.
- 5) Close the PHA graphics window by L-Clicking DISPLAY again.
- 6) Close the **WDS SETUP** window by L-Clicking the WDS button in the main **SETUP** window.

Congratulations. The microprobe is now ready for analysis.