PHILIPS SEM 525 INSTRUCTIONS

Start-up:

Turn master mains switch on (if not already on)
Turn the water cooler/circulator timer on to HOLD (counter clockwise)
Press the SEM mains button ON (lower left panel, listen for the mechanical vacuum pump to come on)
Press the vacuum ON button

The red high vacuum light (left most panel) will be lit. After about 30 minutes, this light indicator will turn off indicating a sufficient vacuum to operate the microscope. However, to ensure maximum filament life, do not turn the beam on until the vacuum gauge reads well below “10”.

Turn on the exposure light column switch.
Turn on the “marker” switch.
Turn ON the detector and adjust the “Black Level”.

Set the detector to GREY SCALE and adjust brightness and contrast for the display screens (once set, this does not change appreciably over time).

If using the Polaroid camera, press and hold the photo start button and press the scan rate 2000 and 32 buttons. This sets the scan rate for the camera screen. The photo scan can be stopped by pressing the interrupt button.

Adjust operating parameters appropriate to the specimen characteristics.

Select accelerating voltage:

For maximum resolution and high magnification work, use 25 kV or above with gun bias setting of “1” and a spot size of 20 or 10. This electron beam will penetrate well into most samples and require a lower secondary electron detector gain setting. The detector bias may be turned to negative to detect backscattered (BS) electrons if desired, but there will be a lot of BS electrons with this kV setting. A better BS image will be obtained using a lower accelerating voltage.
Non-conductive samples may charge with these settings. Biological specimens that have been coated usually can be viewed with a lower accelerating voltage, usually 10 – 15 kV. With this setting, set the gun bias to “2” so as to keep the emissions current in a safe range of 30 – 40 \( \mu \text{A} \). A lower kV setting usually improves the surface image but also adds “noise” to the detector signal.

With the Wehnelt gun cap fitted with a 200 \( \mu \) aperture, and the filament to Wehnelt distance adjusted 6 small divisions, the following should be expected at filament saturation:

Bias 1, 25 kV, saturated filament current = 40 \( \mu \text{A} \).

Bias 2, 10 kV, saturated filament current = 35 \( \mu \text{A} \).

To lower the saturated filament current, increase the filament to Wehnelt distance.

**Select spot size:**

Generally speaking, use a large spot size (200 or 500) for low magnification work under 1000 x magnification. Smaller spot size is used for higher magnifications. As spot size is reduced, the detector gain must be increased (and visa versa) because fewer secondary electrons are generated. Black level is not affected as much. For 5K to 20K magnification work, use a 10 - 20 \( \mu \) spot size.

**Stage controls:**

Do not adjust the “Z” control. This is set to provide an optimal distance (10 – 12 mm for secondary electron detector and 20 mm for the EDAX detector).

Do not force any stage control knob.
The “X” and “Y” controls are approximately centered at 500 X 500 with TILT at 0 degrees.

Do not adjust the TILT less than 0 degrees nor greater than 90 degrees.

**Turning ON the electron beam:**

Turn ON the high tension. There should be a “blip” in the filament current gauge. Adjust the current to saturation (usually 5-6 clicks). **Do not over-saturate the filament.**

**Aligning the electron gun (if necessary):**

With a spot size of 500 and minimum magnification, turn up the filament current until the current no longer rises on the gauge. Adjust the detector gain to provide a circular illumination. Center this circle on the screen using the “gun shift” then use the “gun tilt” controls to give the brightest illumination.

**Changing or inserting a new specimen:**

Be sure the filament current and high tension is off and the filament has cooled for at least 1 minute.

Press the OFF button on the vacuum control panel.

Press the air button.

Wait for the specimen chamber door to release and then carefully pull the stage assembly out.

Use the 1.5 mm Allen wrench to secure the specimen stub to the holder. Do not over-tighten the set screw. It only needs to be firm.

Push the stage assembly into the chamber being **absolutely** sure that the specimen will not hit the EDAX detector.

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Do not handle specimen stubs with your bare fingers. Contaminates from your fingers will volatilize in the high vacuum and contaminate the final beam aperture and detectors which in turn will significantly degrade the image.
Press and “release” the AIR button and press the vacuum ON button. You should hear the vacuum pull the door sealed shut.

When the vacuum is ready (10 or less on the vacuum gauge and RED high vacuum light is off), the electron beam may be turned on.

**Shutting down the SEM:**

Turn the filament current to zero.
Turn off the high tension by pushing the HT button and letting it release.
Turn the black detector gain down 1 turn (clockwise).
Turn off the SE detector (push the button and let it release).
Turn the spot size to 500 (this is the lowest lens current setting).
Turn the magnification control to maximum (lowest lens current setting).
Turn off the light column switch.
Turn off the “marker” switch.

The SEM can be left in this setting as standby mode for the day.

Do not leave the SEM on overnight.

To completely shut down the SEM (overnight or longer):

Press the vacuum OFF button.
Press the power OFF button.

Set the cooling water timer to at least 30 minutes to cool the diffusion pump (quickly turn clockwise past OFF to >30).

If the microscope will not be used again for an extended period (1-2 days or more), then turn off the master mains switch.

Please be sure that specimens are prepared properly and are free of volatile substances. Otherwise, the detector and final pole piece aperture will become contaminated and degrade the SEM performance.