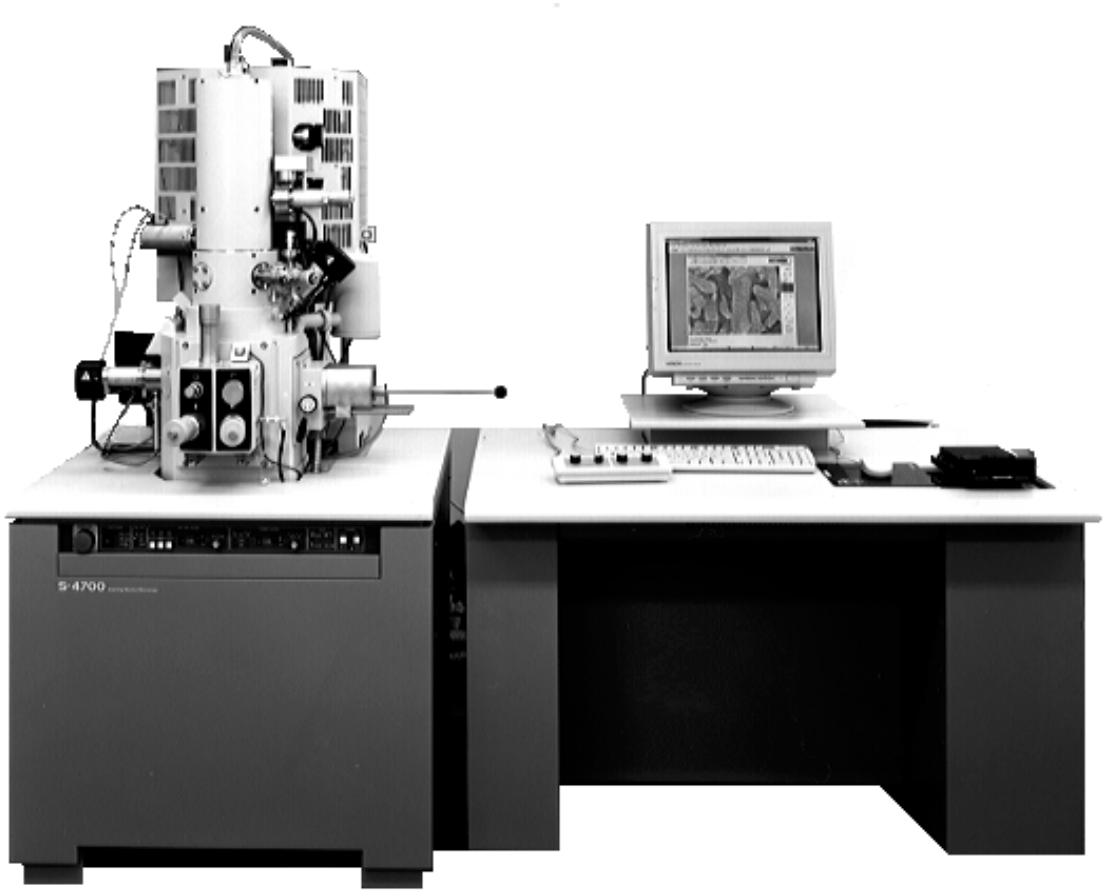
Hitachi S-4700 SEM
Training & Reference Guide

January 2007



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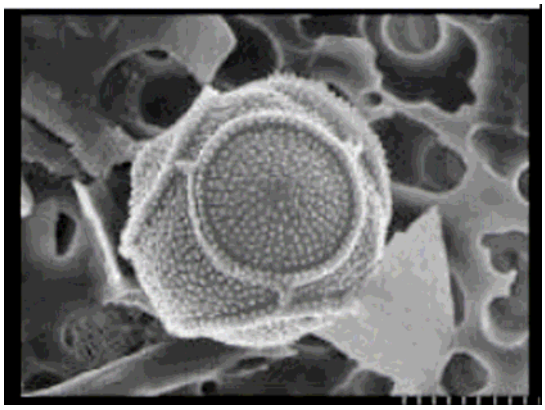

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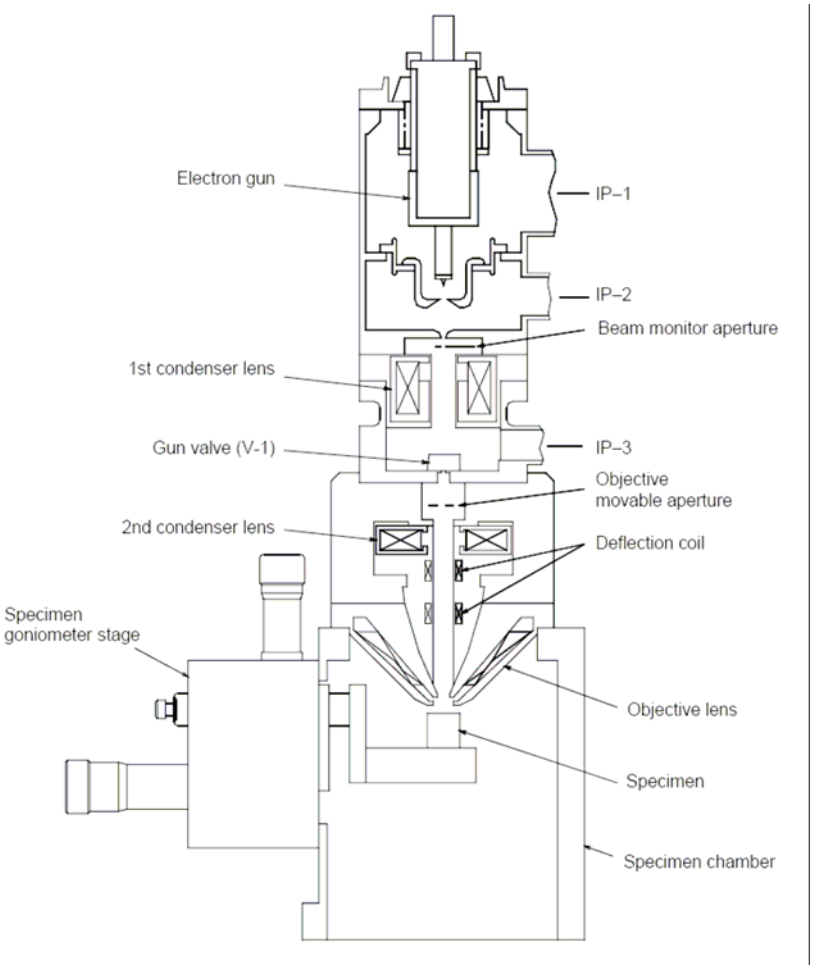


1. The Basic Components

Before getting into the details of how to operate our Hitachi scanning electron microscope (SEM), it is worthwhile to understand the technique and they type of instrument we have. Very simply, the SEM scans a sample with a beam of electrons that interact with the sample. Some of those electrons and other electrons generated during this process escape from the sample and reach a detector. The number of electrons that reach the detector at each point on the sample depends on the topology of the sample and the atomic weight of the atoms at the surface, and these variations in signal strength lead to image formation. A schematic of our instrument is shown on the previous page, and it illustrates the different instrumental components that are discussed below.

1.1 Electron Source

The Hitachi S-4700 has a cold cathode field emission source (Figure 1). The term "cold" is used because heat is not used to lower the work potential (as it is for some other types of emission sources). The tip is made up of a sharply etched piece of mono-crystalline tungsten. A field is applied to the tip to lower the work potential until the final 5-10 Å barrier is achieved at which point the electrons "tunnel" through the tungsten metal and emit down the column. There are two voltages applied, V_{acc} and V_{ext}. V_{ext} (V_1 in the figure) is the voltage applied between the tip and the first anode to extract electrons from the tip. The electrons can then be accelerated or decelerated by applying another voltage (V_{acc}, accelerating voltage, V_0 in the figure) between the tip and a second anode slightly farther from the tip than the first anode. Ultimately, it is the combination of these two anode potentials which sets the final electron speed down the optical column. Typically, the user sets the V_{acc}, and the software will automatically adjusts V_{ext}.

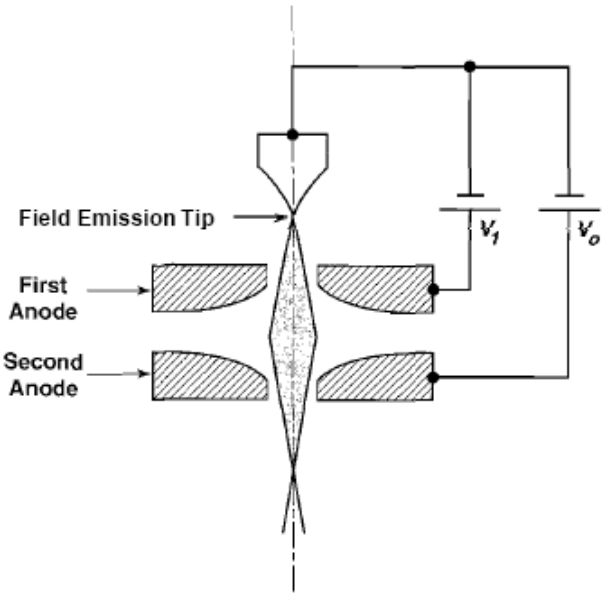


Figure 1. Schematic diagram of the Butler triode field emission source. V_1 (V_{ext}) is the extraction voltage of a few kilovolts and V_o (V_{acc}) is the accelerating voltage.

1.2 Lenses & Apertures

The electron beam is focused with a series of electromagnetic lenses. The Hitachi S-4700 has two condenser lenses, and an objective lens. The condenser lenses help to demagnify the electron beam (reduce it's size), and the objective lens acts to focus the beam onto the sample. The objective lens is much stronger than the condenser lenses, and in addition it must also contain space for the scanning (deflection) coils, the

stigmator, and the beam-limiting aperture. The objective lens in the Hitachi S-4700 is a "snorkel" lens (Figure 2) which has low aberrations (described below), and it can accommodate large specimens. In addition, it can simultaneously accommodate both a lower (i.e., an E-T) and an upper (through-the-lens) secondary electron detector as described below, providing valuable flexibility for imaging.

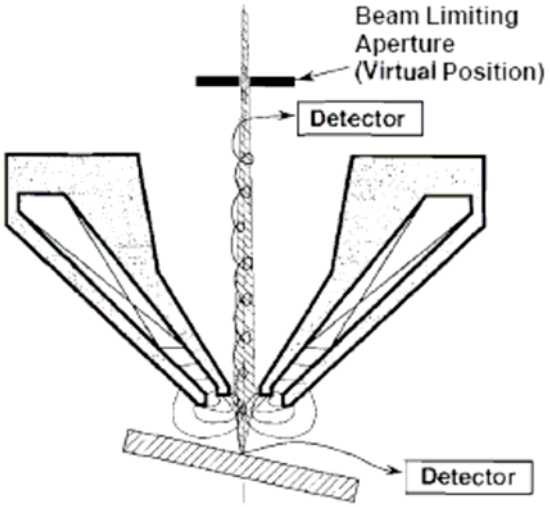


Figure 2. Illustration of a snorkel lens, where a large specimen is placed outside the lens, but the magnetic field extends out from the lens to the specimen. Lens aberrations are small.

All lenses suffer from defects or aberrations in their performance, which need to be minimized. These aberrations include: i) spherical aberration, ii) aperture diffraction, iii) chromatic aberration, and iv) astigmatism. Spherical aberration arises because electrons in trajectories further away from the optic axis are bent more strongly by the lens magnetic field than those rays near the axis (Figure 3a) resulting in a blurred image at the image plane. Aperture diffraction results when very small apertures are used and the wave nature of electrons gives rise to a broad circular "Airy disk" intensity distribution instead of a point at the Gaussian image plane (Figure 3b). Spherical aberration dominates at high aperture angles (defined as α in Figure 3b), while aperture diffraction dominates at low aperture angles, resulting in an optimal aperture angle that minimizes these effects. The aperture angle, α can be adjusted by the size of the aperture, which also regulates the number of electrons that reach the sample.

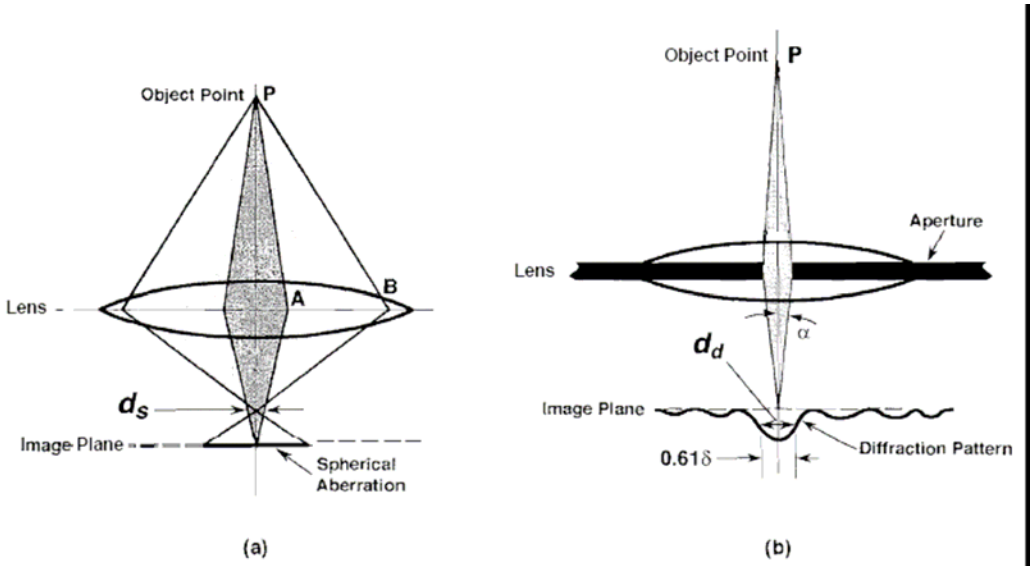


Figure 3. Schematic diagrams showing how (a) spherical aberration and (b) aperture diffraction in a lens cause a point object at *P* to blur into an enlarged spot at the Gaussian image plane. The disk of minimum confusion d_s and one-half the Airy disk d_d are used in calculations of probe size.

Chromatic aberration arises because electrons emerging from a point source with slightly different energies will be focused at different locations in the image plane (Figure 4a), but only becomes significant at energies <10keV. Finally, machining errors, inhomogeneities in the polepieces, asymmetry in the lens windings, and dirty apertures all may lead to a lens that is not perfectly cylindrical, but is "astigmatic". The effect of this asymmetry is illustrated in Figure 4b. We can use a stigmator, or a device that applies a weak supplement magnetic field, to make the lens appear symmetric to the electron beam.

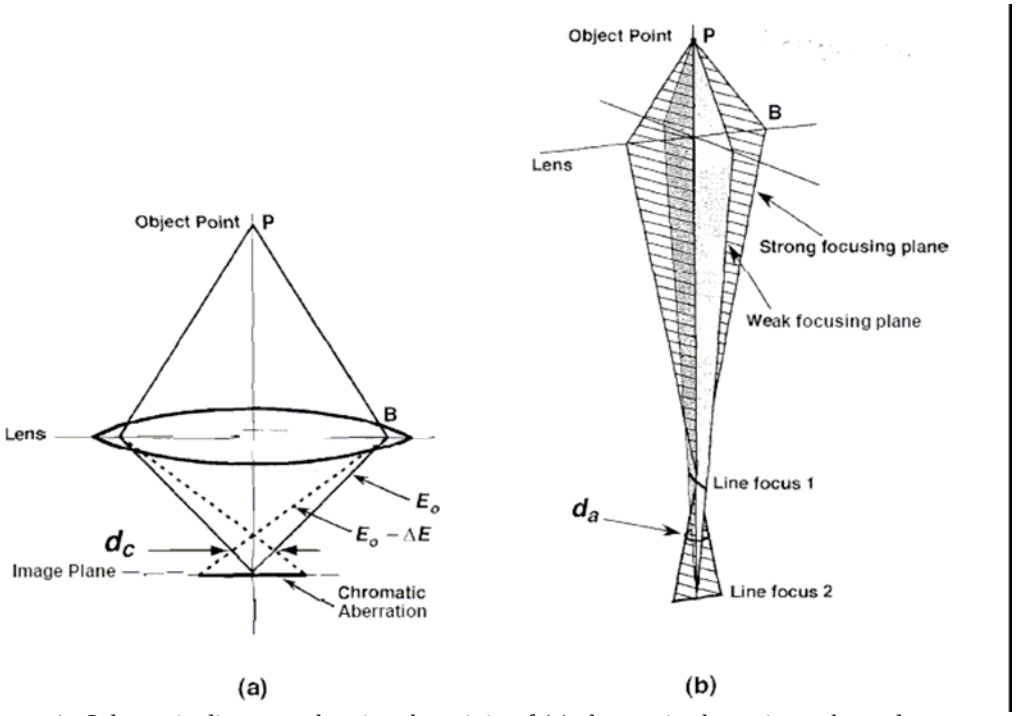


Figure 4. Schematic diagrams showing the origin of (a) chromatic aberration, where electrons of differing energy are focused at different locations. (b) A point object is focused to two line foci at the image, and the desired small focused beam can only be obtained by forcing the two line foci to coincide using the stigmator.

1.3 Deflection System

The deflection system rasters the electron beam over the sample, and allows an image to be formed. Located on each side of the column, the electromagnetic deflection coils act to first bend the beam off the optical axis, and then back onto the axis. As the beam sweeps across the sample in a point-to-point manner, the beam-specimen interactions produce electrons, x-rays, and other information-carrying signals. The Hitachi S-4700 detects the electrons that come from the sample.

1.4 Electron Beam-Specimen Interactions

After electrons are extracted from the source, they travel through the lenses and then interact with the specimen of interest. As the electron beam enters the specimen, the electrons will interact as negatively charged particles with the electrical fields of the

specimen atoms. The positive charge of the protons is highly concentrated in the nucleus, whereas the negative charge of the atomic electrons is much more dispersed in a shell structure. The beam electron-specimen atom interaction can deflect the beam electrons along a new trajectory ("elastic scattering" with no kinetic energy loss), causing them to spread out laterally from the incident beam footprint. This elastic scattering can, after numerous events, actually result in beam electrons leaving the specimen (a process called "backscattering", Figure 5). The probability of elastic scattering increases strongly with atomic number (Z), approximately as Z^2, (because heavier atoms have a much stronger positive charge on the atomic nucleus), and decreases as the electron energy increases, approximately as $1/E^2$.

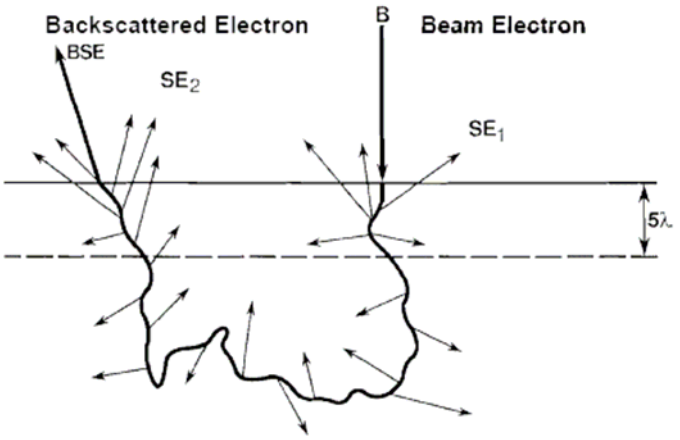


Figure 5. Schematic illustration of the origin of two sources of secondary electron generation in the sample. Incident beam electrons (B) generate secondary electrons (SE_1) upon entering the sample. Backscattered electrons (BSE) generate secondary electrons (SE_2) while exiting the sample. λ is the mean free path for secondary electrons.

Simultaneously with elastic scattering, the beam electrons lose energy and transfer this energy in various ways to the specimen atoms ("inelastic scattering"), but this transfer takes place gradually, so that the beam electrons propagate through many atom layers into the specimen before losing all their energy. Inelastic scattering gives rise to useful imaging signals such as secondary electrons and analytical signals such as x-rays. Secondary electrons (SE) are loosely bound outer shell electrons from the specimen atoms which receive sufficient kinetic energy during inelastic scattering of the beam electrons to be ejected from the atom and set into motion. The SE thus created will propagate through the solid, and those that are created relatively close to the sample surface may escape from the surface. Thus, there are multiple types of SE: those that are created with the probe electrons enter the specimen (SE_1), and those that are created as a BSE is leaving the specimen (SE_2). (Figure 5) The ratio of SE_2/SE_1 increases with increasing atomic number (0.18 for carbon, and 1.5 for gold). In addition, there are BSE that hit either the chamber walls or the polepiece before entering the detector (Figure 6). These "indirect" BSE can also cause the

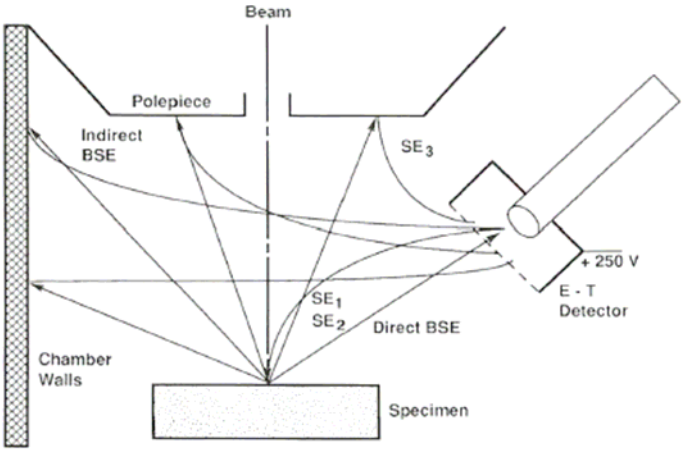


Figure 6. Schematic illustration of the indirect collection of backscattered electrons by a positively biased E-T detector. The BSE strike the chamber walls, where they create secondary electrons. These SE are collected by the E-T detector with high efficiency. Although nominally a contribution to the SE signal, these SE actually represent the BSE signal component because their number must rise and fall according to the behavior of the BSE.

emission of secondary electrons (SE_3) from these surfaces which can also be detected.

The incident electrons interact with a certain volume of the sample called the interaction volume. There are numerous analytical expressions that have been used to model the size and shape of this interaction volume, which will not be covered here. The interaction volume depends on a number of factors, including the beam energy, the atomic number of the specimen, and the angle the incoming probe beam makes with the sample surface. The number of backscattered and secondary electrons (BSE and SE) produced will depend on these parameters and others such as the topology of the sample, and will result in the image contrast observed.

1.5 Detector

By far, the most common detector in use in SEMs is the Everhart-Thornley (E-T) detector (Figure 7). When an energetic electron (~10 keV) strikes the scintillator, material (doped plastic or doped glass, or a crystalline compound such as CaF_2 doped with europium), light is emitted. The light is conducted by total internal reflection in a light guide (a solid plastic or glass rod) to a photomultiplier. Because the signal is now in the form of light, the signal can pass through a quartz window, which now forms a vacuum barrier, to the first electrode of a photomultiplier. At this photocathode, the photons are converted back into electrons, and the electrons are accelerated onto the successive electrodes of the photomultiplier, producing an ever-increasing cascade of electrons until the final collector is reached.

The Hitachi S-4700 has two detectors referred to as the upper ("through-the-lens", TTL) detector and the lower (Everhart-Thornley, E-T) detector. (Figure 7) The strong "snorkel" type objective lens just above the sample produces a strong magnetic field that is projected into the specimen chamber to reach the specimen plate. One major consequence of the strong magnetic field is to trap with high efficiency those SE emitted from the specimen, that is the types SE_1 and SE_2, especially at short working distances. The SE spiral up along the magnetic field lines and pass up through the lens bore. By placing a scintillator with a high bias (+10 kV) on the surface electrode above the objective lens, a TTL secondary electron detector is created. The TTL detector virtually

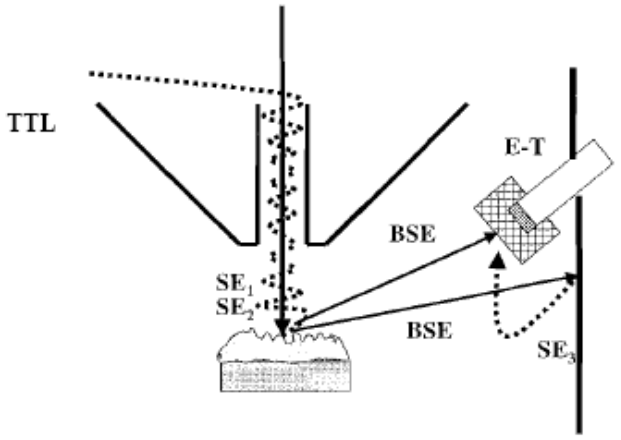
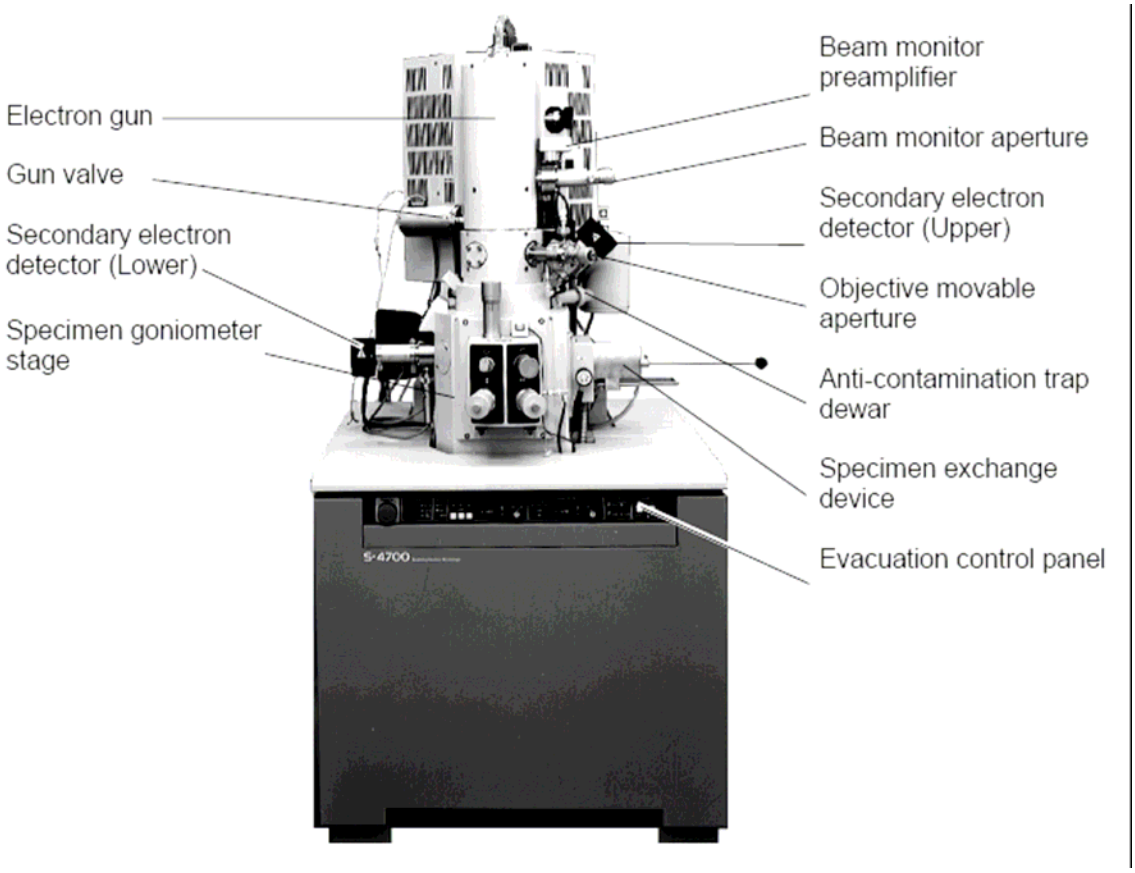


Figure 7. "Through-the-lens" (TTL) detector for SE used in high-performance field emission SEMs. The magnetic field of the objective lens projects into the sample chamber and the SE (types SE_1 and SE_2) are trapped by this field and follow spiral trajectories around the lines of magnetic flux up through the lens. A scintillator-light guide-photomultiplier detector above the lens collects the emerging SE and the high potential on the scintillator raises their kinetic energy sufficiently to cause light emission. Note that secondary electrons of type SE_3 are excluded by the TTL because they are formed too far off-axis to be collected by the on-axis magnetic field. The strong magnetic field of the snorkel lens captures most of the SE_1 and SE_2, greatly reducing the number available for capture by the more remote E-T detector. BSE and SE_3 collection by the E-T detector is relatively unaffected.

eliminates the SE_3 component produced remotely by the BSE because the vast majority of these SE_3 are created well off the optic axis by the BSE colliding with the bottom of the lens and chamber walls. The TTL also discriminates against the BSE, because the magnetic field does not act to collect off-axis BSE, and those on-axis BSE that are emitted directly into the solid angle of the lens bore tend to travel in straight lines and are too energetic for collection by the potential on the scintillator above the lens.



2. Operation

A picture of the Hitachi S-4700 is shown above. Users should be familiar with the location of basic components on the instrument (electron gun, apertures, detectors).

2.1 Specimen Preparation

During specimen preparation, observe the following:

(1) Use clean gloves when exchanging specimens. Holding the specimen or specimen stub with bare hands should be avoided to prevent the introduction of hand oils into the vacuum system.

(2) When using double-sided adhesive tape to fix a specimen to the stub, use a small amount to minimize out-gassing. The use of double-sided adhesive tape may also cause specimen drift.

2.1.1 Specimen Preparation According to Materials

The method of specimen preparation varies with materials. Below are preparation methods for typical types of specimens.

(1) Conductive Specimens such as Metals

These types of specimens can be observed without preparation. However, coating with heavy metals may result in better contrast.

(2) Non-conductive Specimens such as Semiconductors, Fibrous Specimens and Polymeric Materials

Coating with conductive materials is recommended. Our sputter coater currently has a Au/Pd alloy target, and a thin (~2nm) coating should prevent the sample from charging, a build-up of charge on the sample surface that distorts the image. To observe these kinds of specimens without a conductive coating use low accelerating voltages (1 kV or lower). However, at higher magnifications a metallic coating may be required.

(3) Biological Specimens

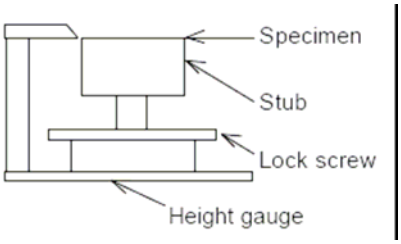
After dehydration, dry the specimen by using a method such as critical point drying, freeze drying or other drying techniques, then coat the specimen with conductive material.

⚠ CAUTION ⚠

Pulverized ferromagnetic specimens should NOT be introduced into the specimen chamber. If particles from such a material are attracted to the objective lens due to its strong magnetic field, the microscope performance may be degraded. Because ferromagnetic samples strongly interact with the magnetic field of the objective lens, they should be attached firmly to the specimen stub.

2.1.2 Adjustment of Specimen Height

Put the specimen stub on the specimen holder and adjust it to the proper height using the specimen height gauge as shown in the figure. To adjust, loosen the lock screw and adjust the specimen height so that the highest point of the specimen is the same as the bottom of the height gauge. Then, tighten lock screw.



⚠ CAUTION ⚠

Specimen height must be adjusted carefully. It must not be 0.5 mm higher than the bottom of the height gauge. If it is higher than this, the specimen may strike the objective lens and cause damage when operating at a short working distance or at a high tilt angle. Also, accurate setting of specimen height minimizes image shift during specimen tilting.

2.2 Preliminary Operation

2.2.1 Check of Column Vacuum

At the beginning of SEM operation, check the evacuation control panel. The following conditions must be met:

(1) **IP1**, **IP2** and **IP3** lamps are lit (See figure below).

(2) Ion pump readings are better than the following.

IP1: $2 * 10^{-7}$ Pa
IP2: $2 * 10^{-6}$ Pa
IP3: $7 * 10^{-5}$ Pa

Adjust the knob to the various gauges (IP1, IP2, IP3) to read the pressure at these points in the chamber. If vacuum readings do not satisfy the above conditions, gun baking is required. (Notify Carrie Donley at 843-2859, Chapman 243.)

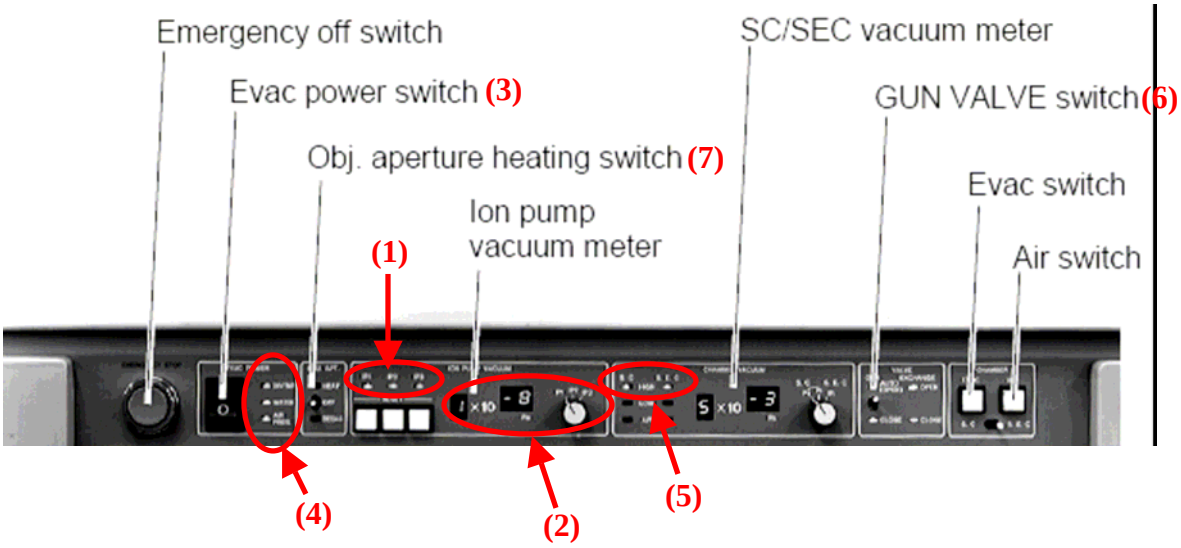
(3) **EVAC POWER** switch is set at **1** (ON).

(4) **DP/TMP**, **WATER** and **AIR PRES** lamps are lit.

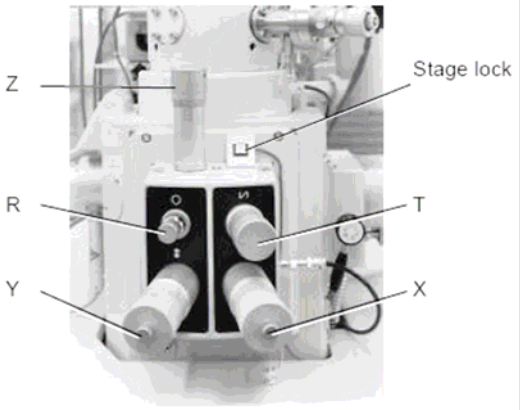
(5) **HIGH** lamps of **S.C VACUUM** (specimen chamber) and **S.E.C VACUUM** (specimen evacuation chamber) are lit.

(6) **GUN VALVE** switch is at **AUTO** and the **OPEN** lamp is flickering.

(7) **OBJ. APT**. switch is set at **HEAT**. Usually keep the OBJ. APT. switch on the evacuation control panel at HEAT. The aperture is heated to about 150°C to remove contaminants to one tenth or less of what it would be at room temperature.



2.2.2 Check Specimen Exchange Position



(1) Confirm that the Stage Lock is free (the lamp of Stage Lock switch is off), and the **Z** knob of the specimen stage is set to the specimen exchange position (Z: 12.0 mm).

(2) Click the **Go to Home** button in the **Stage Control** dialog window (accessible from the **Operate** pull down menu). The stage is then moved to the specimen exchange position, and the color of the indicator button turns green. The previous user should have left the sample stage in this position. (Exchange positions: Z=12.0 mm **X**=12.5 mm **Y**=12.5 mm **R**=0° **T**=0°.)

2.2.3 Introducing a Specimen into the Vacuum Chamber

Before introducing a specimen into the chamber, make sure to check the height of the specimen with the height gauge.

(1) Turn on the infrared chamberscope by pressing the red button on the small screen between the instrument and the computer monitor. Turn on the monitor by pressing the power button on the remote control.

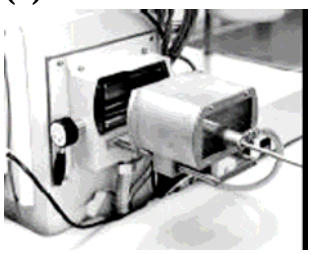
(2) Confirm that the **EXCHANGE VALVE** is at **C** (Close) position, and press the **AIR** switch on the column vacuum control panel (see figures below).

(3) After the specimen exchange chamber has reached atmospheric pressure (It takes about 10 secs), open the specimen exchange chamber.

(2 & 10)

(2)

(3)



(4)

(6)

(7)

(4) Push the specimen exchange rod slightly to unlock it, and screw it into a threaded hole of the specimen holder. (Do not screw in the rod with too much force. Threads may be crushed.)

(5) Pull the rod and make sure it is locked. Then, close the specimen exchange chamber.

(6) Press the **EVAC** switch on the column vacuum control panel. Wait until vacuum in the specimen exchange chamber is improved enough and the **HIGH** lamp of **S.E.C VACUUM** is lit.

(7) Turn the EXCHANGE VALVE to O (Open) position.

(8) Looking into the specimen chamber, push in the exchange rod and set the specimen holder into the stage by sliding it along the guide rails.

(9) Turn the exchange rod counterclockwise until it is separated from the threaded portion of the specimen holder, and pull out the rod fully.

(10) Turn the **EXCHANGE VALVE** to **C** (CLOSE).

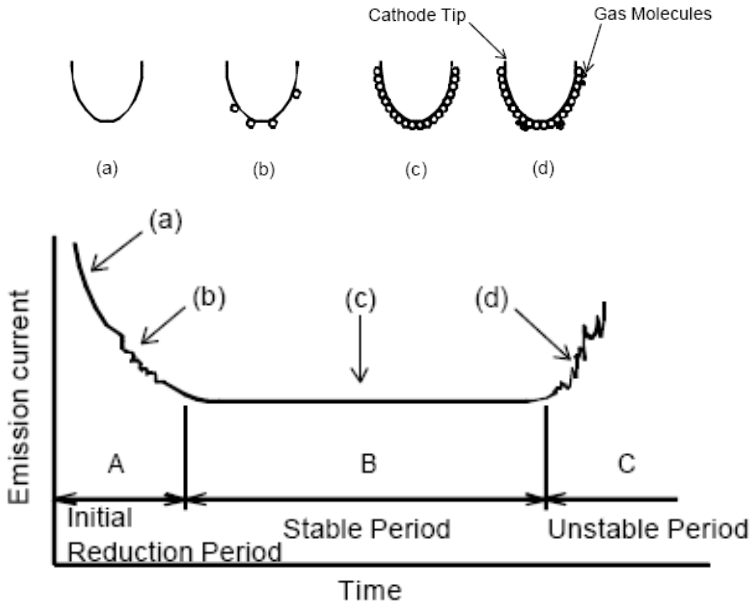
2.3 Application of High Voltage

2.3.1 Flashing

2.3.1.1 Purpose of Flashing

Flashing is a procedure for removing gas molecules which have been adsorbed on the surface of the cathode (FE tip) in the electron gun. The figure below illustrates the

emission current as a function of time after flashing, and the corresponding illustrations indicate the amount of gas adsorption onto the tip.



A: In the initial reduction period, just after flashing, gas is completely discharged from the cathode tip, (a). As gas molecules are gradually adsorbed onto the cathode, emission current decreases. In this stage, gas molecules are desorbed, adsorbed and transferred, so noise (fluctuation of emission current) occurs, (b).

B: In the stable period approximately one layer of gas molecules is adsorbed onto the cathode. In this status a stable emission current is usually available, (c). Although noise (fluctuation of emission current) may appear occasionally due to desorption and adsorption of gas molecules a stable status returns in a comparatively short time. Images should be recorded after confirming the stable status. In this period emission current slightly decreases with time.

C: In the unstable period a large quantity of gas molecules desorb and adsorb. Consequently, the emission current fluctuates considerably while increasing, (d). The tip should be flashed again once the current enters this unstable period.

The periods sectioned on the time axis in the above graph vary depending on electron gun vacuum, residual gas component, cathode/anode cleanliness and emission current value. With the Model S-4700, they are roughly given as follows.

· Initial reduction period: 30 min to 3 hours
· Stable period: 2 to 12 hours

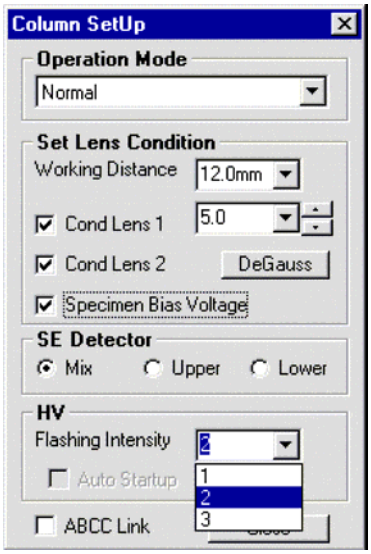
When the initial reduction period is half an hour, the stable period lasts 2 to 4 hours, and 4 to 10 hours when the initial reduction period is 1 to 2 hours (approximate values).

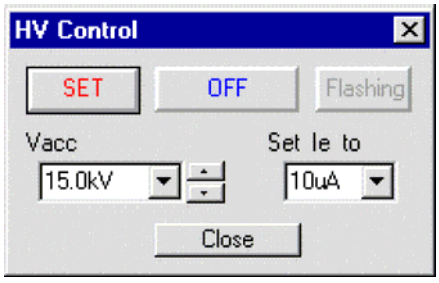
2.3.1.2 Flashing Procedure

(a) Check the flashing intensity and setup by clicking **Setup** from the Menu Bar and choosing **Column Setup** (see figure below). Set the Flashing Intensity in the screen to **2**.

(b) Select the **HV Control** from **Setup** on the menu.

(c) Click **Flashing** button and then **Execute** button. (Flashing is only executed with high voltage in OFF status.) Then for two seconds the **EMISSION CURRENT** indicator reads an emission current for flashing. Record this current in the log book under "Comments / Problems".





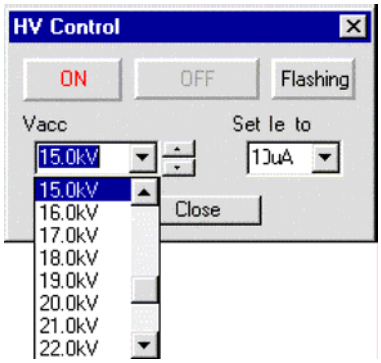
The tip should be flashed once everyday before use, and again if the emission current becomes unstable.

2.3.2 Setting of Accelerating Voltage and Emission Current

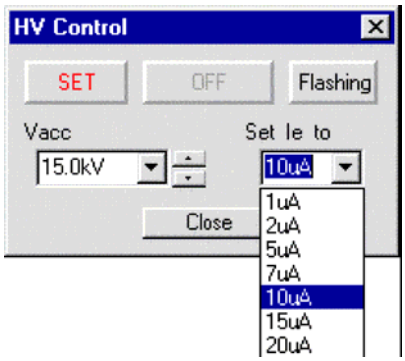
To set the accelerating voltage and emission current, use the **HV Control** dialog window.

(1) To set an accelerating voltage, select a voltage from the **Vacc** listbox. The accelerating voltage can also be set by using the keyboard to input a value followed by the Return key. For tips on setting the accelerating voltage, see section 2.7.1.

(2) To set an emission current, select a current in the **Set Ie** to listbox. Set it at 10 µA for normal operation. Both accelerating voltage and emission current are adjusted immediately upon changing them if the HV is on.



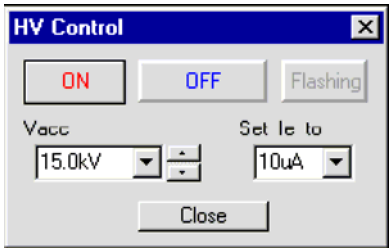
Accelerating Voltage



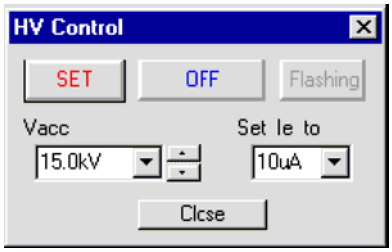
Emission Current

2.3.3 Application of High Voltage

Click the **ON** button in the **HV Control** dialog window. Accelerating voltage (Vacc) and extracting voltage (Vext) are then applied with the values indicated in the HV display area. The **ON** button is changed to read **SET** when the high voltage is applied. You can set emission current to a selected value in the Set Ie to box by clicking this button.
You can also turn the HV On and Off using the ON and OFF buttons on the tool bar when you do not need to change the high voltage value.



HV OFF



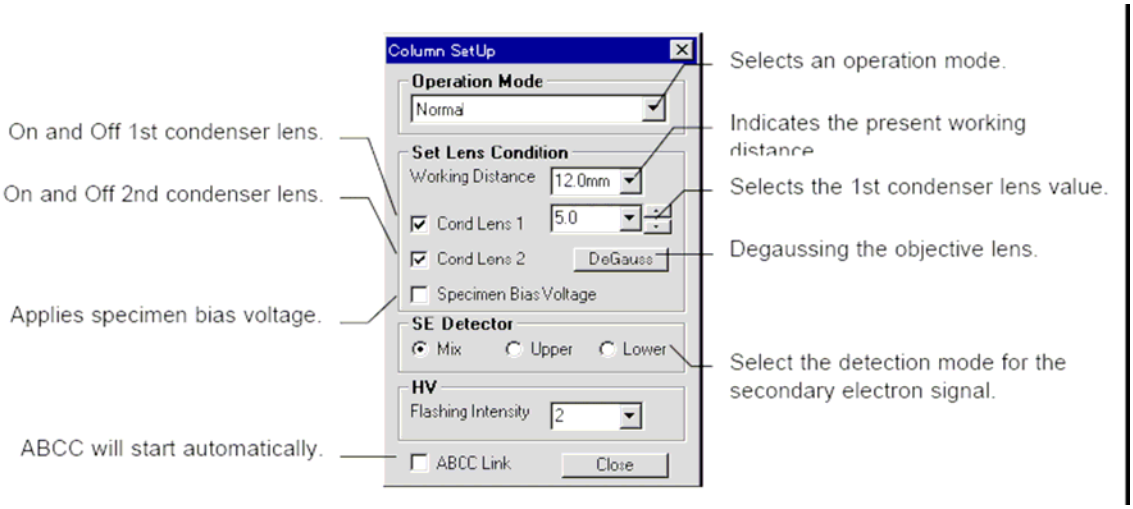
HF ON

Additional controls for HV control

2.4 Optimizing the Electron Beam

2.4.1 Selecting Electron Optical Column Conditions

The electron optical column conditions need to be selected before alignment and operation of the SEM begins. Select the **Column** command from the **Setup** menu to open **Column Setup** dialog window.



2.4.1.1 Operation Mode

The S-4700 has 5 operation modes listed below. Select a mode of interest to suit the specimen and the purpose of microscopy.

(a) Ultra High Resolution mode: Highest resolution is attainable. Use this mode for high-resolution observation at a short working distance (WD, described below) of 6 mm or shorter. This mode can be operated at longer WD than 6 mm.

(b) Normal mode: Optimized for use with WD at the specimen exchange position (12 mm). Recommended range of WD is 6 mm to 15 mm.

(c) Long WD mode: Optimized for use at a long WD of 15 mm or longer.

(d) Analysis mode: Large probe current is attainable. Use it for X-ray analysis, or when a large probe current is required.

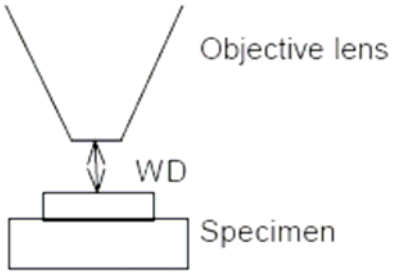
(e) Magnet Sample mode: Astigmatism correction range is enlarged. Use it for observation of ferromagnetic specimens such as iron that make astigmatism correction difficult.

⚠ CAUTION ⚠

Pulverized ferromagnetic specimens should not be introduced into the specimen chamber. If particles from such a material are attracted to the objective lens due to its strong magnetic field, the microscope performance may be degraded. Because ferromagnetic samples strongly interact with the magneticfield of the objective lens, they should be attached firmly to the specimen stub.

2.4.1.2 Set Lens Condition

(a) Working Distance (WD)



Working Distance is the distance between the bottom face of the objective lens and the surface of the specimen. At a shorter WD, higher resolution is attainable. At a longer WD, a larger tilt angle and a greater depth of focus is attainable (see table below). When a WD is selected in the **Column Setup** dialog window, the objective lens current is adjusted in order to focus an image at a specified WD. The actual working distance must be adjusted manually by using the Z position knob on the instrument to match that entered in the **Column Setup** window.

⚠ CAUTION ⚠

Care should be taken when adjusting the working distance so that the sample does NOT hit the objective lens. You can view both the sample stage and the objective lens with the infrared chamberscope camera display on the small monitor between the instrument and the computer monitor.

	Short	← WD →	Long
Resolution	High	← →	Low
Depth of focus	Shallow	← →	Deep
Specimen tilt angle	Small	← →	Large

The available WD value is limited for each Operation Mode and accelerating voltage and is given in the bottom left corner of the computer screen. The following table shows available WD values and recommended ranges.

Mode	WD: Type I (Type II)	Recommended Range (WD: mm)
Ultra High Resolution	2.5* to 15 (15)	less than 6
Normal	6* to 27.5 (30)	6 to 15
Long WD	6* to 27.5 (30)	more than 15
Analysis	6* to 27.5 (30)	more than 6
Magnet Sample	6* to 27.5 (30)	more than 6

*: The minimum WD varies with accelerating voltage.

(b) Cond Lens 1 (First Condenser Lens)

It is possible to adjust the probe current by setting the Condenser Lens 1 value in the **Column Setup** dialog window. Select a number from the pull-down menu, or key in a number (1.0 to 16.0) followed by the Return key. Typically this is set at 5.0. Both the probe current and the beam size will increase with a smaller value. For tips on setting the condenser lens 1 value, see section 2.7.2.

(c) Cond Lens 2 (Second Condenser Lens)

Condenser lens 2 can only be turned ON or OFF on the **Column Setup** dialog window. Normally, it is set to ON.

(d) Specimen Bias Voltage

A specimen Bias Voltage (-15V) is applied for optimum secondary electron detection. Set it to OFF in the **Column Setup** dialog window for normal operation.

(e) Degauss Operation

The Degauss operation eliminates hysteresis of the magnetic field in the objective lens. When focus is changed greatly, accuracy of magnification or alignment of the electron optical axis may degrade due to hysteresis of the focusing magnetic field. Click **Degauss** button in the **Column Setup** dialog window under the following conditions:

· After changing focus widely.

· Before making the electron optical axis alignment.

Degaussing is automatically effected when the WD is changed in the **Column Setup** dialog window, when the accelerating voltage is changed, or when a new operation mode is selected.

2.4.1.3 Secondary Electron Detector

The S-4700 has two secondary electron detectors: the upper and the lower detectors. The upper secondary electron detector is placed above the objective lens and secondary electrons are detected through the magnetic field of the lens. The lower secondary electron detector is placed in the specimen chamber, where a large amount of the signal is due to backscattered electrons. Signals from these two detectors can be selected individually, or mixed together. The image contrast of these detector signals is unique in that each has its own special characteristics. To select a detection mode, open the **Column Setup** or **Signal Select** dialog window and select upper, lower, or mixed.

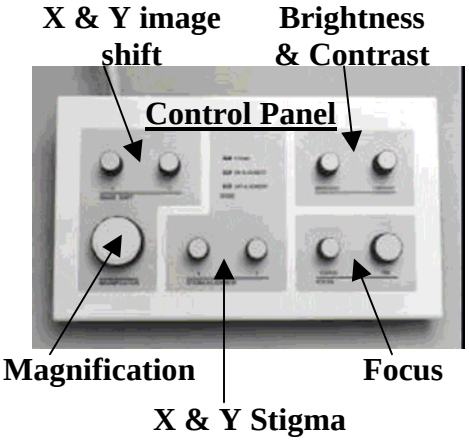
2.5 Operation for Image Observation

Follow the operation below for observation of a scanning image. More detailed instructions for adjusting the magnification, moving the stage, focusing, and astigmatism correction follow. Most adjustments can be made on the **Control Panel** shown to the right.

(1) Adjust the magnification with the magnification knob on the control panel. Start in low mag mode. Adjust the focus & make sure you are imaging your sample. Zoom in if necessary, and refocus.

(2) Once you have chosen a general area to image, switch to high mag mode by clicking on the "H/L" button. Continue to zoom in and refocus.

(3) Alternatively correct for the stigmatism and adjust the focus.



(4) Adjust the brightness and contrast on the control panel, or the  button will adjust the brightness and contrast automatically.

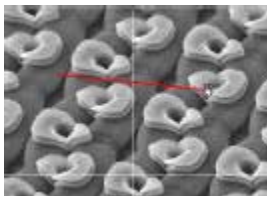
(5) Change the scan speed using the  buttons.

2.5.1 Selecting Magnification

The magnification mode (low or high) is indicated by the yellow LM or HM in the Magnification window. Switch between low mag. and high mag. with the "H/L" button. Adjusting the magnification is most easily done with the knob on the **Control Panel**.

2.5.2 Moving the Specimen Stage

When the mouse cursor in the Scanning Image area is turned into the Stage-Hand mark,  , the stage can be driven in the following way. (To turn the mouse cursor to the Stage-Hand mark, click the mouse icon  in the Stage area of the **Scanning Image** window.) Place the mouse cursor at a starting point on the scanning image. Move the mouse to an end point while holding down the left button (a red line is drawn), and release it. The stage is then driven so that the image at the start point moves to the end point. At high magnifications such as 50,000x or more, the stage may not move correctly. In such a case, use the electrical image shift on the control panel.

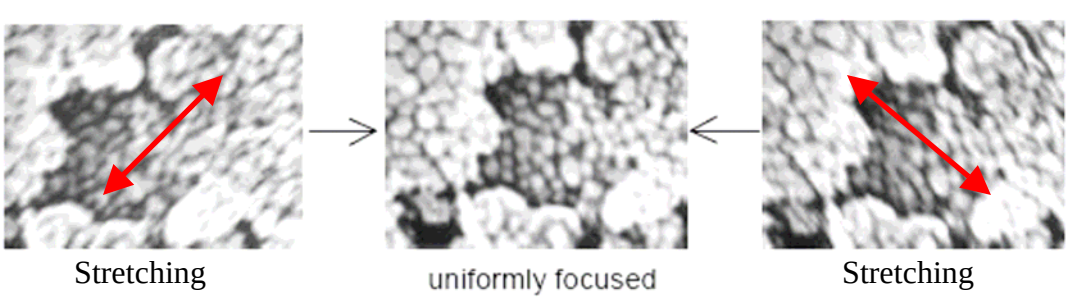


2.5.2 Focus and Astigmatism Correction

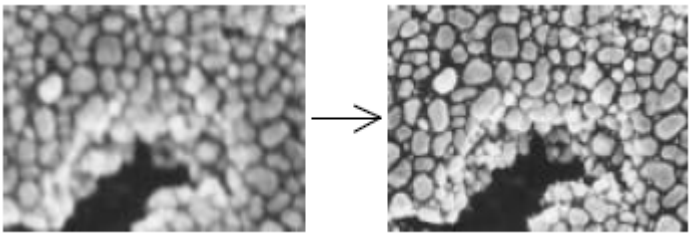
Focus and astigmatism correction can be done manually or automatically.

Focusing and astigmatism corrections are related to each other and need to be repeated alternately.

(a) Focus the image. When there is no astigmatism, the sharpest image is obtained at the best-focus point. When there is astigmatism, the image looks like it is stretching in one direction at an overfocused condition and stretching in another direction (90° from the first) at an underfocused condition. It will appear uniformly focused at the best-focus point.



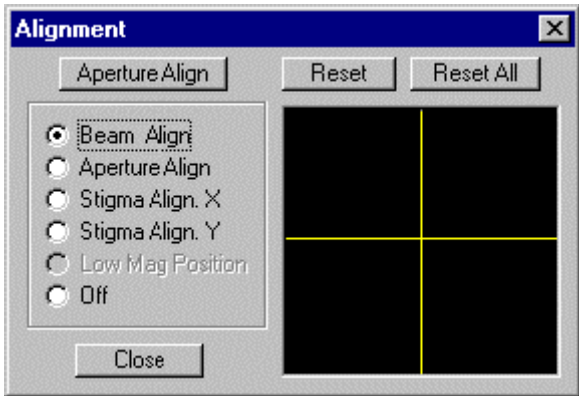
(b) Adjust the X and Y stigmators alternately for the sharpest image.



(c) Focus again and check image drift and sharpness.

(d) Repeat steps (a) to (c) until no additional improvements in image quality are obtained.

2.6 Column Electromagnetic Alignment



For the best performance of the instrument, alignment of the electron optical column axis is necessary. Generally, an electromagnetic alignment is sufficient, while a mechanical alignment may be needed for more critical applications. Perform the following alignment when you change the accelerating voltage, operation mode, or setting of Cond Lens 1. If you notice the image moving while focusing or correcting astigmatism, only perform an Aperture Alignment or Stigma Alignment, respectively. For all electromagnetic alignments, adjust the **STIGMA/ALIGNMENT X** and **Y** knobs on the control panel.

2.6.1. Beam Alignment

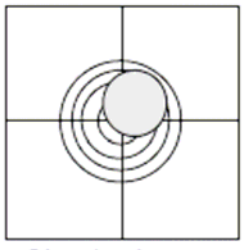
Beam Alignment is necessary in order to align the electron beam down the center of the electron optical column and through the center of the objective lens aperture.

(a) Click the **DeGauss** button in the **Column Setup** dialog window.

(b) Open the **Alignment** dialog window by clicking the  icon on the toolbar, or by selecting the **Alignment** command from the **Operate** menu.

(c) Click the **Beam Align** button in **Alignment** dialog window. A circular image will appear along with a target in which to center it. If the image is faint, increase the brightness.

(d) Make adjustments with the alignment knobs (stigma X and Y) so that the circular image appears in the center of screen.


Circular image

(e) Turn off the Beam Align mode (click **Off** button in **Alignment** dialog window).

2.6.2 Aperture Alignment

Aperture Alignment is necessary to align the electron beam through the center of the objective lens.

(a) Set the magnification to about 5,000x and position a point of interest in the center of the display.

(b) Focus the image and correct astigmatism.

(c) Click the **Aperture Align** button in the **Alignment** dialog window and make adjustments so that the wobbling motion of the image is minimized.

(d) Turn off the Aperture Align mode (click the **Off** button in **Alignment** dialog window).

2.6.3 Stigma Alignment X, Y

Stigma Alignment is necessary to minimize image drift seen when correcting the astigmatism.

(a) Set the magnification to about 5,000x and position a point of interest in the center of the display.

(b) Focus the image and correct astigmatism.

(c) Click **Stigma Align X** button in the **Alignment** dialog window and make adjustments (Stigma X & Y knobs) so that the wobbling motion of the image is minimized.

(d) Click the **Stigma Align Y** button in the **Alignment** dialog window and repeat the same adjustment as above (c).

(e) Turn off the Stigma Align mode (click **Off** button in the **Alignment** dialog window).

NOTICE: Stigma Alignment may not completely stop image movement. Use 50,000x or lower magnification for adjustment.

2.7 Image Quality

The following are references for getting better image quality.

2.7.1 Accelerating Voltage and Image Quality

There is a large range of accelerating voltages to choose from. Resolution, image quality, charging and other effects are greatly determined by the selected accelerating voltage. Below is a guideline for selecting an accelerating voltage with which to image various specimens.

(1) Resolution: Higher spatial resolution can be obtained at higher accelerating voltages.

(2) Charging: For uncoated insulator specimens, accelerating voltages lower than 1 kV are recommended for minimizing charging. In some cases, high accelerating voltages (20 kV or higher) may produce a better image.

(3) Contamination: Influence of contamination appears more at low accelerating voltages

(4) Magnetic Field Disturbance: Disturbance by leakage magnetic field (wobbling or distortion of the image) is greater at low accelerating voltages.

(5) Contrast: Generally a soft-tone image is obtained at low accelerating voltages because more secondary electrons are detected than backscattered electrons at low accelerating voltages.

2.7.2 Condenser Lens Setting and Image Quality

Probe current is adjusted by changing the Cond Lens 1 value. To increase probe current and beam size, select a lower Cond Lens 1 value (larger spot size). Information necessary for setting of Cond Lens 1 is as follows.

(1) Resolution: The electron beam size is smaller with a higher excitation value of Cond Lens 1. However, image resolution also depends on the S/N ratio of the image and on the ease of focusing and astigmatism correction.

(2) S/N ratio: The signal to noise ratio is better with lower excitation values of Cond Lens 1.

(3) Charging: Charging of insulator specimens is greater at smaller excitation values of Cond Lens 1.

(4) Signal source: Generally the backscattered electron imaging needs higher probe current than the secondary electron imaging. X-ray analysis needs much higher probe current.

2.7.3 Objective Lens Aperture Size and Image Quality

The objective lens aperture (see figure on page 8) has four openings: 100, 50, 50 and 30 micrometers (numbered 1, 2, 3 and 4). For normal operation, use number 2 or 3 (50 micrometers). The electron optical column of S-4700 is designed to achieve highest resolution with a 50 micrometer aperture. When a larger probe current is required, for example for X-ray analysis, use number 1 (100 micrometers). Resolution may degrade with this large aperture. Use number 4 (30 micrometers) to reduce probe current, for example to reduce charging. Resolution is not improved with the smallest aperture, but the depth of focus is better.

2.8 Preparing Images for Recording

2.8.1 Freezing an Image

The Run/Freeze Button  will freeze an image on the screen. You will need to press the run button to resume scanning after pressing the freeze button or after capturing an image.

2.8.2 Capturing and Saving an Image

(1) By pressing the  button, an image is captured and frozen. Image size depends on the Capture Resolution setting in the Image Setup dialog window (640 x 680, 1280 x 960, or 2560 x 1920 pixels). The image is stored in the **Captured Image** window.

(2) The image is not saved until you click on the  button at the bottom of this window. By selecting "All Save", you will save all of the images in the Captured Image window each with a different suffix (Ex: Iron_m01, Iron_m02, Iron_m03, etc.). You can also chose to save just the selected image by choosing the "Off" save option in the lower right hand corner of the save window.

(3) Images are usually saved in the d drive: d:/Image/Group name/Your name. You may need to create a folder for yourself and your group if they do not already exist.

2.9 Shut Down Procedure

2.9.1 Reset Parameters to their Normal Settings

In order to leave the instrument in the same state that you found it, set these common parameters back to these typical settings:

Vacc = 2 kV
Operation Mode = Normal
Condensor lens 1 = 5.0

Focus on your sample, so that the next sample introduced will be close to in focus.

2.9.2 Turning High Voltage Off

(1) Open the **HV Control** dialog window by selecting **HV** command in **Setup** menu.

(2) Click the **OFF** button in the **HV Control** dialog window.

2.9.3 Setting the Stage at the Specimen Exchange Position

(1) Click the **Go to Home** button in the **Stage Control** dialog window (accessible from the **Operate** drop down menu). The specimen stage is then moved to the

specimen exchange position, and the color for the indicator button is changed to green.

(2) Make sure to reset the working distance to 12.0 mm on both the **Column Setup** window and the Z knob control on the instrument.

2.9.4 Removing a Specimen

(1) Turn the **EXCHANGE VALVE** lever to **O** (Open).

(2) Looking into the specimen chamber, push in the exchange rod and screw it into the threaded hole of the specimen holder. (Do not screw in with too much force or the threads may be damaged.)

(3) Pull out the rod fully. Turn the **EXCHANGE VALVE** lever to **C** (Close).

(4) Press the AIR switch on the column evacuation control panel. Check that the specimen exchange chamber has reached atmospheric pressure (about 10 seconds).

(5) Open the specimen exchange chamber. Release the mechanical lock by slightly pushing the exchange rod.

IMPORTANT: **Wear gloves to handle the specimen holder.**

(6) Holding the specimen holder, turn the specimen exchange rod counterclockwise and unscrew it completely.

(7) Pull out the specimen exchange rod fully.

(8) Close the specimen exchange chamber and press **EVAC** switch on the column evacuation control panel.

3. References

Significant portions of the text and figures above were taken from the following sources:

Goldstein, J.; Newbury, D.; Joy, D.; Lyman, C.; Echlin, P.; Lifshin, E.; Sawyer, L.; Michael, J. Scanning Electron Microscopy and X-ray Microanalysis, Springer: New York, NY, 2003.

Hitachi Model S-4700 Field Emission Scanning Electron Microscope Instruction Manual

FE-SEM Training Manual (Hitachi High Technologies America)