

STANDARD OPERATING PROCEDURE

HITACHI S-3400N-II

SCANNING ELECTRON MICROSCOPE

(SEM)



Resolution 3 nm (SEI) and 4 nm (BSI)
Variable Pressure

Oxford Instruments Energy+ (EDS), resolution ≤ 136 eV at MnK α FWHM

Oxford Instruments INCAWave (WDS), resolution ≤ 10 eV

Samples up to 20 cm in diameter by 11 cm high and weighing up to 3 kg

Emergencies

Power failure

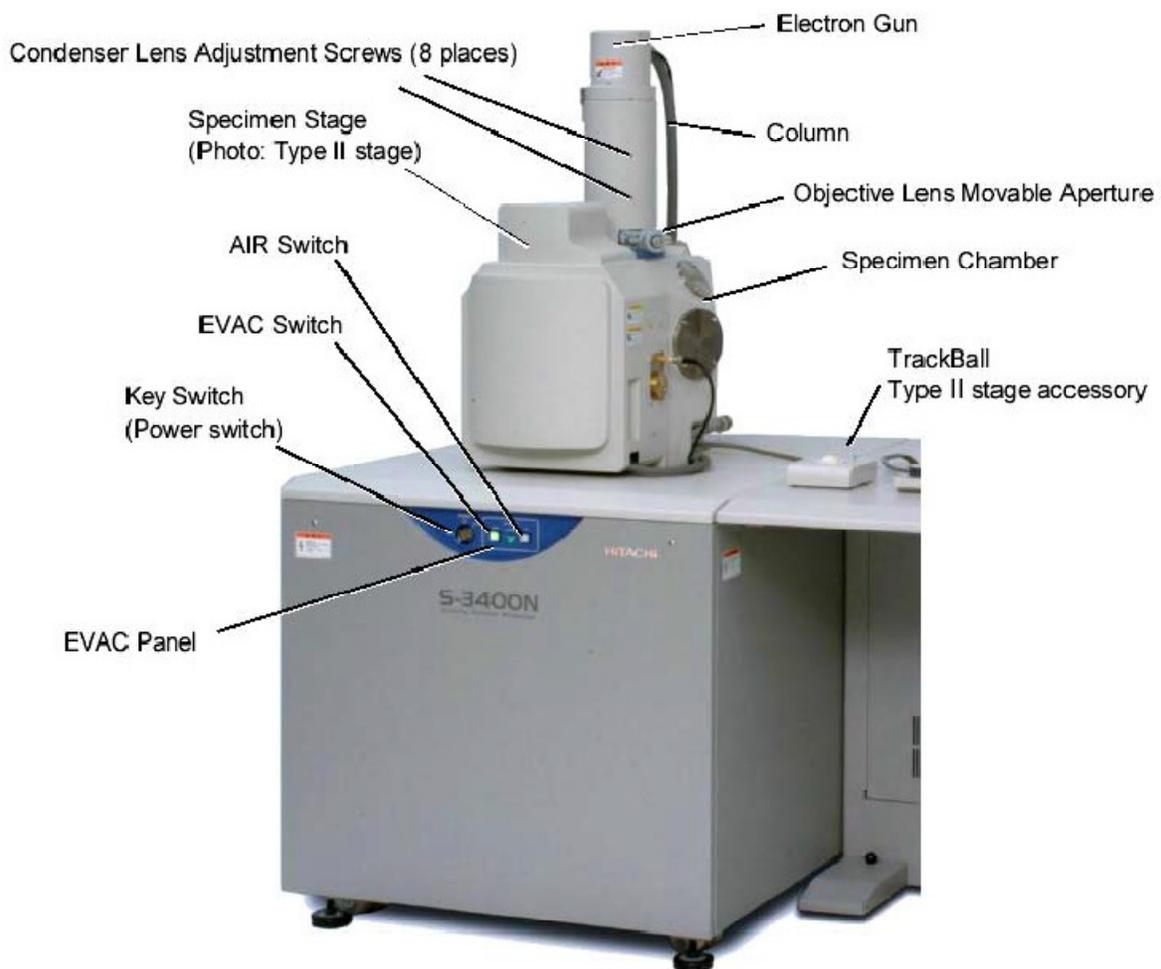
The microscope will shut down safely. When power is restored, it is necessary for BSCMC staff to restart the microscope manually.

Cooling Water Suspension

The microscope does not use cooling water of any kind.

Emergency Stop

In case of fire or severe flood, shut off power using the key in the main unit below the column and/or the lever on the safety switch located behind the SEM. Do NOT remove the key! Avoid these methods of shutdown unless absolutely necessary.



Start-Up Procedure

Preliminaries

Make sure the SEM and computer are ON.

If the SEM Manager software (PC_SEM) is not already running, double-click to load it.

Username: S-3400

Password: <leave this space blank>

Ensure that the SEM vacuum is ready (EVAC green light in main unit). If not, wait for it.

Ensure that both green and blue LEDs are lit on the EDS detector.

Always use gloves when handling samples, stubs, and mounts.

Always clean stubs thoroughly before mounting samples.

Minimize the use of double-sided tape and non-conductive adhesives for sample mounting in order to avoid outgassing.

If analyzing powders dispersed directly on a stub, ensure that the stub is well-polished first, as machined lines and scratches will detract from the powder images.

Check the amount and quality of oil in the rotary pump.

Make sure that the rubber tubes in the evacuation system are not loose or disconnected.

Close the drain cock of the compressor.

Is the compressor stop valve open?

Is the breaker on the safety switch turned on?

Are all breakers on the system power distribution board (back of main unit) turned on?

In the event of any problems or unexpected conditions, contact BSCMC staff and do not attempt to use the microscope.

Loading a Sample

Mount your sample on an appropriately-sized round aluminum disc.

Screw the aluminum disc into a brass stub.

Slide the stub into the sample mount, which has a dovetail base.

Make sure the sample stage is at the home position
(a blue marker next to the **Home Z65** box in the upper-right corner of the screen).

Make sure that the high voltage is **OFF** and **emission** current = 0.0 μA .
(a blue marker next to the OFF button in the top-left corner of the screen).

Air the chamber
(**AIR** button in upper-right corner of screen OR **AIR** button on main unit)

Wait for the chamber to reach ambient atmospheric pressure (~ 1 minute). A beep will sound at the end of the venting cycle.

Measure the height of your sample with the plastic measuring tool.

Slide the sample-holder assembly onto the stage.

Enter the height (above standard) and width (largest diameter) of your sample into the sample information window.

Close the chamber and evacuate it (**EVAC** button in upper-right corner of screen OR **EVAC** button on main unit). Green light will flash until vacuum is ready (~ 1 minute).

Setup

Click on the **Setup** icon in the lower part of the control panel and select the **Optics** tab. Select the desired voltage and beam current.

A high voltage will generally increase resolution but reduce the surface selectivity of the signal.

A low beam current will generally increase resolution but decrease the signal-to-noise ratio.

Ensure that the filament range is set to **Mid** and the **Auto Gun Bias** option is selected.

Turn the high voltage **ON** (top-left corner of the screen).

Select an appropriate aperture (2 or 3 for low- to medium-magnification) by turning in the correct aperture on the column. Available aperture diameters are:

1. 150 μm
2. 80 μm
3. 50 μm
4. 30 μm

A small aperture increases the depth of field but may reduce resolution.

Click on the **ABS** button to perform automatic filament saturation, beam alignment, brightness & contrast, and focus. You can always manually adjust brightness, contrast, and focus with the knobset. You may need to focus manually on samples with low contrast.

Select an appropriate **working distance** (focal length). If you change WD, remember to enter the new position in the **Z** control box, which is in the **Stage** tab of the control panel, and that the magnification will change!

A small WD increases resolution but decreases the depth of field.

For chemical analysis, a WD of 10 mm should always be used.

Click the **DeGauss** button to remove residual magnetic fields from the objective lens. It is recommended that you degauss whenever focus is changed substantially.

Alignment

Set the magnification to at least 5,000x or, ideally, the highest magnification expected to be needed and bring a recognizable spot to the center of the screen. Perform focus and stigmatism corrections to sharpen the image as much as possible.

Click on the **Align** button under the Adjust label at the top of the screen.

Click the **Beam Align. Shift** radio button then click **Auto**. This operation can also be done manually by maximizing the brightness of the image with the **STIGMA/ALIGNMENT** knobs.

Click the **Beam Align. Tilt** radio button then click **Auto**. This operation can also be done manually by maximizing the brightness of the image with the **STIGMA/ALIGNMENT** knobs.

If the moveable aperture has been changed, align it manually using the two knurled screws so that the brightness of the focused image is maximized at relatively low magnification. There is no need to perform this operation unless you have changed the aperture or suspect its alignment.

Click on the **Aperture Align** radio button. The focus and image position will be wobbling periodically. Bring an identifiable spot to the center of the screen then click on the **Auto** button in this window to automatically align the aperture so that the motion of the image is minimized. You can also align it manually with the **STIGMA/ALIGN** knobs.

Click on the **Stigma Align. X** radio button next and click **Auto**. You can also perform this alignment manually with the **STIGMA/ALIGN** knobs.

Click on the **Stigma Align. Y** radio button next and click **Auto**. You can also perform this alignment manually with the **STIGMA/ALIGN** knobs.

You may need to iterate the above alignment procedure (from aperture alignment) for best results.

Click on the **AFC Align.** radio button and minimize the motion in the image using the **STIGMA/ALIGN** knobs.

Operation

Always focus at a higher magnification than that at which your image will be recorded.

To maintain optimal imaging conditions, the probe current should be reduced as magnification is increased.

Resolution will be maximized for high kV, small probe current, and large aperture size.

Depth of field will be maximized for large WD, low magnification, and small aperture size.

Contrast can be increased by increasing the beam current or increasing the scan time.

To change the scan rate, click on the **Scan** buttons at the top of the screen. Slower scans give higher quality images. F modes are fastest scans possible. TV modes are television-equivalent scan rates. The different F and TV modes differ by the amounts of frame averaging. S modes are slow scans ranging from 1 s/frame (S1) to 160 s/frame (S6).

To switch to BSE mode, click on the **Image** tab of the control panel and click the **BSE** radio button under the DETECTOR label. Make sure you are using a slow scan rate, S2 (4s per frame) or slower to avoid excessively noisy images..

To view SE and BSE images simultaneously, click on the **Dual** radio button under **SCREEN MODE**.

To set up the way images are recorded, click on the **Setup** icon in the lower part of the control panel and select the **Record** tab of the Setup window. Tick all experimental details to be recorded.

To record an image, click on the **Capture** icon (next to the Save icon). The captured image will appear at the bottom of the screen. To save it, click on the **Save** icon in the panel next to it. Do *NOT* leave image files on the pc. Images left on the pc will be deleted periodically.

End of Session

Turn **magnification** down to ~ 50x.

In the Image control tab, make sure the **SCREEN MODE** is small and the **SE DETECTOR** is selected.

Turn OFF the **high voltage** (top-left corner of the screen).

Return the stage to the home position (**Home Z65** box in the upper-right corner of the screen).

Vent the chamber
(**AIR** button in upper-right corner of screen OR **AIR** button on main unit)

When it is completely vented (~ 1 min), open the chamber and, wearing gloves, remove your sample holder.

Close the chamber and evacuate it (**EVAC** button in upper-right corner of screen OR **EVAC** button on main unit). Green light will flash until vacuum is ready (~ 1 minute).

Remove sample from holder. DO NOT STORE SAMPLES IN ENGR 108 OR MEC113/113a.

Shut off the Oxford Instruments pc.

Turn off both pc monitors.

If you are the last user before a weekend or long break, shut off the Hitachi pc and power down the SEM using the key on the main unit. Do *NOT* remove the keys!

Record hours in the on-line BSCMC log.
<http://coen.boisestate.edu/vcalendar/BSCMC/index.php>