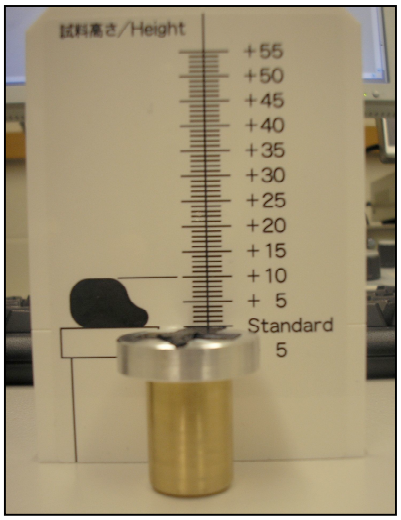
Introduction to Hitachi S3400N operation

1. Safety (of machine) Issues

A. Sample height is #1 concern, if incorrect diameter/width and especially height is entered in software, the backscatter detector, CL detector and EBSD camera can be seriously damaged/ruined.



There are 3 pieces involved in the sample holder, 2 are shown together in picture to right. The 3rd is a flat aluminum piece with a "dovetail" grove in the bottom that slides onto the stage and has a hole to accept the rest; the top 2 pieces are screwed together (brass rod bottom, round aluminum plate top) and these slide snugly into the dovetail base.

The user must use the white measuring tool to measure each and every sample that is to be put in the machine. Place sample on whichever mounting holder you use and then measure **the total height of the sample plus the holder**, relative to the bottom edge of the ruler, with the maximum height the sample reaches. For example, in the case here, there is piece of sticky tape with 100 micron fragments on it, so "standard" is the appropriate height (=0 mm). If I mounted it on a shorter brass rod, it might measure -5 mm.

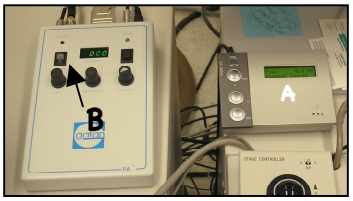
Do not forget to measure the width or diameter of the <largest of> either the sample or the holder for the sample: for the case here, it is 26 mm. The software calls this "Size".

Note: this system works only if you insert the sample (on its holder setup described above) **down all the wa**y on the dovetail holder. If inserted sloppily and half way in, there could be serious damage done.

There is ONLY software protection IF and ONLY IF the height is entered correctly in the software. The stage WILL crash into the backscatter detector as it will only stop AFTER it makes electrical contact. Damage will have been done.

(Note: there are two different sizes for the specimen base, the brass piece. You would not use the shorter one for a thin sample, as there would not be a valid height to enter in the software).

B. You must ensure that there are no detectors inserted which could be collided with. Thus, both in starting up, and in finishing up, you must observe the following procedure:



(1) Verify that the EBSD detector is retracted –A-- (0.2 mm on the small box) and the power on the KE box is OFF.
(2) Verify that the CL mirror is retracted – blue knob is all the way (pick correct one): (a) counterclockwise ? (b)clockwise? And the high voltage (HT) is OFF -- B.

2. Computer structure

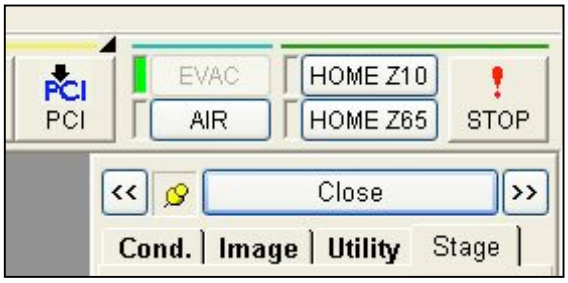
A. There are two different computer systems. The SEM uses the computer to the right of the user, with a outer door that hides it. The left monitor goes with the bottom right computer. The EDS

computer on the other hand, sits to the far left on the floor. The EDS monitor is the right monitor. Remember: left monitor to right computer; right monitor to left computer.
C. Once there, click the PC-SEM icon on the left desktop, and it will automatically take you to the PC SEM Manager environment. Master login is S-3400 – only need to do it if the computer has been shut off. Otherwise it stays active. Login (usually not needed) is johnf
D. Storage of images must be done with attention paid to first time, to locate the proper folders on the D drive, with individual users having folders under name of professor or workgroup -- D\All SEM User Files\ [groups here] \ [your own named folder]
E. We'll talk about the EDS computer later.

3. Start Up
A. Can you see anything on the left monitor? If not, first turn it on to see computer state, i.e. to see if computer is turned on/off or just sleeping.
B. Cold Start Up – Not Normally Needed! (Key off: pumps and computer off). Turn Key to ON and then to START (like car ignition switch) for an instant, then EVAC to start pumps pumping, turn on left monitor, then follow C below
C. Warm Start Up – Not Normally Needed (Key ON: computers off, but pumps running): open Hitachi computer door, push button to turn on;
D. Hot Start Up – Normal state (Key on, computers on, pumps running). Nothing needs to be done.
E. If Computer shut down, you will need to start up. At Windows prompt, enter geouser and password; The Hitachi PC-SEM operating system will start up. If not on desktop, check menu-bar to see if hiding. If not running, then click the PC-SEM icon on desktop, and login with account name "johnf".

>> System can reach high vacuum (turbo pump) in 5 minutes from cold start.
>>AIR button means "vent" chamber; EVAC button means "pump" chamber

4. Steps to open machine to place new sample:

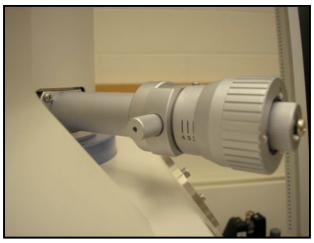


A. If high voltage is on, turn it off. Wait three minutes for filament to cool down before venting chamber. While waiting, follow steps B-D.
B. Turn on the chamberscope IR light (right button on box below little monitor).
C. Be sure CL and EBSD detectors are retracted (see other sections for details).
D. Move the stage to the safe position, Z65 by clicking HOME Z65 at top right menu bar (this drops stage down away from detectors).
E. Click the AIR button next to Home Z65. Wait until you hear the second signal in about 2 minutes; grasp door by both sides and pull toward you.
F. You should have already mounted and measured the size (diameter and height) of your sample, per the instructions in the Safety section. Write them down if your memory is poor. Insert sample (see section on sample holder). Hold door closed while pressing the EVAC button above the AIR button you pushed to vent. Let go the door once the 'sucking' sound stops.
G. A window will pop up and ask you to give the sample size. Click "Specimen" and 30 seconds later you will get another window where you enter the Diameter ("Size") and Height in mm. Enter the correct values and click OK.
H. For general EDS and back-scattered imaging, click the Analysis button on the **STAGE** menu to move the sample to a nominal "working distance" of 10 mm (10 mm below the bottom of the pole piece, or 6 mm below the bottom of the BSE detector). High resolution SE imaging may require a

working distance of 6-7 mm; CL and EBSD will be different and you will those instructions separately. You want to watch the sample rise up and have the cursor ready to click the red! STOP button if you see the sample rising too close to the BSE detector. Normally this should not be a problem but IF YOU ARE NEW OR WORKING WITH NON-UNIFORM SAMPLES OR HOLDERS, YOU MUST BE EXTRA CAREFUL.

5. Set aperture appropriate to your project:

There are 5 options: 4 different apertures, 1 thru 4, plus "none" or wide open. If you are not sure, see what it is set to currently and if close to what you want, leave it there. It is fairly simple to change, but you need some training for alignment if you change the aperture.



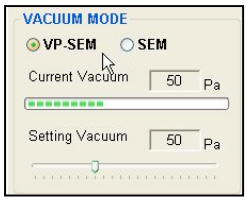
0 – none – EBSD, EDS
1 - 150 μm EDS, CL
2 – 80 μm SEM
3 – 50 μm high resolution SEM
4 – 30 μm highest resolution SEM

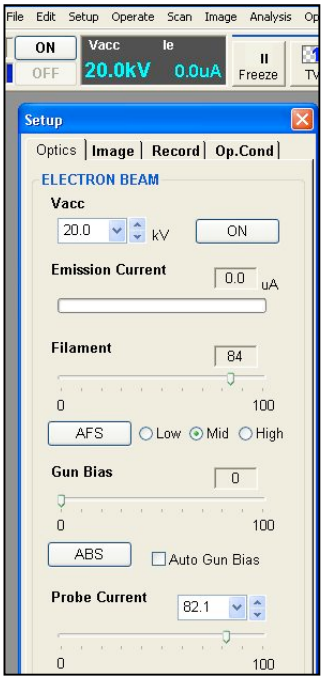
6. Set Image Type

In right vertical menu bar, click Image at top if necessary and then select the image type (SE, BSE, CL=other2, forescatter detector=other1). For initial alignment, if you have a sample with topography (not polished), SE is best. If polished but not perfectly mirror finished, SE still ok, or BSE may be fine.

7. Check/set the vacuum range:

There are 2 ranges that you view/set on the far right vertical menu bar, under Cond: SEM = High vacuum (normal, traditional) and VP-SEM (variable pressure, where air is bled into the chamber, sometimes called "environmental" mode). VP-SEM is useful if samples are non-conductive electrically and not carbon-coated; however, the only detector than can be used for imaging is the BSE detector, versus for high vacuum mode where both SE and BSE detectors can be used. You cannot get as fine scale detail with BSE detectors as SE detectors (but may not be a big deal for your samples). The other concern is use of EDS: in VP-SEM, the electrons bounce around a lot and hit other regions, so the x-rays are also from other areas and not necessarily from the area you are aiming at. However, for EBSD, it is advantageous not to have any carbon coat, so VP mode is standard for EBSD. Set the pressure to 15 P a.



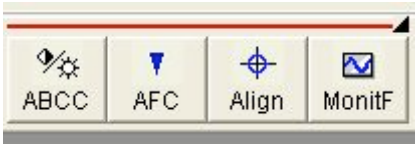


There is a message on the bottom left of the screen that tells you if the vacuum is or is not ready, i.e. for turning on the high voltage.

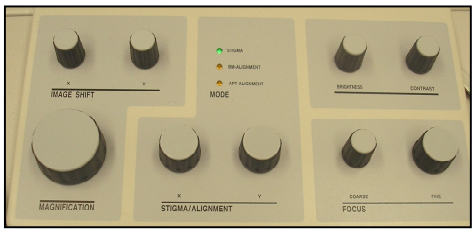
8. High voltage ON and initial setup

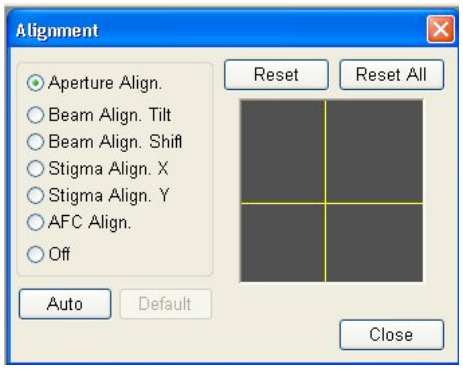
A. If Vacc Ie window Blue and ON active, click ON to turn on HV to the last used value (here 20 kV). Typical values 15-25 kV (high for EDS if

spatial resolution not an issue). If different kV desired, set it first by clicking on Vacc window, before clicking ON.
B. Ie= emission current should be between 80 and 120 microamps (if filament installed 'properly' this should be ok), else need to realign filament externally, or move filament inside Wehnalt up or down (up to get more current, down to get less current).
C. On the Stage window (click tab if not on top) on right menu bar, click the Analysis button to move to 10 mm working distance, if not already there (i.e. Z10 before).
D. Gun bias - leave this at zero for the moment. Probe current should be >50-60 to start
E. Assuming you have an image, Click AFS (auto saturate the W filament); choose "med" (medium) for normal; high for highest resolution images (but this wears the filament out so don't use unless necessary)



F. Click the AFC (auto focus) on the top menu bar, wait, if see nothing go to lowest mag to see something, then manually focus if necessary with coarse focus knob, and fine, going to higher mag. You want to do all this in SE mode, so if polished sample, need some contrast, e.g. crack or pit.
G. Click ABCC for auto brightness/contrast IF there is enough in image (else do it manually) -- may have to go back/forth AFC / ABCC until you get close.
H. **Also may want to do AFS again, as the saturation is based upon optimizing the signal from an image**.
I. Probe current/"size": smaller number is lower electron flux on sample so less signal but smaller probe size; 100 is highest; for EDS >50; high for EBSD, CL.





11. Fine adjustments
A. At higher mag (best in SE, on tiny blebs, regular shapes preferably), adjust the Stigma x and y knobs for a sharper image.
B. Click Alignment in top menu bar (middle), and image will be wiggling. The top selection =Aperture Align. You adjust the 2 knobs on the aperture rod sticking out, first with one to minimize the wobble in one direction, then the other knob for other direction. These should only be small adjustments, you do not want to turn the screws a whole lot. They should be pretty close already.
C. Beam Align Tilt and Shift: want to maximize brightness of image
D. Stigma Align X and Y: minimize side to side wobble
E. AFC Align ??
F. Reset button: recenters the adjustment if you are far to one side (thus, gives more adjustment potential)?

12. Other possible operations:
Focus Link: to keep in focus (roughly) if you change the working distance
DeGauss: if change mag a lot, DeGauss lenses to get rid of hysteresis, esp acquiring high res image.

13. Image setup: Set optimal conditions
A. Can use Red 1/2 = reduced size for focusing; can split screen, SE and BSE
B. Can use line profile to manually set brightness and contrast
C. Record: can add info to label as base of image; can embed into image.
D. Operating conditions: can save to load later under specific sample type label. Use button for setup and loading. (Guide provides step by step help)
E. For BSE, turn off the IR camera
F. Can superimpose signals with Signal Mixing
G. Show Histogram (under Image at top menu): useful for adjusting brightness contrast

11. Image acquisition
B. Select the speed at the top center (add more detail; can set averaging etc)
C. Select the pixel dimension top right
D. Set up: optics image report and operating conditions
E. Click the Go arrow.
F. Thumbnails show up at bottom: click Save and give name/directory
G. When finished, click Run to go back to active scan

12. Navigation
A. Register: saves points for later retrieval (save as stage history)\
B. Under Navigate, capture low mag image for navigation. Register by finding coincidence points on image and sample
C. Memory: can load points for sample if latger loaded on stage in exact same location

Highest resolution SE/BSE images
1. AFS = high (e.g. >2500x)
2. Vacc = lowest feasible for type detector (e.g. for SE, 1-5 keV; BSE 5-10 keV)
3. aperture - 30 um
4. Probe current/size - small value (enough to get an image)
5. If sample charging, use rapid scans that are averaged together rather than one slow scan
6. BSE: generally COMP mode (summing all); be sure NOT inverted!

EDS (e.g. x-ray mapping)
1. AFS = medium
2. Vacc = 20-25 keV (unless need 30 keV for overvoltage)
3. aperture = 80 or 150 um
4. Probe current - 50-100

Other odds/ends
A. Raster Rotate - electronically rotate in image frame
B. Tilt compensation: dynamic focus, electronically compensate for 'stretched' tilted image
C. Can navigate with map of sample dimensions. X shows position.
D. Click Eucentric to bring new position to center, to rotate around it

1) *** High pitched alarm: LN sensor for Thermo EDS detector. Consult John or Phil.