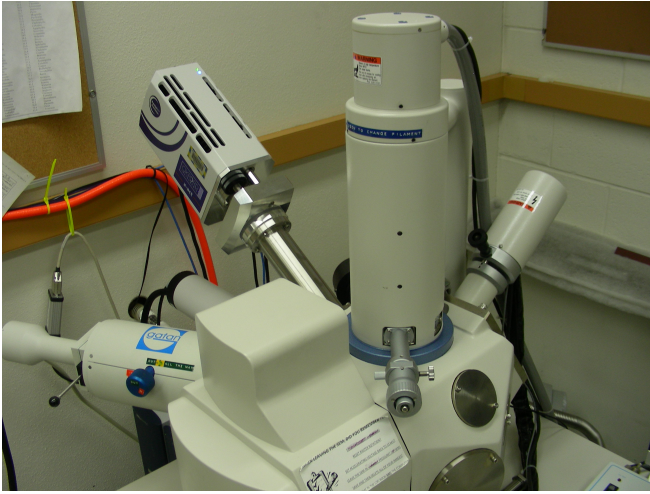


## Geology 777 Lab 1: Introduction to SEM and Hitachi S3400N operation

For the next several weeks you will be learning about the proper operation of the Hitachi Variable Pressure S3400N Scanning Electron Microscope in Weeks 308. Each student will have various opportunities, from “cold start up” to alignment and focusing and then imaging with the various detectors. Today we will set up and run in “normal” high vacuum mode.

The main point in lab is to pay attention and ask questions. I will be demonstrating a variety of operations, and then each one of you will be involved in doing some activities that will mainly be aimed at getting you “up to speed” for acquiring images. This week we will focus on setting up the column for “normal” high vacuum operation.

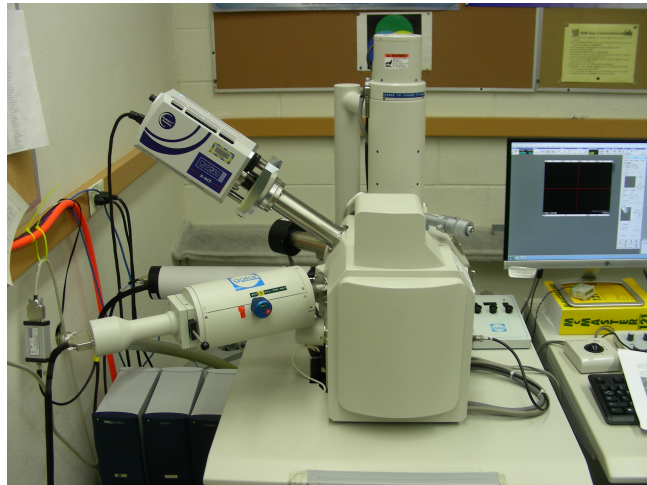


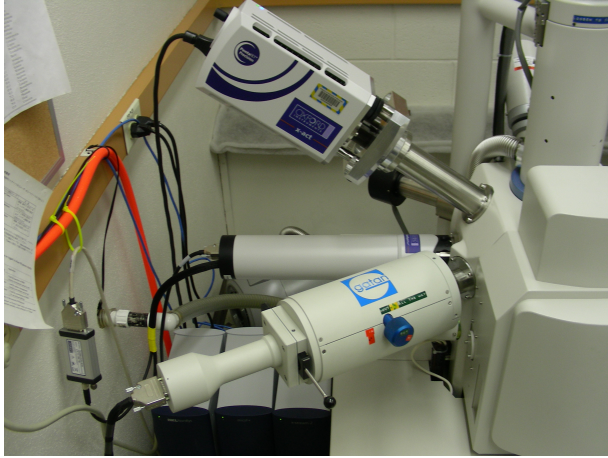
1.Orientation: On the picture to left, locate/label:

- A) Gun
- B) Column
- C) Aperture Holder
- D) ESED (Environmental Secondary Electron Detector)

On the picture to right, locate/label:

- A) EDS detector
- B) CL Detector
- C) Aperture Holder
- D) Gun
- E) Column





In picture to left, locate/label:

- A) EBSD Detector
- B) EDS Detector
- C) CL Detector
- D) Secondary (SE) Detector

## 2. Gun: Learning about the Wehnelt and W filament

We will now go into some detail with some specific features, starting with the Gun: Filament, Wehnelt cap, and Anode. After venting the instrument (and waiting for the filament to cool down if it has been in operation!), we can “open the gun” and examine the Wehnelt and Tungsten filament inside it. This is all done with gloves on. This is not something most users do.

## 3. Sample insertion

- a. Protection - Software: measure sample + holder; position in machine; enter in correct height; raise sample to final height; check sample passes under gauge; close door.
- b. Vacuum setting = high vacuum; then Pump (evacuate)
- c. Turn on chamberscope
- d. Check that raster rotation is off
- e. Dynamic Focus mode should be off (Operate->Image Adj->Tilt Comp)

## 4. Set up column:

- a. Set HV
- b. Brightness/Contrast, Focus, Stigmation
- c. Saturation of filament
  - i. Filament image, with guideline max value on whiteboard
- d. Gun Bias
- e. Probe Current

## 5. Apertures

(#1 – 100  $\mu\text{m}$ , #2 – 80  $\mu\text{m}$ , #3 – 50  $\mu\text{m}$ , #4 – 30  $\mu\text{m}$ )

- a. Aperture should be set to Zero. If not, ask experienced user how to set to zero. Do not adjust any of the knobs without permission.
- b. As a general rule for new users, do not use any apertures without assistance.
- c. And if you ever do, do not adjust more than 5-10 degrees (fraction of a turn)

## 6. Stage controls: Height vs X-Y

- f. Height: (Physical working distance vs. focal working distance)
  - i. When you insert the sample, it is located at a “physical working distance of 5 mm”. However, this is TOO short for most users. You should drop the stage to 10 mm working distance. What is the “code word” on the **Stage** tab for this? \_\_\_\_\_
  - ii. You can drop the stage lower, all the way to ~50 mm working distance. You can see a much wider field of view, though lose spatial resolution. Depends what you need.
  - iii. To do CL, you need to be VERY careful with stage height. Start with working distance of 15 mm.
  - iv. NOTE: Very stupid Hitachi confused wording: on Left side under the column conditions, there is “working distance”. This is NOT a linear working distance measurement. This is the focal point for the electron beam, attained by turning the “focus” knobs, which are formally known as “objective lens adjustment”.
- g. Sample orientation: Top of image (“north”) is toward the left wall
- h. X-Y: There are several ways to move in X and Y:
  - i. Manual controls (trackball)
  - ii. Click On Stage Image
  - iii. Jog arrows on Stage Controls (you can set the ‘jumps’)

## 7. Imaging

- i. Types
  - i. SE
  - ii. BSE
  - iii. CL (not this week)
  - iv. ESED (not this week)
- j. Files
- k. Histogram:
  - i. 8 bit images (256 gray scales)

8. Student imaging exercise: the instructor will provide a sample, and each student should spend ~5 minutes acquiring several SE and BSE images of the material, and save those images to a class folder.

9. Shutdown: Turn off HV, wait 3-5 minutes; vent; remove sample; pump—leave chamber in pumped condition.

10. Computers in room; transferring files to Weeks server A320; memory sticks only in computer across the room.

Your assignment:

Turn in at next week’s lab session (or at lecture if completed):

These pages with images labeled; and a writeup including 3 paragraphs:

1 short paragraph on instrument and detectors;

1 short paragraph on column setup and alignment

1 short paragraph on imaging (demonstrate you understand the difference between SE and BSE images)