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JEOL JSM-IT200 Operating Instructions

Written 08/12/21 B.B.

Location: **1710 Gilman Hall**
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Safety

All researchers working in 1710 Gilman Hall must complete the EH&S course: *"Fire Safety and Extinguisher Training"*. When preparing samples in this room, please wear all appropriate personal protective equipment. Safety glasses and rubber gloves are available in 1710 Gilman Hall. If solvents are involved, please prepare your sample in your own lab space.

Properly dispose of plastic sharps in the container provided. All of the computers in this lab have direct links from the desktop to MSDS sheets, the EH&S Laboratory Safety Manual and to the CIF Safety Manual.

Fig.1 JEOL JSM-IT200 Scanning Electron Microscope

Introduction

The JEOL JSM-IT200 is a rugged scanning electron microscope with a tungsten source capable of resolution below 10 nm. In addition to imaging, the system is equipped with X-ray elemental dispersive spectroscopy for elemental analysis which can be both qualitative and quantitative. The system is controlled from a single software package.

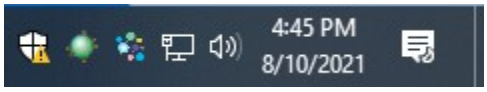
This guide is presented as an overview and concise flowchart; for some operational details you may need to refer to other guides. There are some sample types which require pretreatment to be imaged successfully, such as coating with gold to provide conductivity.

The Desktop

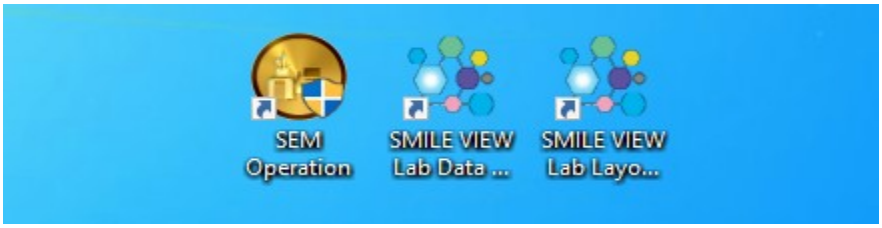
The instrument computer should always be logged on as .*cif*. Do NOT logoff when you are finished- you will use the *LockScreen* program to log on and off the computer. This program also tracks instrument time automatically.

> *In the event the computer logged off unexpectedly or installed updates, the password is jsm-it200.*

The notification section of the Taskbar should show the *OperationServer* icon. This is an important background process for SEM operation. If you don't see it in the taskbar, restart that process from the desktop icon.



All programs you will need are at the bottom of the desktop:



The *SEM Operation* program drives the microscope, and the SMILE VIEW programs are a data file directory and report layout editor.

Directories and File Structure

During your training session, a data folder will be established for you at this location:

E:\DATA\IT200*<your user name>*.

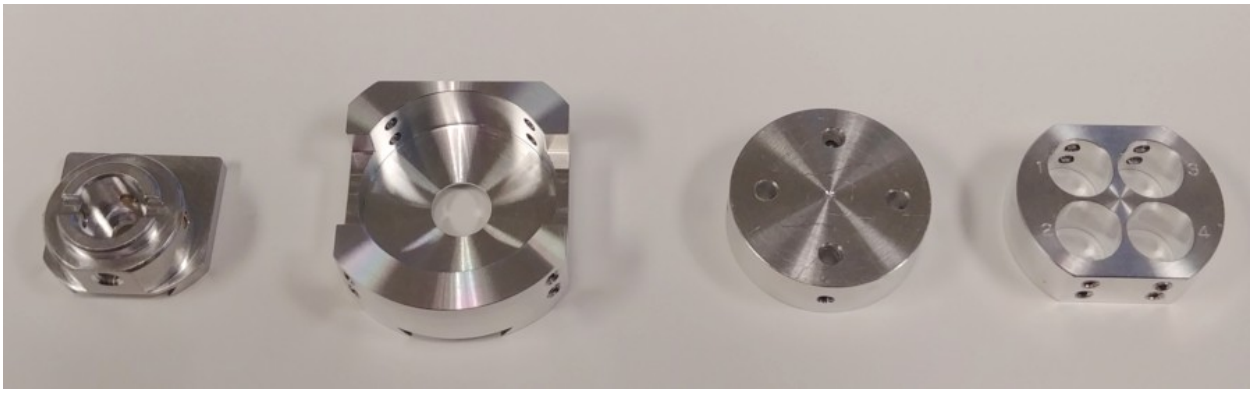
All data files must be stored in this location. Filename format is up to you, but it is suggested to use date codes to best store your images, EDS data, etc. This partition will be backed up daily to F:\

Setting up a Measurement

In the event of errors: If at any time you notice a strange response or error state, please proceed accordingly: open **Word** and then use a snipping tool (WinKey+Shift+S) to capture the software/error window. Paste the captured region into the date-coded **Word** document. This will allow staff to keep records on problems with the setup and assist all users in the long run with overall system stability.

Filament Failure: This system is equipped with a hairpin tungsten filament as the electron source. Every few hundred hours, these filaments burn out, similar to a conventional Edison light bulb. If this happens during your imaging, let Brett know right away and he will help you get a new filament installed. There is just a short cleaning cycle to run on the Wehnel cap and we have spare filaments on hand.

Sample Preparation: The SEM should always be under high vacuum when not in use. This maintains all the internal surfaces and hardware. In addition, any time samples and sample holders are going in or out of the chamber, gloves should be worn and the sample holders should be kept as clean as possible. There are two 10mm sample insert holders, a 32 mm frame that can hold a 4-way holder for 10 mm stubs and a standard pin stub insert, and custom sample mounts can be fabricated as well.

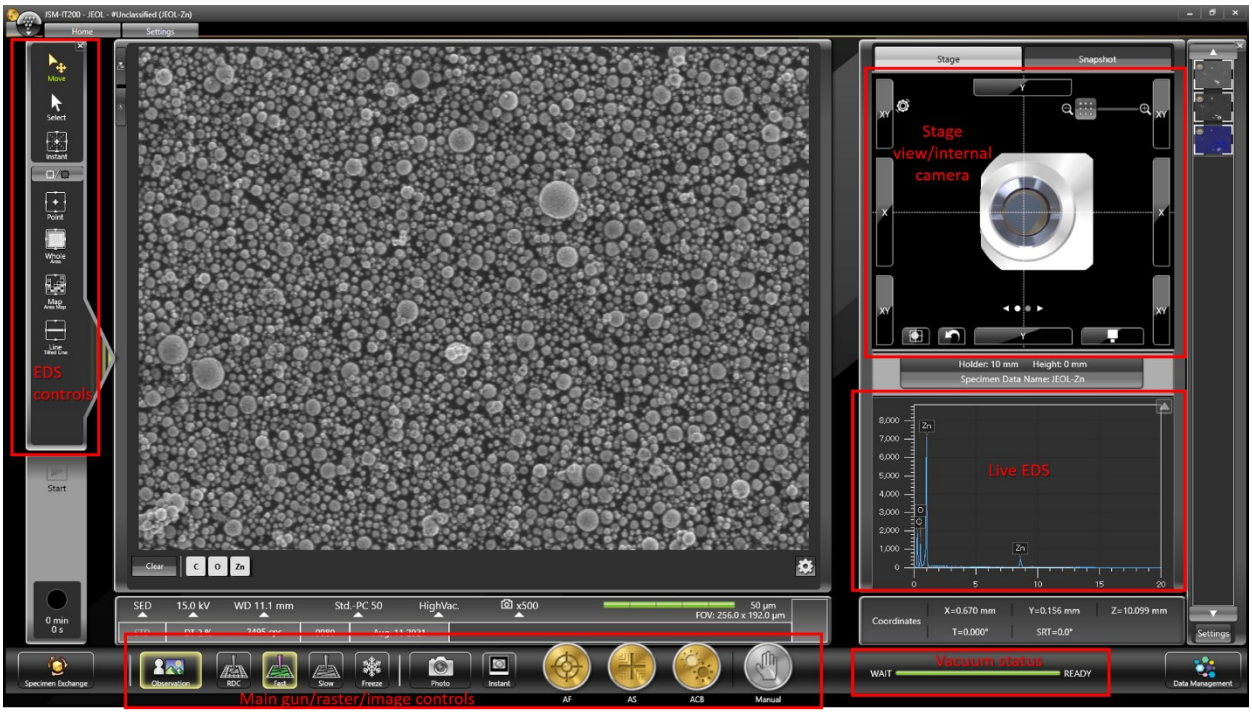


Currently we have 4 sample holders for this instrument, from left to right a 10 mm round holder (with brass inserts), a 32 mm multipurpose mount, a 3 mm pin stub adapter insert and a 4-position 10 mm insert, both of which fit to the 32 mm mount.

> *As a rule, samples must be well secured and moderately conductive for best imaging. Powder samples may not be loaded loosely as they will cause damage to the gun.*

Starting a new sample run: After logging in with the *LockScreen* program, launch *SEM Operation* from the desktop. This program controls all aspects of the SEM.

The main interface is shown below, with several functions labeled in red.



All of the main controls below the live image are available on the manual user interface as well, just find what works best for you. In addition to the stage navigation view in the upper right, there is also a chamber camera available to view your sample inside the SEM. This is especially useful for tilting samples carefully to avoid damage. All EDS controls are at the right of the live image, though EDS will be covered in a separate manual.

To load your sample, press the *Specimen Exchange* button in the lower left corner. After confirmation this will begin venting the chamber. *Don't be alarmed as both the turbo and rough pump will turn off during this time.* A series of images will prompt you to do the following:

1. Open the chamber fully when atmospheric pressure has been reached
2. Provide sample information

   Give your sample a name, as well as select the sample holder being used. Prior to inserting the sample, use the JEOL ruler to measure the height of the sample above the holder. This is critical to not crash into the backscattered detector or other internals.

3. Select instrument settings

   The working voltage, probe current, and other settings will depend on the type of information and resolution you are hoping to achieve, as well as the electron density of the sample. This may be skipped if not needed.

4. Take a sample overview photo

The software will ask you to manually move the Z-axis control to a height based on the height of your sample and the stage, and record a photo. This photo can be used to get a top view low mag image of your sample to help with navigation during imaging.

5. Close the chamber door and evacuate the instrument

   It is a good practice to gently hold the chamber door closed until the rough pump has begun evacuating the instrument.

6. Begin imaging your sample(s)

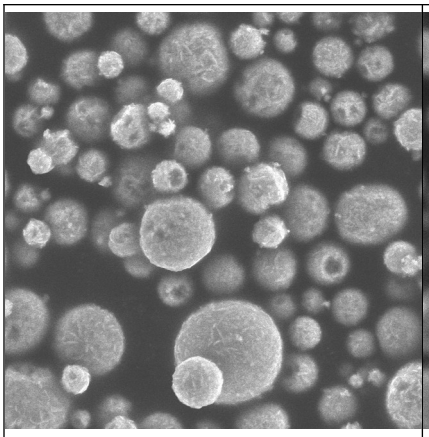
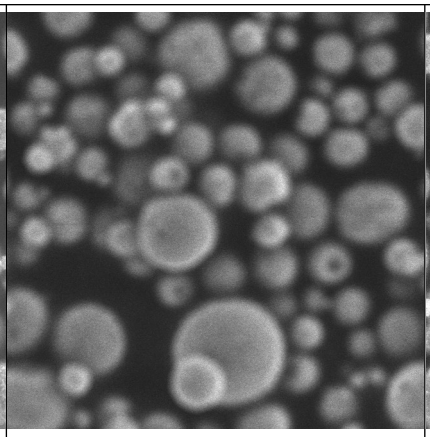
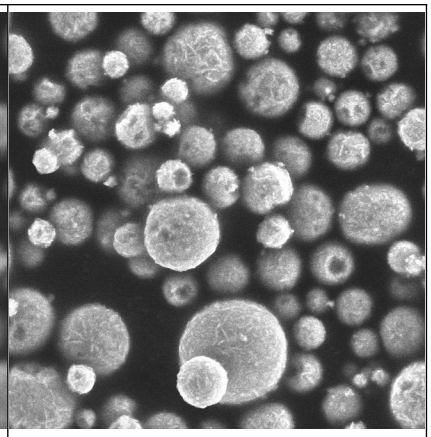
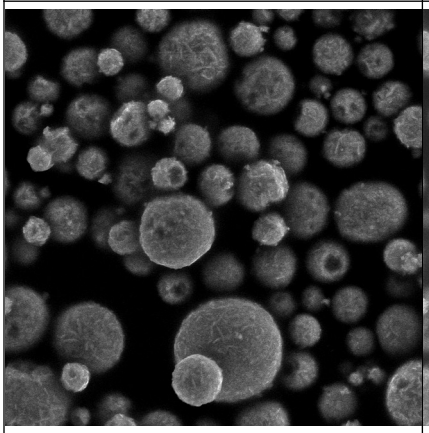
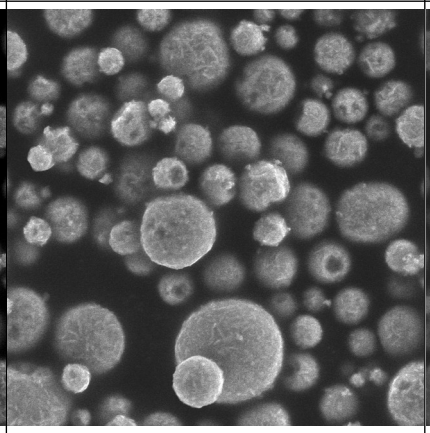
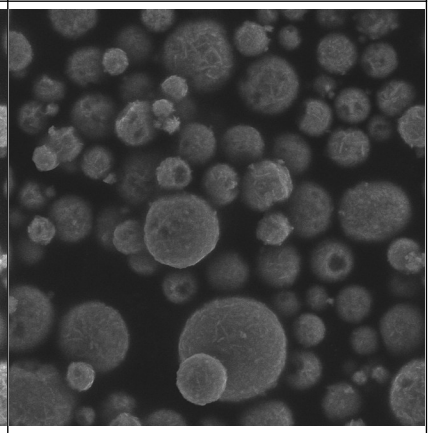
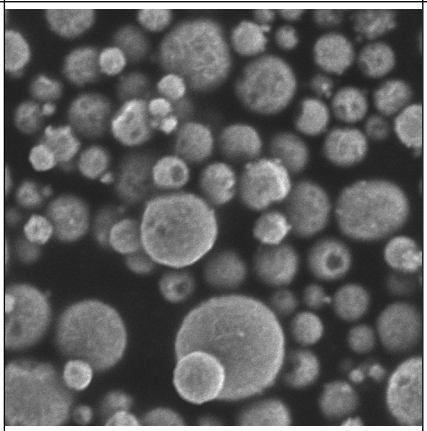
Sample Imaging: Exit the *Specimen Exchange* routine and press the *Observation* button to start the gun. Over the next ~10 seconds an image will begin to form as the beam current comes up and stabilizes. The sample holder is always off to the side when samples are loaded, so use the navigation image to drive the stage to see your samples.

Any time you need a break or aren't actively imaging, please pause the Observation to turn off the gun. This will prolong filament life.

Controls are as shown on screen, such as varying the raster rate and image location. All of these on screen controls are on the *Manual User Interface* as well, so use whichever is easier for you. I tend to operate the magnification and brightness/contrast with the manual controls while moving the stage and other controls with the mouse. The mouse wheel also controls magnification. In addition, clicking the *Manual* button to the right of all the automatic routines allows full control of focus, stigmation, etc with the mouse wheel.

After a few uses, you'll get a feel for what works best for you. The only automatic routine I use frequently is *auto-stigmation* as it does a good job and can be difficult to optimize manually. I also like having the image histogram showing to make sure my settings are optimized. To show the histogram, just select *Home -> Histogram* and it will appear in the upper right of the live image view as a transparent plot with green data bars.

To get an idea what the controls will do to a sample image, here is a gallery of images of the Zn standard supplied with the SEM to show common issues with collecting high quality images.

		
Brightness too high	*Out of focus*	*Contrast too high*
		
Brightness too low	*Optimal conditions*	*Contrast too low*
For optimal brightness, the background areas shouldn't be completely black to where the low signal counts are clipping.	 *Stigmation not ideal*	*For optimal contrast, the histogram should span a wide range of the luminance axis without significant populations at full white (255).*

The best lesson in taking high quality SEM images is time and practice. You'll need to take a few lousy images, analyze them, and note your errors. The instrument has *ImageJ* loaded on it for an easy-to-use image analysis program which has lots of useful tools such as thresholds, line profiles, histograms, etc.

Saving an image: Use *Settings* → *Image* to set up the file path for your images to save to, and change any settings such as image format, etc. If you right click on the *Photo* button, you can select the image resolution and scan time (I tend to use *2560x1920 – 40 s* or *80 s*). There is an Auto-save feature which is helpful as well.

Since images take awhile to raster and save, please do not bump the SEM during image acquisition. Though it is on a floating table, it is still susceptible to vibrations, especially at high mag. If this becomes an issue during regular use, we can look for additional vibration mitigation strategies.

Finishing up: When you have collected your images, vent the system by clicking on the lower right *Wait ---------- Ready* progress bar and click *VENT*, then confirm. Once the *VENT* light goes solid green on the instrument, your sample may be removed. Once that is done, slide the chamber door closed and start the *EVAC* process to pump down the chamber again.

It is a good practice to gently hold the chamber door closed until the rough pump has begun evacuating the instrument.

Once the SEM has reached high vacuum, you may exit the *SEM Operation* software, upload your images (such as via *Cybox*, *my.files* drive, etc), and *Logoff* using the lower right button. It is not necessary to power down the computer or log out of Windows.