Usage Policies Notebook for HITACHI S - 4700
Field Emission Scanning Electron Microscope System

Revision date
September 2014

INRF UCIrvine

Emergency Plan for Hitachi SEM S - 4700

Standard Operating Procedures for Emergencies

Contact information

Person	Phone number
Lab Manager	Jake Hes, 949-824-8239 (day), 562-522-8328 (alternate)
Director	G.P. Li: 949-824-4194 (day), 949-824-2047 (alternate)
Staff	Mo Kebaili: 949-824-8239 (day), 949-494-5892 (alternate)
Super User	Carlos Ruiz (818) 527-6349 (Anytime, voicemail or text only)

Hazardous chemicals, gases, and conditions

Hazard name	Description of hazard
High voltage	Electrical shock, ignition source
N2 (nitrogen) gas	Asphyxiant

Alarms or indications of danger

Alarm type	Condition and response
Pungent or foul smell	Oil back streaming from the diffusion pump or the mechanical pump. Shutdown the tool at once and evacuate the area. Contact the staff and the lab manager.

Emergency shutdown plan

In the event of an emergency, when there is very little time, *press the large red emergency shut-off button at the entrance of the room*, this will shut off the power from the receptacle outlets, *press the large red emergency shut-off button on the SEM tool*, this will shut off the SEM tool. Leave the facility at once, and then contact the lab manager or Staff.

Usage Policies for Hitachi SEM S - 4700

Standard policies for usage

Contact information

The INRF staff or the lab manager can be reached at (949) 824-8239 or (949) 824-9831.

Authorized users

Only INRF registered users who have completed the training and passed the certification on the SEM tool may use this equipment. Users may only use the portion of the system for which they have been trained.

Training

Users must have received direct training from the staff in order to use this equipment. Users are expected to know how to operate the system. Contact the staff for details and to arrange for a training session.

Usage logs

Users are required to log in all their activities in the log sheets provided. All users must log in when they used the SEM (date and time), and when they completed their process in the user log sheets. If users notice anything unusual, they should record it in the user log sheet, and add details in the main comments area on the log sheet. Any maintenance to the tool will be logged in the maintenance log sheets (maintenance staff only).

Safety equipment

As safety equipment for use on the SEM tool, cleanroom gloves and tweezers should be used when handling samples in the SEM sample chamber. Care should be taken to avoid burns when handling and using liquid nitrogen (LN_2).

Before using liquid nitrogen, be sure to put on protective gloves and eye guard glasses. If liquid nitrogen splashes on your skin, you may suffer frostbite. In addition, the room should be well ventilated to prevent oxygen depletion.

Standard equipment and materials

The laboratory provides the following: LN_2, SEM sample holders, SEM conductive paste, and SEM double-sided conductive adhesive tape. Other materials must be cleared with the lab manager.

User maintenance

Users are requested to use the proper SEM sample holder with their sample.

Waste disposal

Dispose off the alcohol soaked wipes in a waste container marked for flammable solid waste.

Scheduling

Reservation can be done online also the system can be used on a first-come, first served usage if no reservation was made.

Other issues

Users should remain physically present in the clean room facility during the entire use of the SEM tool.

At no time should a user adjust a pressure regulator on a gas line. Gas control should be “on” or “off” only, using only the appropriate valves. For most gases, this is usually the valve at the cylinder head.

Non-standard use

Users may not modify any hardware on the SEM tool. For use of non-standard processes, or materials, contact the staff or the lab manager.

Preliminary Operation:

Check the Column Vacuum, (At the beginning of the SEM operation, check the evacuation control panel, the following conditions must be met):

1. IPI, IP2 and IP3 lamps are lit.
2. Ion pump readings are better than the following:
   - IP1: – 2X10-7 Pa
   - IP2: – 2X10-6 Pa
   - IP3: – 7X10-5 Pa
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 and S.E.C VACUUM are lit.
6. GUN VALVE switch is at CLOSE and AUTO lamp is blinking.
7. OBJ. APT., switch is set at Heat.

Note: Keep the OBJ. APT., switch on the evacuation control panel at HEAT. If the objective lens aperture is contaminated, charging will degrade image quality and the image will drift because of micro discharge. Such problems are noticeable at low accelerating voltages. The aperture is heated to about 150° C to remove contaminants to one tenth or less of what it would be at room temperature. The switch should be turned off only when introducing air into the specimen chamber.

.

LOGGING ON to the SEM System:

1. Ensure that the EO CONTROL switch is set to on (1) and then turn the DISPLAY switch on to (1). During routine operation, EO CONTROL need not be turned off to (0).
2. Log on by Pressing Ctrl, alt and Del keys simultaneously. Input your login name and password.
3. Double-click on the PC-SEM icon on the desktop. The S-4700 system will start and the initial login dialog window appears.

Use of Anti-Contamination Trap and Cold Trap

For image observation at high magnifications or low accelerating voltages, the use of the anti-contamination trap and cold trap is recommended to prevent specimen contamination by hydrocarbon build-up. Both traps need to be filled with liquid nitrogen.

1. The anti-contamination trap is a plate above the specimen that adsorbs gas around the specimen. The capacity of the liquid nitrogen dewar is about 0.9 liters and is usable for about 5 hours at an ambient temperature of 24°C. For initial filling, about 1.3 liters of liquid nitrogen is required.
2. The cold trap is located above the diffusion pump and is used to improve the vacuum of the specimen chamber. The capacity of the liquid nitrogen dewar is about 3.4 liters and is usable for about 8 hours at an ambient temperature of 24°C. For initial filling, about 5 liters of liquid nitrogen is required.

CAUTION!

Before using liquid nitrogen, be sure to put on protective gloves and eye guard glasses. If liquid nitrogen splashes on your skin, you may suffer frostbite. In addition, the room should be ventilated to prevent oxygen depletion.

Never introduce air into the specimen chamber while the anti-contamination trap is filled with liquid nitrogen. The anti-contamination trap will frost up and the vacuum will deteriorate. Before introducing air into the specimen chamber, wait for a few hours after the liquid nitrogen dewar has completely emptied. The air introduction valve does not have a protection link with the cold trap.

Specimen Setting: (*Caution on 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.
2. Avoid using an excessive amount of conductive paste to fix a specimen on the specimen stub. Ensure that the paste has dried before placing the specimen in the chamber.
3. Select the correct specimen stub for each specimen.
4. When using double-sided adhesive tape to fix a specimen to the stub, use the least amount to minimize out-gassing. The use of double-sided adhesive tape may also cause specimen drift.

Specimen Preparation according to Materials: (*The method of specimen preparation varies with materials.*)

1. **Conductive Specimens:**

   Such as Metals (These types of specimens can be observed without preparation. However, coating with heavy metals by using a vacuum evaporator, an ion sputtering or magnetron sputtering unit may result in contrast.

2. **Non-conductive Specimens:**

   Such as semiconductors, fibrous specimens and polymeric materials are examples of non-conductive specimens.

   Coating with conductive materials is recommended. To observe these kinds of specimens without a conductive coating, use low accelerating voltages (1 kV or lower). However, coated particles may be more visible at higher magnifications.

3. **Biological Specimens:**

   After dehydration, dry the specimen by using a method as critical point drying, freeze drying or other drying techniques, than coat the specimen with conductive material. A cryogenic system is available as an option.

4. **X-ray Analysis Specimens:**

   Generally, polish the surface of the specimen, than fix it to the specimen stub using conductive paste. Non-conductive specimens should be coated with conductive material using a vacuum metal deposition system.

Adjustment of Specimen Height:

Put the specimen stub on the specimen holder and adjust it to the proper height using the specimen height gauge. 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 the lock screw.

CAUTION!

The specimen height must be adjusted carefully. It must not be 0.5 mm higher than the bottom of the 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 the specimen height minimizes the image shift during tilting of the specimen.

Specimen Exchange Position: (*5-axis motorized stage*)

Click the **Go to Home** button in the **Stage Control** dialog window. The stage is then moved to the specimen exchange position, and the color of the indicator button turns green. To open the **Stage Control** dialog window, click the Stage operation area of the **Scanning Image** window, or click the icon on the toolbar. Selecting the **Stage Control** command from the **Operate** menu can also access the **Stage Control** dialog window. Specimen exchange position:

X:	25.0 mm	**Y**:	25.0 mm		
R:	0 Deg	**T:**	0 Deg	**Z**:	12.0 mm

NOTICE: Do not repeat clicking the **Go to Home** button, otherwise the **Stop** button may become ineffective.

CAUTION!

Start specimen exchange operation after the color of the indicator changes to green.

How to Set Specimen: