

Argonne National Laboratory Advanced Photon Source GeoSoilEnviroCARS

Standard Operating Procedures for JEOL JCM-6000Plus scanning electron microscope in Building 434A, Room A030 January 24, 2019

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GSECARS

SOP for JEOL JCM-600Plus SEM in Laboratory A030, Bldg 434A December 5, 2018

1. Introduction

This is a Standard Operation Procedure to operate the scanning electron microscope listed in Table 1 in lab A030. This SEM is designated as a Radiation Generating Device (RGD) as specified in LMS-PROC-109. It is registered with the Argonne RGD Safety Officer on form ANL-847 form with inventory number 275. Please follow this SOP strictly to ensure both personnel and instrument safety and to keep the instrument in good working condition.

Name of RGD custodian: Michael Fries
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Table 1: SEM Specifications

	SEM
Brand	JEOL NeoScope
Model	JCM-6000Plus
Serial number(s)	MP1230004250425
ANL Inventory number	275
Quantity	1
Accelerating Voltage	15 kV, 10 kV, 5 kV
Power	Single phase AC 100V
RGD Class	1

2. Hazards

The SEM is completely self-contained within a benchtop enclosure engineered by the manufacturer. The JCM-6000Plus is manufactured to maintain a high level of electrical and radiological safety. When the SEM is in operation the main potential hazard is from X-ray leakage, but this is certified by the manufacturer to be well below the stipulated value of Radiation Standards legislated by the Japanese Government (JIS) Japan Analytical Manufacturers Association (JAIMA) and European Union (EU) EN61010 ($< 2 \mu \text{ Sv/hr}$). These safety values are maintained as long as the instrument is not modified and only the

associated high voltage power supply is used. As such, no modifications and/or improvements on the electro optics column and associated high voltage power supply are allowed. Only certified JEOL Field Engineers (certified under ISO-9001) are allowed to service these components, as they are not designated as components requiring routine maintenance by operators.

3. RGD normal operation instructions

Instrument Startup and Sample Exchange:

1. Ensure you have obtained training for use of the instrument. Only trained and authorized SEM users are allowed to operate the SEM. Log all usage on the provided log sheet.
2. Set the MAIN BREAKER of Power Supply Box to ON if not on already.
3. Press the START switch on the NeoScope SEM front panel. When started, the Green LED on the front panel will light up.
4. Start the control PC and log in to the JCM-6000 control system.
5. Log in with your name and badge number and certify that you have read the SOP and will abide by the stated protocols.
6. Prepare your specimen using suitable protocols. Please validate that samples are suitable with designated Operators. Affix your sample to the Specimen Block. Loosely fixed powder samples and highly magnetic materials (particularly powders) are NOT ALLOWED in the instrument.
7. Insert the Specimen Block in the Specimen Holder. Adjust the position of the specimen to the surface of the specimen holder. Turn the "Height adjust screw" using a flat-blade screwdriver from the bottom of the specimen holder to match the observation surface with the holder height, and fix with a locking screw.
8. Press the VENT button on the SEM. The Blue LED on the SEM should turn off.
9. Pull out the stage after the front door opens.
10. Turn the locking knob counterclockwise to remove the specimen holder that is in the SEM.
11. Place the specimen holder with your sample onto the stage and turn the locking knob clockwise to secure the specimen holder.
12. Close the front door firmly. Keep pressing on the door until the VENT button light stops flashing.

Sample Imaging:

1. In the operating software, set the appropriate Vacuum Mode, Signal, AccV, Filament Current, and Probe Current, if you necessary.
2. Click the AUTO buttons in the following order for initial image configuration: Brightness, Focus, and Stigma
3. Select scan speed (Fast or Slow)
4. Adjust the field of view using the MOVING button on the GUI.
5. Click on the image with the mouse button to change magnification.
6. Click the PHOTO button to take an image.

Instrument Shutdown:

1. Confirm that the SEM is at vacuum before shutting down, the column should be

- kept under vacuum conditions in shutdown mode (The Vac. Indicator will show 3 orange LED lights).
2. Click the POWER OFF button on the GUI.
 3. If you've done EDS analysis, make sure the power toggles for the EDS Power Supply and Digital Pulse Processor are turned to OFF.
 4. Close the GUI.
 5. Log out of the Computer (DO NOT leave the PC logged in if not in use) and shutdown the personal computer if no other users are scheduled.
 6. Set the MAIN BREAKER on the Power Supply box to OFF if the system will not be used for extended period (ask staff).

4. Emergency response, including off-normal alarms, abnormal external radiation level, and proper notification

It is unlikely that this SEM will enter an abnormal operational state. However, you may encounter normal two situations that require attention:

1. The instrument detects an Overcurrent state between the SEM and Power Supply. In this situation the MAIN BREAKER on the power supply will trip. Do not attempt to reset the breaker. Contact designated RGD Operator who will contact JEOL Service Office for inspection.
2. If abnormal noise or abnormal odor is issued from the instrument, perform an emergency stop of the instrument as follows:
 - a. Set the MAIN BREAKER of the power supply box to OFF (○). DO NOT turn ON the power supply if the main breaker shut off automatically before you had an opportunity to perform the emergency stop procedure. If the breaker is shut off, contact your local JEOL service office because it is considered that an abnormality has occurred in the instrument main unit.
 - b. Turn off the PC and the peripheral equipment of the PC as necessary.
3. You may also encounter routine maintenance messages from the system during operation, for example requiring replacement of the tungsten filament. If this occurs, shutdown the instrument as described above and inform one of the designated RGD Operators. Users are NOT allowed to undertake filament replacement on their own under any circumstances.

5. Methods and occasions for locking and securing the RGD

Shutdown procedures are described above and the SEM should always be placed in a shutdown mode when not in immediate use.

6. Calibration and alignment of the useful beam, as needed

Routine sample calibrations are performed for each specimen using the available alignment tools in the GUI. Electron Column alignment is ONLY ALLOWED to be performed by a certified JEOL Field Engineer. Periodically, the electron filament will require routine replacement. **ONLY designated GSECARS RGD Operators are allowed to perform this task.** If the SEM filament needs replacement, please contact a designated operator, do not attempt to replace the filament yourself (which will result in loss of SEM privileges).

Filament Replacement:

1. The GUI should show a message stating that the filament should be replaced. Clear the message and select MAINTENANCE > EXCHANGE FILAMENT from the Menubar.
2. Follow the onscreen instructions for leaking the gun, column and specimen chamber to atmosphere.
3. Wait for a message from the GUI stating that the electron gun chamber, the column, and the specimen chamber have reached atmospheric pressures.
4. CAUTION: If the system has been in use, the Wehnelt cup housing the tungsten filament is likely to be at high temperature. Allow 30 minutes following venting to allow the temperature to reach ambient.
5. Use gloves to keep replacement filaments clean.
6. Slide the top cover in the right side until it stops to unlock and then open the top cover on its hinge.
7. Confirm that the specimen chamber is at atmospheric pressure and the specimen stage door is open.
8. Turn two electron gun fixation screws on the electron gun counterclockwise and remove the electron gun from its housing.
9. Vertically lift the electron gun, and put it on the filament exchange hall installed in the inner cover.
10. Loosen the Wehnelt ring with the special tool provided.
11. Turn the Wehnelt ring a little counterclockwise by using special tool. Remove the special tool when the Wehnelt ring loosens.
12. Turn the Wehnelt ring counterclockwise and detach.
13. Install the new filament. Two filament feet behind Wehnelt are set to the position of the electron gun side filament terminal, and insert it so that there is no space.
14. Next, turn the Wehnelt ring clockwise, and fix the Wehnelt firmly. DO NOT use the special tool in insert the new Wehnelt. Tighten ONLY to finger tightness.
15. Return the electron gun to the SEM mount.
16. Put the guide hole installed on the electron gun side along two guide sticks on the column side, so that the electron gun is in its former position.
17. Turn the electron gun fixation screws on the electron gun clockwise and fix the electron gun. DO NOT overtighten!
18. Return the top cover to former position

7. Control of computer passwords for RGD operation

The GSECARS designated RGD Operators have control of computer passwords.

8. Interlock test procedure

No X-ray tube is incorporated in the JEOL JCM-6000Plus. The interlocks for the device are incorporated within the electron optics column and the specimen chamber door. The interlocks ensure that the accelerating voltage of the electron gun cannot be energized when the electron optics column is at atmospheric pressure, which occurs when the specimen chamber is opened. Therefore, the emission current cannot be switched on, and therefore no X-rays can be generated from the electron gun, with the specimen chamber opened and no vacuum detected in the chamber.

9. Inspections and testing (forms to follow)

Vacuum Pump: Check the oil level of the rotary pump routinely and make sure it does not decrease lower than the lower limit. Driving the rotary pump in a state of insufficient oil might cause a malfunction of the pump. Check the amount and stain (coloration) of oil using the oil level indicator of the rotary pump (once every three months). If the instrument use frequency is high, shorten the inspection cycle. GSECARS engineers will replenish or exchange the oil as needed.

10. SEM training (forms to follow)

Two lists of SEM users will be maintained:

- 1) GSECARS staff that are familiar with RGD JHQ's, have read and familiarized themselves with this SOP and that have been fully trained to both operate the SEM and perform routine maintenance tasks. This list is kept with the SEM in a binder.
- 2) General Users that have received training on routine operation of the SEM and have read and familiarized themselves with this SOP . This list is maintained electronically in an EXCEL spreadsheet (\\CARS5\Data\sem_user\SEM_Registration_Form.xlsm), with use logged every time the SEM control software is initialized.

