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

Prepared by:
RGD Lead Operator for GSECARS:

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APS RGD Custodian:

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Approved By:
APS AES Division ESH Coordinator

Date

Approved By:

Date
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
RGD Lead Operator for GSECARS: Antonio Lanzirotti

Table 1: SEM Specifications

<table>
<thead>
<tr>
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<th>SEM</th>
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<tbody>
<tr>
<td>Brand</td>
<td>JEOL NeoScope</td>
</tr>
<tr>
<td>Model</td>
<td>JCM-6000Plus</td>
</tr>
<tr>
<td>Serial number(s)</td>
<td>MP1230004250425</td>
</tr>
<tr>
<td>ANL Inventory number</td>
<td>275</td>
</tr>
<tr>
<td>Quantity</td>
<td>1</td>
</tr>
<tr>
<td>Accelerating Voltage</td>
<td>15 kV, 10 kV, 5 kV</td>
</tr>
<tr>
<td>Power</td>
<td>Single phase AC 100V</td>
</tr>
<tr>
<td>RGD Class</td>
<td>1</td>
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</tbody>
</table>

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 μSv/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.
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 control system.
5. Prepare your specimen using suitable protocols. Please validate that samples are suitable with designated Operators. Affix your sample to the Specimen Block.
6. 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.
7. Press the VENT button on the SEM. The Blue LED on the SEM should turn off.
8. Pull out the stage after the front door opens.
9. Turn the locking knob counterclockwise to remove the specimen holder that is in the SEM.
10. Place the specimen holder with your sample onto the stage and turn the locking knob clockwise to secure the specimen holder.
11. 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.

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 and posted on the hallway door

1) Users with RGD JHQ’s that have been fully trained to both operate the SEM and perform routine maintenance tasks.
2) Users without RGD JHQ’s that have received training on routine operation of the SEM
## Inspection Logs

Log of operational checks, inspections, and testing

<table>
<thead>
<tr>
<th>Date</th>
<th>Name</th>
<th>Inspection Type</th>
<th>Signature</th>
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<tbody>
<tr>
<td>01/23/2019</td>
<td>Lanzirotti/Bristol</td>
<td>Initial commissioning</td>
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GSECARS

Users with RGD JHQ’s that have been fully trained to both operate the SEM and perform routine maintenance tasks

Signature Sheet for 434-A030 SEM Operations

Persons signing this form certify that:
1. They are fully familiarized with the contents of LMSPROC109
2. They have received an SEM Safety training overview under supervision of the Lead RGD Operator for GSECARS.
3. They are fully familiarized with the contents of this GSECARS Standard Operating Procedure (SOP) for SEM operations in laboratory 434-A030SOP
4. They agree to fully abide by the contents of this SOP.

<table>
<thead>
<tr>
<th>ANL Badge No.</th>
<th>Print Name</th>
<th>Affiliation</th>
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GSECARS

Users without RGD JHQ’s that have received training on routine operation of the SEM

Signature Sheet for 434-A030 SEM Operations

Persons signing this form certify that:
1. They have completed On-the-Job SEM operations training under supervision of a designated GSECARS RGD operator listed above.
2. They are fully familiarized with the contents of this GSECARS Standard Operating Procedure (SOP) for SEM operations in laboratory 434-A030SOP
3. They agree to fully abide by the contents of this SOP.

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