

Standard Operating Procedure JOEL JSM-IT300

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|---------------------|----------------------------|-------------------------|---------------|-----------|------------|
| School: | Engineering | Campus: | Waurn Ponds | Location: | KE2.103.02 |
| Date Prepared | 29/04/2019 | Date to be Reviewed: | 29/04/2020 | | |
| Category Rating: | Specific training required | | | | |

SCOPE & PURPOSE:

The purpose of this SOP is to show proficient users and staff how to operate the JOEL SEM IT300 safely to obtain surface and compositional information of the tested materials.

TRAINING REQUIREMENTS:

User will have completed Level 1 online and Level 2 materials labs inductions, and need to be trained by technical staff in a level 3 induction. Before using this equipment, required safety documentations must be completed and signed off by the technical staff and the user's supervisor.

POTENTIAL HAZARDS/HEALTH EFFECTS:



Mechanical entanglement in the stage or internal parts inside the SEM chamber.

Crushing by being trapped between the plant and materials or fixed structures.

Cutting, stabbing and puncturing due to coming in contact with moving parts of the plant during testing, inspection and operation. Ergonomic due to other factors – long periods of sitting.

Electrical due to contacting live electrical conductions or working in close proximity electrical conductors.

MANDATORY CONTROLS:

✓ Safety glasses

Safety gloves



 Use supports to position workpieces

Project Safety
 Assessment

Precaution Requirements:

DO NOT USE AFTERHOURS (OUTSIDE OF MON – FRI 8AM – 6PM) WITHOUT TECHNICIAN AUTHORISATION.

- Be mindful there are no collision sensors/detectors installed on the machine.
- Sample must be prepared properly consult with technical staff.
- Nitrile free safety gloves must only be used.
- Sample must be stored in the designated dessicator at least a day before the booked date.
- The SEM is extremely sensitive to contamination. Gloves must be worn when touching your sample, sample holder, placing the sample holder into the instrument chamber, or cleaning the chamber seal. Gloves are to be removed after pumping the SEM chamber.
- Do not run the equipment if suspicious conditions (e.g. contaminations) are observed inside the chamber.
- Keep clear of the equipment during operation.
- **DO NOT** change the location or names of any files/projects/folders once created.
- The side camera must be live all the time before, during and after experiment.
- Do not attempt to open the chamber door before it is completely vented.
- The technical staff are only to perform the aperture alignment.
- **DO NOT** tilt the stage with the sample underneath the pole-piece (and the backscattered electron detector) unless there is sufficient clearance.

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INSTRUCTIONS:

Booking Procedure: must have a booking in the Outlook Calendar *G SEBE ENG Lab Eqpmt KE2.103.02 SEM (even if free and available).

Before Use:

- User must be TRAINED for the JEOL IT300 and SEM sample preparation by SEM technical staff(s).
- User must have organised technical assistance for the duration of the booking.
- Ensure the stage is in lowermost and neutral position (Z coordinate value of 75 mm). The tilt/rotation angle of the stage must be 0 degree.
- Ensure there are no items on the stage before venting the chamber.
- Samples must be prepared properly and mounted on stage for insertion into chamber. If using stubs, the sample must be appropriately attached to the stub and the stub should also be fully fixed on/within the stage.
- The sample should be safely introduced to a medium pressure blow of nitrogen or at least air to ensure all parts of the sample are secure and will not be removed once placed under vacuum.
- Samples must have been dried either in a desiccator or vacuum chamber/cabinet for at least 6 hours prior to analysis.
- Using a Vernier Calliper accurately measure the height of your sample.
- Check the aperture adjustment piece to check the spot size get the technician to change if required.

Operation:

Never operate the joystick of the SEM without being in the live chamber view!!!

- Open the IT300 software and select SOE students.
- Make sure the live chamber view on the right hand side of the screen is displayed before physically moving the stage.
- Check that/move the stage into the safe exchange position ie: X, Y, R and T=0, and Z=~75mm.
- Vent the chamber.
- Inspect all detectors and the rubber seal band of the door frame for any abnormalities. If any unusual object (including hair, particles or powders) or defected part or wire is observed inside the chamber, put the out of order sign on the keyboard and quickly report to the SEM technical staff(s).
- Open the chamber and double check that the stage is in a safe position in regards to your sample size before inserting the sample holder onto the stage.
- Ensure that the sample holder is secured onto the stage.
- Close the chamber door very gently not to vibrate the machine parts.
- EVACUATE the chamber and wait until the process bar is complete and the SEM is ready.
- Inform the JEOL IT300 software of the type of sample holder or stage being used and your sample height.
- Change the active display to the still image screen and press the camera icon to take a still image of the sample be aware the sample stage will tilt, do not press stop during this process.
- On the still image screen, double click on the areas of interest on your sample and record the X and Y coordinates.
- Change the active screen to the live chamber view.
- Ensure that the T/Z button is blue on the control panel.
- Push the joystick up to raise the stage in the Z direction (Z coordinate value will decrease from 75mm) to set the distance between the top of your sample and the back scatter detector to the approximate working distance required (≥12mm).
- Turn the observation on.
- Set the imaging parametrs and perfom the required beam and aperture alignments. Aperture alignment must be only done by the technical staff.
- Save your image at the desired magnification in your designated database folder.

WARNING: Everytime you change any of the beam parameters (eg: accelerating voltage, probe current) or working distance you need to check the aperture alignment and stigmation correction.

IF YOU ENCOUNTER ANY ISSUES DURING IMAGING – PLEASE NOTIFY TECHNICAL STAFF.



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After Use:

- Turn the observation off.
- In the live chamber view, using the joystick to return the stage to the safe exchange position ie: X, Y, R and T=0, and Z=~75mm.
- VENT the chamber.
- Remove the sample and sample holder from the chamber.
- Check/clean the rubber seal band of the door frame.
- Close the chamber door.
- EVACUATE the chamber.
- Remove the sample from the sample holder and return the holder to the desiccator.
- Store your samples in your sample container.
- Ensure all tools/equipment used are clean and return to the SEM tool box.
- Make sure the working area is clean and tidy.
- If you observe any dust, spill or mess in the SEM lab, please clean it and report it to the SEM technical staff if anything requires further attention or action.

GENERAL CARE & MAINTENANCE:

- Internal components inside the SEM chamber must be inspected regularly.
- Sample preparation tools and mounts (sub-stages and specimen holders) must be de-greased and cleaned regularly.
- Check the tool box and the dessicator are maintained clean and silica gel well active (light orange in color).
- Make sure you collect all your samples and belonginings and the working area is clean and tidy before you leave.

REPAIRS & CERTIFICATION/VALIDATION SCHEDULE:

Reporting faults/concerns of equipment procedure: Technical staff.

Calibration is conducted by technical staff whenever the filament is changed or as required.

Supplier calibration is conducted annually.

SPILLS/ ACCIDENT REPORTING PROCEDURE:

Report incidents to: Technical staff.

In case of Emergency:

- Contact Technical Staff
- If a serious injury has occurred call 000.
- Alert others if nearby.
- Evacuate to closest assembly area.