NEMO Name: JEOL 7500F HRSEM

Location: QNF Bay 6

A high resolution FE-SEM for simple inspection of conductive samples showing topographic detail and/or material contrast. ***Samples must be vacuum compatible, clean, and conductive****. Thin or small (<100nm) nonconductive features are often compatible.*

Includes:

- *Two Everhart-Thornley secondary electron detectors*
- *Transmission electron detector*
- *Low magnification viewing mode*
- *Backscattered electron detector*
- *EDAX detector for elemental x-ray analysis (EDS.)*

Contents

User Responsibilities and Safety

- **Always wear gloves when handling anything that goes inside the SEM** (including but not limited to: samples, stubs, and sample holders)
- Ask for help if you are uncertain.
- Be aware of the exchange rod and the gate valve when loading or unloading samples.
- **Do not overtighten set screws** (or remove them, unless necessary.)
- Always **check the vacuum status diagram** before opening the exchange chamber.
- Return the **stage to exchange position** before inserting or removing a sample.
- **Do not load samples taller than the top of the sample holder.**
- **Copy your data** in a timely manner from the EDAX computer **with a USB drive**. Data will be periodically deleted when the computer needs space.
- Reserve the tool in NEMO, then enable when you start and disable when you finish.
- **Report problems through NEMO.**
- Clean up after yourself.

Training and Qualifications

- Complete **at least two (2) training sessions** with staff and demonstrate safe and competent operation of the tool to receive independent access (**Prime time only: 9-5 weekdays.**)
- Staff may require (or users may request) additional training before access is granted.
- Additional training for advanced techniques available upon request.
- **24/7 access** may be granted at staff's discretion after completing **at least 4 hours of independent use** on the tool without incident.
- Staff may require **refresher trainings after a period of disuse (e.g. 6 months)** or if a user has demonstrated a lack of competency or understanding on the tool.

Operating Procedures

Prepare Sample

1. Secure the sample to a stub.
    a. Do not use carbon tape in the cleanroom.
2. Secure the stub in a sample holder and adjust so the **surface of the sample is in line with the top of the holder.**

System Startup

1. **Log in to NEMO**.
2. **Load** the sample into the exchange chamber and close the door.
3. **Evacuate** (**EVAC**) the exchange chamber.
4. Open the chamberscope (**IR Camera.**)
5. Ensure the stage is at **Exchange Position.**
6. **Push** the sample onto the stage.
    a. **Do not force the transfer rod**.
    b. STOP if it beeps.
    c. Insert completely and **select holder.**
7. Return the transfer rod to its resting position.
8. **Vent** the exchange chamber.
9. Set initial **voltage**, **current**, and **probe current**.
10. Wait for the vacuum to read **below 2E-4Pa.**
11. **Turn on the beam**.

Alignment and Imaging

1. **Zoom out** in low mag (**LM**) mode.
2. **Navigate to a feature** on the sample.
3. Zoom, focus, and adjust contrast and brightness **until mag > 800X.**
4. Switch to **SEM mode**.
5. Gradually **zoom**, **focus**, and **adjust contrast and brightness** until a magnification just higher than the highest desired imaging mag.
    a. *Adjust initial conditions as needed*.
6. Press **Wobb** and align the aperture with X and Y.
    a. Press Wobb again when finished.
7. Align **stigmators** with X and Y until the image does not stretch when out of focus.
8. **Focus** (you have more control at higher mags.)
9. **Save images** and make annotations.

Shutdown

1. Return stage to **exchange position**.
2. **Turn off the beam**.
3. **Evacuate** exchange chamber.
4. **Remove** sample from stage with exchange rod.
5. **Vent** exchange chamber.
6. Remove sample from the exchange chamber.
7. **Clean up after yourself**.
8. Log out of NEMO.

Extra space for your notes: initial conditions, recommendations, tips, tricks, etc.

Appendices

Basic Troubleshooting

- **Voltage (kV)**
  - High voltage better for lower mag, conductive samples
  - Low voltage (1-5kv) better for surface detail, cross section, less conductive samples
  - **0.1-30kV**
- **Probe current (arbitrary number)**
  - High better for low noise, larger features, EDS.
  - Low better for high magnification, edges (reduces interaction volume, edge effect)
- **Working distance (mm)**
  - **8mm** for most, increase for greater depth of field
  - 5-6mm if struggling with contrast, signal, especially for low voltage, low probe current.
- **Focus depth-resolution (slider)**
  - Focus depth for highly textured surfaces (large height differences)
  - Resolution for flatter samples, surface detail.

Imaging Modes and Detectors

LM

- Useful for **finding features of interest** or **navigating across large distances**
- **Overlaps with SEM mode after about 500-1300X** magnification, depending on the working distance.
- Alignment less useful – perform alignment using the desired imaging mode.
- It is normal to see the round aperture at the lowest magnification.
- About **25-10,000X** zoom.

SEM

- **High magnification** imaging mode, up to 1,000,000X.
- **LEI** detector positioned lower in the chamber than **SEI**. Try both to see which is best for your sample.
- Can image with secondary and/or backscattered electrons (change the slider beneath the r filter)

GB

- Secondary electron imaging mode using the same detectors as SEM.
- **Applies a bias** to the sample and sets the accelerating voltage so the difference is the chosen value.
  - E.g. 4kV accelerating voltage – 2kV sample bias = 2kV voltage.
- Useful for **less- to non-conductive samples.**
- Usable voltage range between **0.1kV and 3.9kV.**

TED

- Transmission electron detector
- Use with TEM grids in the TEM grid holder.
- **Contrast is reversed** (i.e. features of interest are dark, holes are bright.)

LABE

- Backscattered electron detector.
- Improved material contrast.
- **Stage must not be tilted.**
- Slow scan speed for best results.

User Preferences

- Go to: **Setup > Operation Settings.**
- Select your preferred **scan speed presets**.
- Select your preferred **photo presets**.
- Select the **destination folder** for your images.

EDS

- **Energy Dispersive X-Ray Analysis**: Elemental analysis of samples based on **characteristic X-ray** emissions.
- Check characteristic x-ray energies for each element in a sample
    - Set **voltage to 2-3x** the energy for **at least one peak in each element**.
    - Higher voltage typically yields higher counts.
- Use **high probe current** (e.g. 13)
- Ensure sample is **focused near 8mm**
- Aim for **high counts per second** (cps) with around **30% dead time** (DT)

More Troubleshooting

Common Problem	Cause	Solution
Image suddenly loses contrast	mechanical (glitch.)	Turn the beam off and on again
Exchange rod beeps when lowered	Either gate valve is open or stage is not at exchange position	Return the rod to the resting (upright) position. Click Evac if it is not already green. Click Exchange Position if stage is not at (0, 0, 0, 8, 0)
Focus skips ideal focus	A normal effect: lens configuration changes at 6.0, 7.5, 9.0 mm WD's (and more.)	Select Z and change stage height by 0.2mm
Emission current drops <2uA	Source may need to be flashed. It is normal for the emission current to read 0.5-1uA below.	Click Reset beside Emission Current and wait. Contact staff if this does not resolve the issue after 1-2 tries (with waiting.)
System displays Flash alert and beam shuts off	This is automatic: let it happen	Wait. Turn the beam back on and wait. It will take longer to warm up to full current after a flash.
Image not in focus switching from LM to SEM	This is normal.	Zoom to approx. 800X or more in LM and center the image on an area with good contrast/features. Switch to SEM, go to a low mag (approx. 800X) and refocus.
Image in LM, but no image in SEM	These modes "remember" settings from the last use	May be: contrast/brightness; focus; magnification; detector. Go through operation steps.
No data bar on bottom of saved image Annotations not saving on images	Export (save dialog) was unchecked	For future images: ensure Export is checked before saving. For existing images: go to File > Open and open images. (Add annotations if needed.) Then to go File > Save Image and ensure export is selected.
Stage movement error (cannot reach target location)	Either: stage limit hit Or: moved mouse/stage too quickly	Try moving again. If the error persists, a stage limit has been reached. Stage limits are approx ±30 mm in X and Y