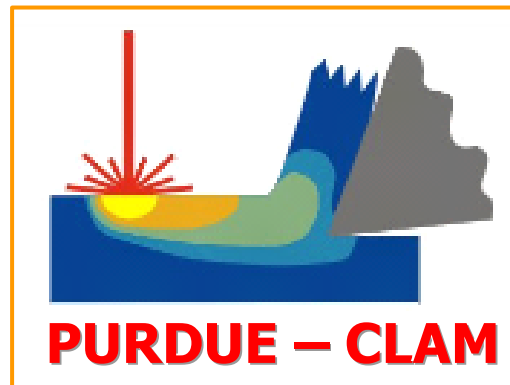


Sample Preparation Procedures and SEM Operating Procedures

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<http://engineering.purdue.edu/CLM>



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Outline

- Sample Preparation
 - Sectioning (LECO MSX255M)
 - Mounting (LECO PR4X)
 - Polishing (LECO SS-1000)
 - Etching (Birck labs)
- Scanning Electron Microscopy (JEOL JSM-T330)
 - Operating Procedures
 - Maintenance and Supplies



Sample Preparation

Sectioning (LECO MSX255M)

- 2865 rpm, fixed table, manual feed, catch basin to collect parts
- Safety glasses required
- Main power switch also activates internal light
- Select appropriate wheel using chart on cabinet

Large wrench holds wheel steady,
small wrench loosens nut

Cardboard spacer needed for thin
wheels

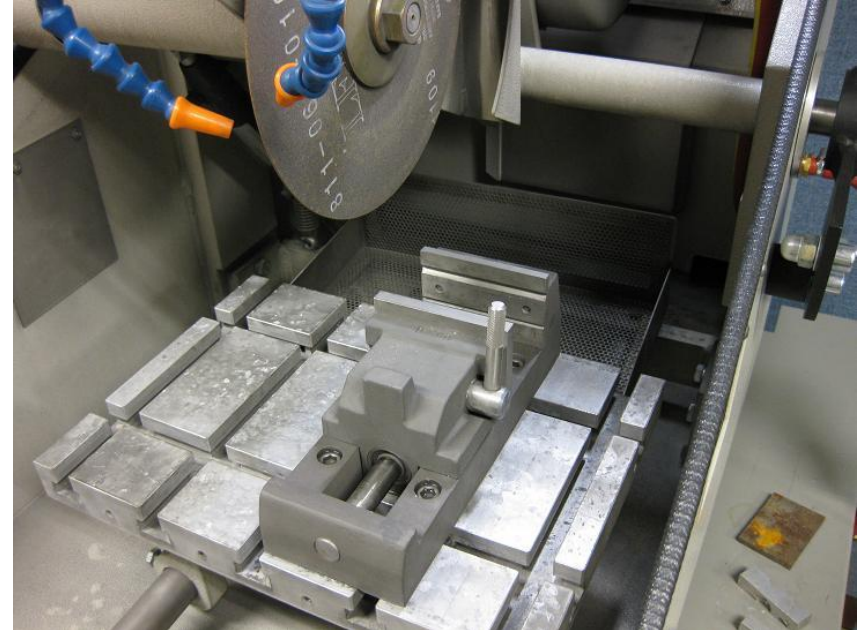


Sample Preparation

Sectioning (LECO MSX255M)

- Secure sample in cam-lock vise
- Check clearances and adjust coolant nozzles
- Close hood and push green button on control panel to start coolant flow and wheel motor
- Make sure coolant flow is sufficient before beginning cut
- Slow, steady force is best

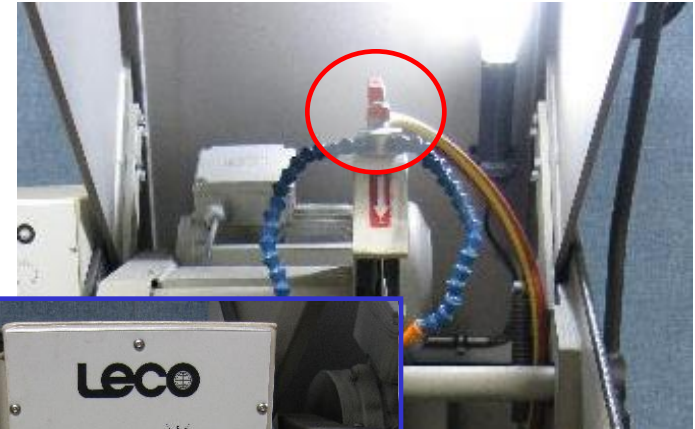
Reduce force if sound and/or vibration
become very strong



Sample Preparation

Sectioning (LECO MSX255M)

- Wash-down interior after each session
 1. Close valve that supplies coolant nozzles
 2. Partially open valve on wash-down hose and aim it inside
 3. Press blue coolant button on main panel to start flow
 4. When finished, close valve on wash-down hose and open valve above coolant nozzles
- Check coolant level after each session: water level 2-3" from top of reservoir



Sample Preparation

Sectioning (LECO MSX255M)

- Add tap water by pouring into top chamber or directly into reservoir
- To service reservoir:
 - Disconnect coolant hose and electrical control cable
 - Remove drain hose then roll reservoir forward and out of the cabinet



Sample Preparation

Sectioning (LECO MSX255M)

- Once per month:
 - Replace filter bag in back part of reservoir
 - Bag is secured by two nested metal frames
- Once every six months:
 - Drain reservoir via port on rear of reservoir
 - Scrub reservoir then refill with tap water and half a bottle of rust inhibitor (kept in cabinet under cutoff machine)
- User manual, bearing grease, and alternate arbor kept in cabinet under cutoff machine



Sample Preparation Mounting (LECO PR4X)

- Mold diameter is 1.25"
- Remove all burrs on surface of interest and dry sample completely
- Main power switch is on back of mounting press
- Control panel (one-touch keys):
 - Switch between parameters with > key
 - +/- buttons choose parameter values
 - Up/down arrows control ram position
 - Green/red buttons start/cancel programs



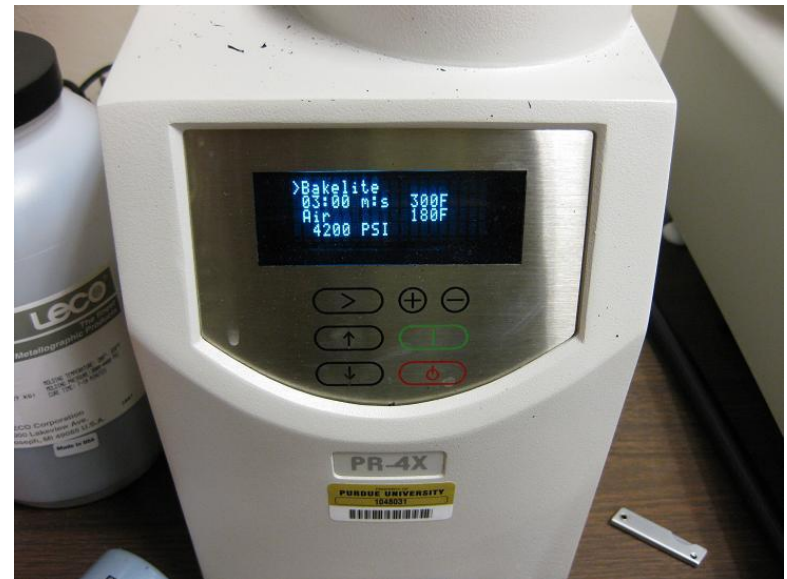
Sample Preparation Mounting (LECO PR4X)

1. Move ram to “up” position as shown at right
2. Place sample on ram with “good” side down
3. Press “down” arrow to lower ram
4. Scoop Bakelite into mold
Depth of Bakelite should be 1.5-2.0 times sample height; too much better than too little
5. Close cover



Sample Preparation Mounting (LECO PR4X)

6. Select program
 - “Bakelite” is default; works well for very first sample
 - “Method 8” goes for 1:30 longer to prevent under-curing in subsequent parts
7. Press “up” arrow to raise ram
8. Press green button to start program
9. Beeping signals end of program



Sample Preparation Mounting (LECO PR4X)

10. Press “down” arrow to lower ram
11. Open cover
12. Ram will raise automatically
13. Be careful – sample is 180°F!
Rinse with water then engrave
14. Clean Bakelite flash from top of mold – be careful of heat
15. Ram automatically rises when cover is closed for subsequent samples
16. Turn off power when cooling fan has stopped



Sample Preparation

Polishing (LECO SS-1000)

- Dual variable-speed wheels
0 – 500 rpm
- Adjustable water flushing
- Timed polishing cycles
0:30 – infinity
- Magnetic attachment system
Adhesive-backed paper attached to
Teflon-coated steel discs
Dedicated discs for 4 coarse grit sizes
and 2 polishing grades
CAMEO system has 2 coarse grit sizes
and 1 polishing grade



Sample Preparation

Polishing (LECO SS-1000)

- Chamfer edges of Bakelite-mounted samples to prevent catching on abrasive paper and for comfort
- Coarse polishing – grits 320-800, CAMEO blue and green – use left-side wheel ONLY
 1. Place disc of desired grit size on magnetic pad
 2. Adjust water flow to desired level
 3. Select time with left knob and speed with right knob: infinite time at 200 rpm is good for beginners
 4. Hold sample flat on abrasive paper and maintain orientation of sample such that direction of scratches is consistent
 5. Polish until all visible scratches are aligned and scratches from previous steps are gone



Sample Preparation

Polishing (LECO SS-1000)

- Coarse polishing (continued)
 6. Rinse sample in water stream to prevent contamination of next paper
 7. Repeat steps 1 through 6 for remaining coarse grit papers. Rotate the sample 90° every time you move to the next grit size so it is clear when the old scratches have been removed.

- Common problems and solutions

Developing multiple facets – Reduce downward pressure on the sample and focus more on keeping the sample flat and your pressure uniform. You may need to return to a coarser grit to eliminate extra facets. Pick the one you want to keep and apply focused pressure to slowly eliminate the others

Some old scratches will not go away – Try polishing longer. If the scratches remain, either replace the current abrasive paper with a fresh one or go back one grit size.



Sample Preparation

Polishing (LECO SS-1000)

- Before polishing, rinse the sample once more and redo the chamfer if necessary
- Final polishing – 6- μ m diamond paste – use right-side wheel ONLY – NO WATER
 1. Appropriate cloth is nylon (labeled bag in drawer)
 2. It is not necessary to add diamond paste to the wheel each time. To add paste, squeeze three or four radial lines from the syringe (pictured) onto the polishing cloth.



Sample Preparation

Polishing (LECO SS-1000)

- Final polishing (continued)
 3. Using a gloved finger, spread the paste over the cloth as uniformly as you can
 4. Squirt a small amount of lapping oil (red liquid in wash bottle) onto the cloth
 5. Choose time and wheel speed
 6. Very little pressure is necessary. Keep the sample flat on the cloth and move it clockwise, in the opposite direction as the wheel. Add lapping oil to improve lubrication if necessary.
 7. Polishing is finished when only fine scratches oriented in all different directions are visible. Groups of aligned scratches indicate incomplete polishing. Confirm with optical microscope.
 8. If unusually large scratches are introduced during this stage, the cloth might be contaminated by large particles. Replace the cloth.



Sample Preparation

Polishing (LECO SS-1000)

- Final polishing (continued)
 9. When finished, rinse the sample very thoroughly with water and dry with hair dryer
- Final polishing – 0.05- μm colloidal silica – use right-side wheel ONLY – NO WATER
 1. Use Imperial polishing cloth (labeled bag in drawer)
 2. Polishing medium contained in blue liquid in wash bottle. Apply a small amount ($\sim 1\text{-}2''$ dia.) to cloth to start.
 3. Use light pressure and move the sample clockwise. Use wash bottle of water for lubrication when necessary.
 4. Sample will appear mirror-like when polishing is complete. Any scratches visible on optical microscope are from previous steps.
 5. Rinse sample thoroughly and dry. Remove silica residue with water and focused, gentle scrubbing



Sample Preparation

Polishing (LECO SS-1000)

- When done turn off water flow and set time and speed to zero. Remove polishing discs and return them to holder. Replace covers on both wheels.
- Replace abrasive paper and polishing cloths as needed
- All consumables ordered from LECO catalog (in drawer)
- Sample prep guide (in drawer) contains polishing and etching suggestions for various materials
- Etching must be done at Birck after completing these courses

(<http://www.purdue.edu/discoverypark/nanotechnology/collaborate/SummaryofBNCTrainingCourses.pdf>)

1. BNC 105: BNC Facility Orientation
2. BNC 150: Fume Hood Training
3. BNC 208: Biosafety Training (BSL-1 only)



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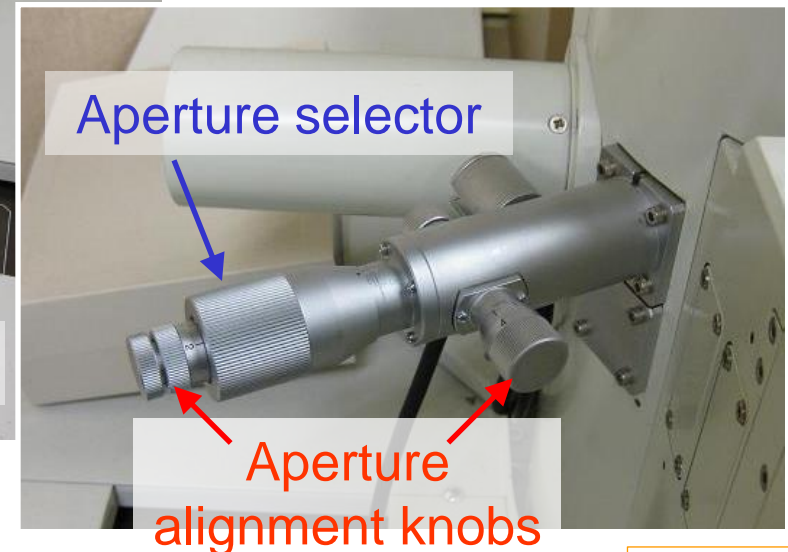
Scanning Electron Microscopy

Preliminary Considerations

- JEOL JSM-T330
 - Accelerating voltage 1-25 kV
 - K-type filaments
 - Maximum magnification is 200,000x
 - Digital image grabber replaces Polaroid film camera
- Wear gloves when cleaning, mounting, and transferring samples in/out of the SEM
- SEM guide can help you select parameters



Scanning Electron Microscopy System Layout



Scanning Electron Microscopy System Layout



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Scanning Electron Microscopy

General Considerations

- “A Guide to SEM Observation” describes the effects of each operating parameter on images
- Accelerating voltage 20-25 kV works best for metals
- To improve resolution:
 - Reduce working distance
 - Use a small aperture
 - Reduce spot size
- To improve depth of field:
 - Increase working distance
 - Use a small aperture



Scanning Electron Microscopy

Sample Preparation Procedures

- Sample cleaning (to be done while wearing gloves)

Unmounted samples:

1. 10-min ultrasonic cleaning in acetone
2. Dry with hair dryer
3. 10-min ultrasonic cleaning in methanol
4. Dry with hair dryer

Samples mounted in Bakelite:

1. 10-min ultrasonic cleaning in Branson cleaner solution
2. Dry with hair dryer

- After cleaning, nonconductive samples must be sputter-coated with apparatus in SEM lab in basement of Armstrong

Sample must be handled carefully with clean gloves afterward



Scanning Electron Microscopy

Startup Procedures

1. Turn on chiller and let it run for 15 min. before applying voltage. While you are waiting:

Sign in on the log sheet.

Select a specimen holder from the drawer and mount your clean sample.

Copper tape can be used to make a conductive path between the sample and the holder.

2. Turn key to 'Start' position then quickly to 'On' position
3. Press 'Vent' and wait several seconds before attempting to open the chamber
4. Load the specimen holder onto the stage. It only fits one way. Adjust working distance and visually confirm clearance between the specimen and objective aperture.



Scanning Electron Microscopy

Startup Procedures

5. Close chamber door. Once the 'DP' light is on, press 'Pump Down' and apply manual pressure to the door until 'EVAC' light illuminates.

If the EVAC state is not reached after 10 minutes, vent chamber and run a GLOVED finger over the gasket that seals the chamber door and its mating surface on the column. Close door and retry pump-down.

6. When 'HT READY' state is reached, select operating voltage. This CANNOT be adjusted once voltage is applied to the system.
7. Select an aperture: #1 is largest and #3 is smallest.
8. Press red 'HT' button to apply voltage.



Scanning Electron Microscopy

Filament Saturation

1. Initial settings:

Scan mode: LSP

Scan rate: EXP

Contrast: maximum

Brightness: adjust so the signal is in middle of screen

Magnification: minimum

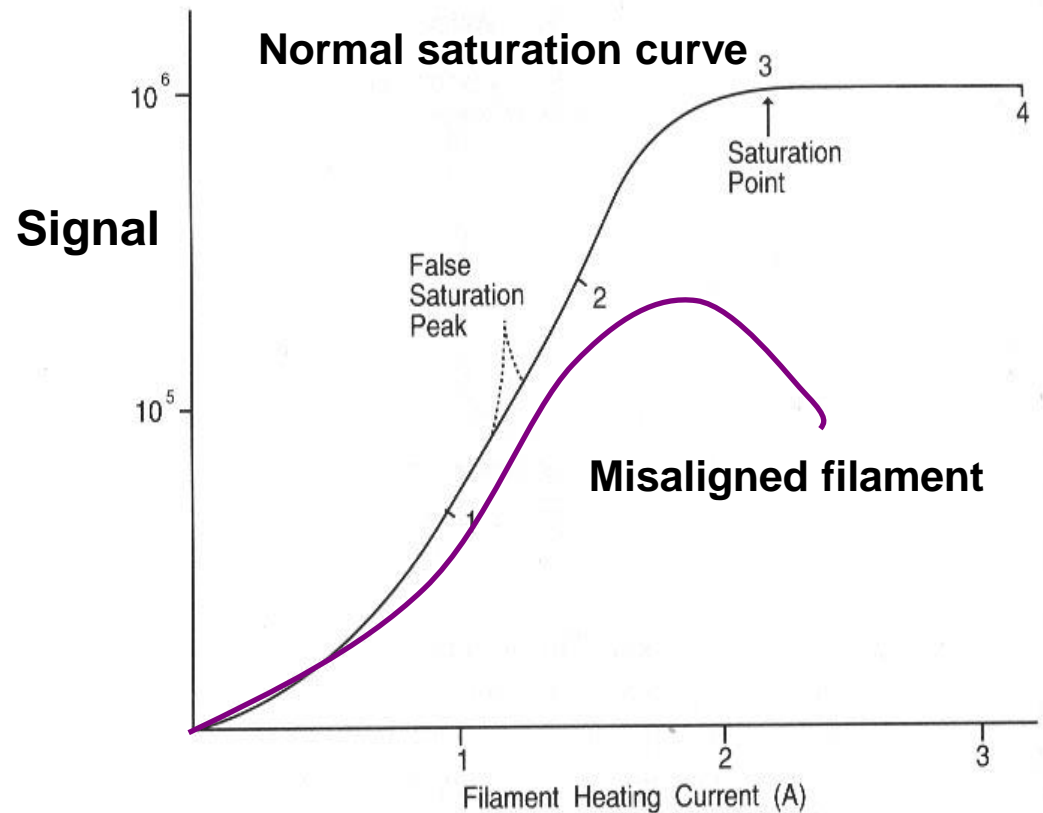
2. Turn up the filament current by advancing the Filament knob no faster than 1 division every 30 seconds
3. Load current and the signal on the screen will start to move once the Filament knob reaches ~12 o'clock
4. Do not let the ammeter reading go above 0.5 (vertical position). Increase Gun Bias if necessary to prevent this.



Scanning Electron Microscopy

Filament Saturation

5. Reduce brightness and contrast to keep the signal easy to see and entirely on the screen.
6. Saturation usually occurs when filament knob is ~2 o'clock. Do not go past 3 o'clock.
7. To correct a misalignment, reduce Filament current to ~12-1 o'clock and adjust gun alignment knobs to maximize signal. Resume saturation.



Scanning Electron Microscopy

Filament Saturation

8. If saturation is achieved, increasing the filament current slightly further will not raise the signal on the screen or increase the load current
9. Once saturation is achieved, adjust gun alignment knobs one pair at a time to move the signal to its maximum position on the screen, then re-check saturation



Scanning Electron Microscopy

Image Adjustment and Capture

1. Switch to PIC mode and focus the image on a non-Bakelite portion of your sample.
2. With the EXP scan rate, adjust contrast and brightness so the bars at the top of the image resemble the following configuration:



3. Using the TV scan rate, find a small round feature at ~1000x. Activate the focus wobbler with the 'ALIGN' button in the 'Function' → 'OL' group.
4. Adjust aperture position with the two silver knobs on the left side of the column, then deactivate the wobbler with the 'ALIGN' button.



Scanning Electron Microscopy

Image Adjustment and Capture

5. Zoom to 7500x or higher, focus, then adjust the X and Y stigmation controls one at a time with the corresponding knobs.
6. When you are ready to capture an image:
 1. Adjust contrast and brightness at EXP scan rate
 2. Open SEM Tech Image Grabber software on computer
 3. Switch to SLOW1 or SLOW2 scan rate – TV rate will not work with capture software
 4. Press ‘Shutter’ button near key on SEM console
 5. To freeze image on screen, click ‘Freeze’ button in capture software at any time during the capture
 6. Add annotations if desired (see ‘Annot’ menu). To measure features, you must first manually enter magnification.
 7. Save image in desired format then click ‘Live’ to return to live image.



Scanning Electron Microscopy Shutdown

- These procedures must be followed before opening the chamber, changing accelerating voltage, or shutting down the system entirely at the end of a session.
 1. Switch to LSP mode and EXP scan rate
 2. Increase magnification to maximum value (200,000x)
 3. Turn down Filament knob at a rate of 20-30 seconds per division to reach its minimum position
 4. Wait a couple of minutes then press red 'HT' button to turn off voltage
 5. Wait about 15 minutes to let the filament cool, then vent the chamber and remove sample while wearing gloves.



Scanning Electron Microscopy Shutdown

6. Close the chamber and pump down the system as before
7. After 'HT READY' state is reached, restore the following settings:

Accelerating voltage = 1.0 kV (minimum value)

'Spot Size' knob at 10 o'clock position

Objective aperture #2 is selected

8. Wait a couple of minutes then turn off power with key
9. Wait 15 minutes then turn off the chiller
10. Record ending time in log book and note any problems



Scanning Electron Microscopy

Maintenance and Supplies

- Filament changing procedures and detailed operating procedures are kept in binder by the SEM and in electronic form on computer desktop
- Cleaning supplies and K-type filaments (new or rebuilt) can be purchased at www.emsdiasum.com (link on computer desktop)
- Acetone and methanol purchased from Purdue Chemistry Stores

