

# Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

## FIBSEM – General Operation

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*These notes are meant to serve as an aid to assist users who have been trained and certified by MCC Staff. If ever you are unsure about the correct operation of the instrument or any of its components, please consult a MCC staff member before continuing. Never use equipment that you are not trained and approved to use.*

*Before using the MCC, please review the MCC User Handbook available through our website.*

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### **Login and Software Start Up**

1. Log on to iLab kiosk and start session
2. Start FEI Xp (quick launch icon on far left) if it's not already open
3. Login as "super", password "super" (not case sensitive)

### **Sample Loading**

1. Check stage position (X, Y, R, and T = 0, and Z = -12500)
2. Make sure that HV is off for both e- and i-beams
3. Click VENT
4. Valve on N<sub>2</sub> tank should be open
5. Load sample stub, tighten set screw and adjust height to match mark on guide
6. Click PUMP, click OK in sample holder dialog

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#### Start Up

1. When VAC OK ( $< 10^{-4}$ ), start HV for e-beam and i-beam [if the ion source is off, click “Beams On” button]
2. Setting the proper working distance
  - a. Start scanning the e-beam.
  - b. Rough focus on the surface of the sample (if no image, skip to steps 3a-d)
  - c. Link Z to free working distance (Z=FWD button)
  - d. Set Z to 7 mm
  - e. Focus at high mag and link Z=FWD again.
  - f. Set Z to 5 mm (optimum working distance for imaging and milling)
  - g. Focus and link Z=FWD
  - h. Set Z to 5 mm and sample should stay in focus
  - i. If necessary, correct astigmatism with X & Y stigmators
3. Session Column Alignments: Gun Tilt and Lens Alignments
  - a. E-Beam menu>Session Column Alignments>Lens alignments
  - b. Drag cross-hairs with FIB mouse to minimize lateral movement of image
  - c. Click OK
4. Finding eucentric height
  - a. Focus and center a recognizable object with X & Y
  - b. Tilt sample to  $30^\circ$  and bring object to center with Z knob on chamber door
  - c. Repeat at  $52^\circ$  so that object remains on center through tilt range
  - d. Tilt back to  $0^\circ$
  - e. Reiterate if object shifts more than  $\sim 5 \mu\text{m}$
  - f. Click STAGE IS AT EUCENTRIC HEIGHT in Stage menu
5. I-beam and e-beam coincidence
  - a. Tilt to  $52^\circ$ .
  - b. Click ZERO BEAM SHIFTS in Stage menu.
  - c. Focus and center recognizable object in e-beam with stage X & Y
  - d. Switch to i-beam (10 or 30 pA aperture and CDM-E detector) by clicking I-Beam Primary button.
  - e. Center object with BEAM SHIFT X & Y
  - f. Focus and stigmatize i-beam if necessary

**Heating the LMIS:** If suppressor is at maximum (+2150V) and ion emission is  $< 1.5 \mu\text{A}$ , then you may need to heat the filament. Please only heat the filament if you have been trained to do so.

1. Log in as supervisor (super/super).
2. Fill in pre-heating values on the heating log sheet in the binder.
3. Determine the necessary heating current from the table in the binder based on the V/I measured after last heating.
4. Set the heating current.
5. Source and HV are on.
6. Set suppressor to -2150 V.
7. Choose 1000 pA aperture.

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8. Start heating.
9. When heating is complete, adjust suppressor and extractor (see note on ion start up) to get a stable current of 2.2  $\mu\text{A}$ .
10. Wait  $\sim 5$  minutes, and click MEASURE to determine V/I slope (record on log sheet)
11. Return to user level

### Standard Platinum Deposition

1. Stage tilt should be  $52^\circ$
2. Sample should be at eucentric height (5mm).
3. Lower stage to  $z = 5.3\text{mm}$ .
4. Insert GIS needle
5. Bring stage back to  $z = 5\text{mm}$
6. If necessary shift image back to center
7. Draw pattern
8. Select Pt\_mag.mtr material file (pattern should be green)
9. Start patterning
10. Remove GIS needle when complete

### Shut-down procedures

1. Retract Omniprobe and GIS.
2. GIS stays ON.
3. Unlink Z and FWD (Stage menu)
4. Zero stage in X, Y, Z (-12,500), rotation and tilt.
5. Zero scan rotation for ion and electron beams.
6. Set lowest magnification.
7. Turn off HV for e- and i-beams, turn off source if you are the last user of the day.
8. Vent chamber.
9. Remove sample.
10. Close needle valve on  $\text{N}_2$  tank.
11. Log off CRF Access.
12. Fill in log in sheet.

### NOTES:

**Ion Source Start Up:** Make sure that i-beam HV is off. Make sure that the Extractor is set to 12.00 kV. Click SOURCE button. If ion emission is  $>2.2 \mu\text{A}$  or +++, reduce the suppressor voltage (steps of  $\sim 10 \text{V}$ ) until emission is  $2.2 \mu\text{A}$ . If emission is  $<2.2 \mu\text{A}$ , increase the suppressor voltage. If suppressor is out of range  $>2150 \text{V}$ , source may need to be heated.

**To clear sample touch alarm:** Make certain that the sample is not touching any part of the microscope using optical camera (OM under Detectors menu). Under File menu, choose LOG IN AS SUPERVISOR. super/super. Then attempt to move sample, and follow instructions in sequence of dialog boxes. When no more dialog boxes appear, choose SUPERVISOR LOG OUT from File Menu. Choose RETURN TO USER LEVEL.

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**Omniprobe use requires specialized training.** Please see CRF manager before attempting use.

**Emergency Shut Off Switch:** In the event of a fire, flood or other natural disaster please use the emergency shut off switch to cut power to the entire system. The emergency shut off switch is the large red button on the left side of the microscope console.

**X-ray Fluorescence:** All users who wish to use the iXRF system need to complete Drexel's Radiation Safety Training. Special note: the iXRF system is interlocked with the microscope vacuum so that the X-ray source is automatically shut off when the chamber is vented. The x-ray source can also be turned off by the OFF button on the controller.