UNIVERSITY OF CALIFORNIA, MERCED

SCIF CLEANROOM FACILITY

STANDARD OPERATING PROCEDURE (SOP)

Focused Ion Beam (FIB) Operation — FEI 800x Ion Beam Microscope

Location: SE1 154 (Cleanroom Class 1000)

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1. PURPOSE

To provide a detailed, standardized, and safe procedure for operating the Focused Ion Beam (FIB) system for nanoscale imaging, milling, and deposition, ensuring reproducibility, equipment protection, and user safety.

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2. SCOPE

Applicable to all trained users operating the FEI 800x FIB system for imaging, material removal, cross-sectioning, and deposition (e.g., Pt).

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3. RESPONSIBILITIES

- **Users:** Follow SOP, ensure correct beam parameters and safe operation
- **Core Staff:** Maintain system, provide training, manage source and GIS
- **Facility:** Ensure safety compliance, utilities, and contamination control

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4. SYSTEM OVERVIEW

The FEI 800x FIB uses a **Gallium (Ga^+) Liquid Metal Ion Source (LMIS)** to generate a focused ion beam for:

- **Imaging:** Secondary electron/ion imaging
- **Milling:** Material removal via sputtering
- **Deposition:** Localized Pt deposition via Gas Injection System (GIS)
- **3D Analysis:** Slice-and-view serial sectioning

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5. REQUIRED SYSTEM CONDITIONS

5.1 Utilities

- High vacuum system operational
- Stable electrical supply
- GIS gases available (Pt precursor)
- Cooling system (if applicable)

5.2 System Status

- Vacuum within operational range
- No alarms or interlock faults
- Beam column stable
- Stage calibrated

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6. SAFETY REQUIREMENTS

6.1 Major Hazards

- High voltage (up to ~30 kV)
- Ion beam radiation effects
- High vacuum hazards
- Toxic precursor gases (GIS)
- Gallium contamination
- Thermal and sputtering effects

6.2 PPE

- Cleanroom garments

- Safety glasses
- Nitrile gloves

6.3 Critical Safety Rules

- Never bypass interlocks
- Never open chamber under vacuum
- Do not expose skin or materials to beam unnecessarily
- Only trained users may operate GIS

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7. DETAILED OPERATION PROCEDURE

STEP 1: SAMPLE PREPARATION

- Ensure sample is:
    - Clean and dry
    - Vacuum compatible
    - Conductive or properly grounded
- Mount sample:
    - Use conductive carbon tape or clips
    - Ensure good electrical grounding

STEP 2: LOAD SAMPLE (LOAD-LOCK OPERATION)

- Place sample on holder
- Insert into load-lock
- Pump down load-lock:
    - Wait until required vacuum reached
- Transfer sample into main chamber

STEP 3: SET EUCENTRIC HEIGHT

- Move stage to working position
- Adjust Z-height:
    - Ensure beam pivot point is centered on sample

Purpose: Ensures accurate milling and imaging during tilt

STEP 4: SELECT BEAM PARAMETERS

Typical Settings

- Voltage:
    - Up to ~30 kV
- Beam current:
    - Low (pA range) → imaging
    - High (nA range) → milling
- Aperture:
    - Select based on resolution vs. current

STEP 5: IMAGING MODE

- Use low beam current
- Focus and stigmate beam
- Adjust:
    - Contrast
    - Brightness
- Capture high-resolution images

STEP 6: PATTERN DEFINITION

- Define milling or deposition region:
    - Rectangle, line, or custom geometry
- Set:
    - Depth
    - Dose
    - Scan strategy

STEP 7: MILLING OPERATION

- Use higher beam current
- Start milling:
    - Monitor sputtering behavior
- Adjust:
    - Current
    - Dwell time

Note: Watch for redeposition effects

STEP 8: DEPOSITION (GIS – Pt)

Pre-Check

- Confirm GIS system ready
- Verify correct gas selection

Operation

- Position GIS needle near sample
- Activate gas flow
- Use beam to decompose precursor

Result

- Local Pt deposition

STEP 9: PROCESS MONITORING

- Continuously monitor:
    - Beam stability
    - Pattern progress
    - Sample charging
    - Redeposition
- Adjust parameters as needed

STEP 10: COMPLETE PROCESS

- Stop beam
- Retract GIS needle (if used)
- Return system to imaging mode

STEP 11: SAMPLE UNLOADING

- Move stage to transfer position
- Transfer to load-lock
- Vent load-lock slowly
- Remove sample carefully

STEP 12: SYSTEM STANDBY / SHUTDOWN

- Set beam to standby
- Ensure GIS is OFF
- Confirm vacuum stable
- Log usage

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8. TROUBLESHOOTING GUIDE

Issue	Cause	Action
Charging	Non-conductive sample	Improve grounding
Poor resolution	Incorrect aperture	Adjust aperture
Redeposition	High beam current	Reduce current
Beam instability	Source aging	Check source
No deposition	GIS issue	Verify gas flow

9. CRITICAL DOs & DON'Ts

DO

- Use conductive mounting
- Start with low beam current
- Monitor process continuously
- Keep chamber clean

DON'T

- Use non-vacuum compatible materials
- Expose sample unnecessarily to beam
- Run GIS without training
- Ignore charging or contamination

10. CONTAMINATION CONTROL

- Use clean, approved samples
- Avoid oils, dust, and debris
- Clean holders after use

- Prevent cross-contamination

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11. WASTE HANDLING

- Dispose:
  - Contaminated wipes
  - Tape
  - Debris
- Follow cleanroom hazardous waste protocols

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12. EMERGENCY PROCEDURES

- Electrical issue → Stop system immediately
- Vacuum failure → Terminate process
- Gas leak → Stop GIS and notify staff
- Beam instability → Stop and report

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13. TRAINING COVERAGE

Users are trained on:

- FIB principles (imaging, milling, deposition)
- System components (column, GIS, detectors)
- Sample preparation and grounding
- Beam parameter selection
- Patterning and process control
- Safety and emergency procedures

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14. ACKNOWLEDGMENT & APPROVAL

Director Name: ________________________________

Director Signature: ______________________________

Date: ____

User Name: ___________________________________

User Signature: ________________________________

Date: ____