UNIVERSITY OF ILLINOIS AT URBANA-CHAMPAIGN
ENGINEERING AT ILLINOIS
OFFICE OF SAFETY

Standard Operating Procedure for Ion Mill Evaporator

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Date SOP was approved by PI/lab supervisor:	10/5/2016
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Type of SOP:	☐Hazardous material	☐Hazardous class	☒Hazardous Process
	(SOP describes a specific hazardous chemical)	(SOP describes a group of hazardous materials)	(SOP describes a hazardous process or equipment)

NOTE: This SOP is intended as an initial resource and as a general reference regarding the topic discussed. It is not a substitute for hands-on training and supervision by experienced laboratory personnel. The Principal Investigator must review and approve of all information in this document for the SOP to be valid and useable.

This SOP is not complete until: 1) Clear and detailed instructions are written that will ensure safe handling of the material or safe performance of the procedure, and 2) SOP has been approved and dated by the PI or laboratory supervisor.

Print a hardcopy and insert into your *Laboratory Safety Manual* and *Chemical Hygiene Plan*.

Table of Contents

Purpose

The purpose of this SOP is to inform users of the hazards while using this ion mill and have detailed instructions on how to use the instrument, including safety protocol. Because this instrument is contaminated with lead (Pb), it is important that users know details about lead and how it can affect them if exposed.

Key Points

- All lead depositions must be staff assisted
- All required PPE listed must be used for all experiments
- Proper cleaning procedures described must be completed before and after experiment

Hazard Awareness

Introduction

A **hazard** is a condition or circumstance that presents a potential for injury, illness or property damage. Hazards vary in nature and can be chemical, biological, physical (includes electrical, radiation, temperature extremes, pressure or vacuum, noise, mechanical), and ergonomic. The negative outcomes associated with hazards include exposure, poisoning, illness, shocks, burns, fires, slips and falls, spills, explosions, and perhaps even fatalities. It is important that you become aware of hazards inherent in the procedures undertaken or materials used in experiments. Only by identifying hazards can solutions and strategies be developed to address the hazards and control them or minimize the **risk** (likelihood of adverse events or negative outcomes associated with the hazards). In addition, there are **laws** that apply to many workplace settings, including academic research laboratories, that require the assurance of a safe workplace, as well as **regulations** that set and enforce standards that need to be complied with to ensure a safe and healthful workplace.

Hazards and pertinent regulations

Lead:

 Health Hazard	 Irritant	 Environment

- Lead can affect you when inhaled or swallowed
- Lead is a carcinogen and may be a TERATOGEN (birth defects)
- Contact can irritate the eyes
- Exposure can cause headache, irritability, and muscle and joint pain
- Repeated exposure can cause Lead poisoning with metallic taste, colic, and muscle cramps
- Lead may damage the nervous system
- Exposure may cause kidney and brain damage, and anemia
    - OSHA PEL/ACGIH TLV: 50 µg/m^3 averaged over an 8-hour period (air)
    - HUD: 40 µg/ft^2 (surfaces)

Copper:

 Flammable	 Irritant	 Environment

- Contact can irritate and burn skin and eyes
- Inhaling copper can irritate the nose and throat; possibly causing a hole in the "bone" (septum) dividing the inner nose
- Can cause headaches, nausea, vomiting, diarrhea, and abdominal pain.
- Can cause a flu-like illness
- Can cause a skin allergy
- May affect the liver and kidneys
    - OSHA PEL/ACGIH TLV: 1000 µg/m^3 averaged over an 8-hour period (dust and mists)
    - OSHA PEL: 100 µg/m^3 averaged over an 8-hour period (fume)
    - ACGIH TLV: 200 µg/m^3 averaged over an 8-hour period (fume)

Chromium:

 Irritant

- Contact can irritate and burn the skin and eyes, possibly causing damage to the eyes
    - OSHA PEL: 1000 µg/m^3 averaged over an 8-hour period (air)
    - ACGIH TLV: 500 µg/m^3 averaged over an 8-hour period (air)

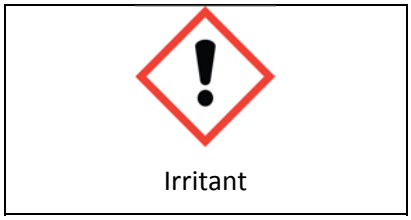
Nickel:

 Health Hazard	 Irritant

- Handle as a carcinogen

- Skin allergen
- May be absorbed through the skin
- Can irritate and possibly burn the skin and eyes
- May cause flu-like illness
- Can cause headache, dizziness, nausea, and vomiting
- Can cause a hole in the septum bone dividing the inner nose
- May affect the liver and kidneys
- Nickel powder is a flammable solid
    - OSHA PEL: 1000 µg/m^3 averaged over an 8-hour period (air)
    - ACGIH TLV: 1500 µg/m3 averaged over an 8-hour period (air)

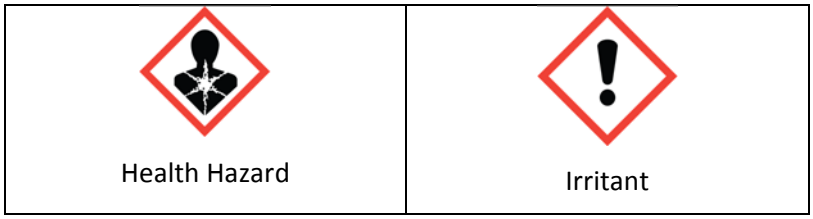
Indium:



Irritant

- Can affect you when you breathe it in
- Can cause irritation to the eyes
- May irritate the nose, throat and lungs – causing coughing and shortness of breath
- May damage the lungs
- High levels could damage the liver and kidneys
    - ACGIH TLV: 100 µg/m^3 averaged over an 8-hour period (air)

Molybdenum Oxide:



Health Hazard

Irritant

- Suspected of causing cancer
- Can irritate the eyes and skin
- Dust or mist may irritate the nose, throat, and lungs causing coughing
- Can cause weight loss, diarrhea, poor muscle coordination, headaches, and muscle/joint pain
- Repeated exposure can reduce red blood cell count
- Can affect the liver and kidneys
    - OSHA PEL: 5,000 µg/m^3 averaged over an 8-hour period (air)

- ACGIH TLV: 500 µg/m^3 averaged over an 8-hour period (air)

Means to control the hazards

Specific means of controlling the above hazardous can be found in the work practices, engineering controls, and PPE section.

Examples of hazardous materials or processes

- **Exposure to Potentially Toxic Evaporated Material**

Some of the evaporation sources used in this system may be toxic, with the most common toxic materials and their hazards listed in the previous section. Users should recognize that even if the current process is non-toxic, any or all of the above toxic materials may be present in the chamber or on other work surfaces due to previous depositions. Note that users who use the ion mill only (and not the evaporator) are at lower risk but should still regard the chamber as contaminated. When handling sources or loading or unloading the vacuum chamber, users should wear sufficient PPE to minimize exposure: safety glasses, long sleeves or a lab coat, nitrile gloves, and a dust mask. The interior of the chamber should NEVER be touched with bare skin.

The chances of exposure are greatest when taking boats or source material in or out of the chamber, and when removing/replacing the cylindrical evaporation shield. When the shield is removed during chamber loading, it should be set ONLY on the dedicated mill/evaporator work bench, to avoid contaminating other surfaces. Extreme care should be taken when moving the shield in or out of the chamber; impact or friction could generate flakes or dust of previously evaporated material. Used evaporation boats, coils, and other containers should be labelled and treated as hazardous material; if disposal is necessary use the Division of Research Safety's waste pickup service. Users evaporating lead should use dedicated tools (hex key to attach boats, tweezers, any screwdriver used to attach sample) that are clearly labelled as being potentially contaminated with lead.

- **High Currents and Voltage**

The ion mill power supply and evaporator power supply in this system regularly experience voltages up to 500 V and currents up to several amps, exposure to which can cause severe injury or death. The evaporator current passes directly through user-installed boats in the bottom of the chamber, in addition to the thick copper cables below/behind the chamber. The mill current is supplied through the shielded cable attached to the white disk on the upper left side of the instrument. When using either the mill OR evaporator functions, current should be ramped up slowly. A short or other electrical malfunction may be indicated by: an interlock or warning light on the control panel, power getting "stuck" at lower than expected values or fluctuating wildly, or a burning smell or other indication of a fire. If any of these occur, power should be lowered and a staff member consulted.

Both the milling and evaporating power supplies should be turned on ONLY when the chamber is sealed and pumped. Before venting the chamber, ensure that both supplies are turned off. In particular, before adding/removing/adjusting an evaporation boat or other source material, ensure that the evaporator percentage reads zero (or the rate controller is turned off) and the source-selection switch in the lower-right cabinet is set to "off."

- **Pressurized Gas**

The argon gas ionized in this system is stored in a pressurized vessel than may exceed 2,000 PSI. High pressures may also be present in the helium compressor used to drive the systems cryopump. Both these gases are inert, so their only potential hazard is the high pressure itself. A leak in the Ar cylinder, Ar regulator, or He compressor lines can potentially cause a dangerous explosion, and even small leaks can displace air from the lab space, causing asphyxiation. The high-pressure components of this system should not be modified by users, but a staff member should be contacted if a user suspects any damage to pressurized gas plumbing.

- **Cryogenic Fluid**

Liquid nitrogen (LN2) is potentially used to cool the sample stage during ion milling, in which case liquid or cold vapor is present in the large gray storage vessel next to the instrument, the ¼" tubing feeding through the center of the chamber door, and any lines connecting the two. Note that LN2 may be present in the storage vessel even when the user does not use stage cooling.

The extremely cold temperatures of LN2 (-196 degrees Celsius or 77 Kelvin) can cause tissue to freeze immediately upon contact causing burns or frostbite to the skin or eyes. Indirect contact through cold evaporated vapor or a thermally conductive container may still be dangerous. During any cryogen transfer or interaction with a cooled surface, users should wear cryogen gloves that are loose-fitting (to be easily removed in case of a spill), safety glasses, closed-toe shoes, and long pants. Note that contact with LN2 is potentially MORE dangerous than other, colder cryogens because of its high latent heat (resistance to evaporation). Note that LN2 will condense oxygen out of the air into a flammable liquid, so heat and ignition sources should be avoided when sample stage cooling is used. In particular, avoid routing cryogenic lines near the milling and evaporation power supplies.

The high evaporation rate of LN2 poses an asphyxiation hazard. In many ways a LN2 vessel may be addressed similarly to a pressurized gas vessel (see above). Cryogenic fluid expands by a factor of several hundred when vaporized and may displace oxygen rapidly. Gradual asphyxia may not be noticeable. To avoid leaks or sudden boil-off during sample stage cooling, all fittings and tubing attached by the user to the sample stage feedthrough should be leak-tight and any tubing should be rated for cryogen or thick-walled. Typically, this means using hose clamps on thick "gum rubber" tubing. Thin-walled tubing (e.g. Tygon) should never be used for cryogenics. If any leak is observed (e.g. a hissing sound), the user should immediately shut off LN2 flow, let the system warm up, and repair it or seek staff assistance. To avoid oxygen displacement and thermal shock of cryogenic plumbing, only low flow rates of LN2 should be used for stage cooling. High flow rates require that the exhausted cryogenic vapor be directed outside of the lab space. The LN2 storage vessel should never be pressurized by an additional gas source – its pressure from internal boil off should be sufficient for all operations.

- **Explosion/Implosion Risk of Vacuum Systems**

Because of the large pressure difference, weak points in a vacuum system can present the possibility of implosion or explosion. The mill/evaporator's vacuum system should not be modified, even temporarily, without consultation of a staff member and any components under vacuum should be rated for the pressures present. Impact-resistant safety glasses should be worn during any usage of the instrument. The risk of a sudden vacuum breach is greatest near mechanical feedthroughs and

viewports or other glass components, so care should be taken when interacting with these parts. The attached Bayard-Alpert ion gauge warrants particular caution; during normal operation it will become hot enough that the glass is somewhat susceptible to thermal shock or breakage. The area near the gauge must be kept clear and under no circumstances should the hot gauge be touched while the chamber is under vacuum.

- **Burn Hazard**

Both the ion mill grids and thermal evaporation sources will heat up to several hundred degrees Celsius during normal operation. In principle this may present a burn hazard. However, under standard operation the vacuum chamber should never be opened while these components are hot, and thus the user should not be exposed to high temperatures. After either an ion milling process, or an evaporation, the user should wait a minimum of **30** minutes (preferably 45+ minutes) before opening the chamber. Additionally, users should NEVER touch the ion-mill electrodes/grids inside the chamber. This serves the dual purpose of protecting the user from high temperature and protecting the instrument from thermal shock or oxidation damage.

- **Sharps**

Two types of sharps are potentially present in this system: 1) silicon, microscope slides, or other thin, hard wafers that may be used as substrates and 2) razor blades that may be used to remove samples from the sample stage, if secured with adhesive. If contaminated with lead (i.e. because the wafer or blade was used during a toxic deposition process), the sharp should be disposed in the “Lead Contaminated Waste” container., otherwise sharps should be disposed in a standard sharps disposal container. Lead-contaminated razor blades may be retained for multiple uses in the designated container of lead-contaminated tools.

Important considerations

Prior approval from PI required? Choose an item.

Yes

Consultation of other reference material, documents or knowledgeable persons required?

User needs to consult with staff.

Pre-requisite training or skill?

Yes. Training is required. They have to be a MRL user and have to be given access from a MicroFab staff member after reviewing their specific SOP.

Experiment Risk Assessment required?

If necessary, consult the ***Reference*** section for help with Risk Assessments:

Research must receive approval from staff for their deposition material.

Other important considerations:

- User must schedule a 'staff-assisted session' with staff
- User must list their usage in the log books provided
- Staff must be present for loading, heating, depositing, and venting
    - Staff does not need to be present when pumping

Emergency response

Introduction to emergency response

While prevention of lab accidents is preferred, preparation for emergency situations is an essential part of good lab practice.

Necessary emergency equipment

There is a fire extinguisher in the room. A shower is located outside of the room, down the hall in the north east corner. The electrical panel is located on the right side of the door to the room, if power needs to be cut to the machine.

Possible emergencies:

Exposure to toxic materials

Alert staff of the incident and seek medical attention right away.

Electrical shock

Injuries caused by electricity include electrical shock, burns, and falls due to electrical shocks and burns. Electrocution is a fatal electrical shock. Electrical shock occurs when current passes through the body. The severity of the shock depends on:

- Amount of current flowing through the body,
- Path of current through the body,
- Length of time the body is in the circuit.

Fuses and circuit breakers are safety devices that protect equipment from high currents or voltages and prevent overheating of electrical wires. They are rated for a certain voltage and maximum current and come in two types: fast and slow blowing. Too much current flowing through a wire can cause a power cord to overheat and start a fire. Sparks from electrical equipment can ignite flammable materials

The electrical panel is located on the right side of the door to the room, if power needs to be cut to the machine.

Cryogenic exposure (asphyxiation)

Cryogens are liquefied gases such as nitrogen, argon, or helium that pose significant hazards due to their low temperatures. They are colorless, odorless and extremely cold. Solid carbon dioxide, referred to as dry ice, has handling hazards due extreme cold (-78 ºC). The high rate of evaporation of cryogens displaces oxygen in the surrounding air and can reduce oxygen levels to the point where rapid suffocation can occur without warning. One volume of a cryogenic liquid will expand to about 700 equivalents of gas when evaporated. Oxygen deficiency sets in when the oxygen content drops below 19.5%. The decrease in oxygen accompanied by gradual asphyxia is usually NOT noticeable by the victim. One pound of dry ice will produce 250 L of carbon dioxide gas. Within 24 hours, 5-10 pounds of dry ice

can sublimate. Concentrations of more than 0.5% (5000 ppm) carbon dioxide in the air can lead to unconsciousness.

Respond immediately if there is an accidental condensation of oxygen or argon. Open the system to the atmosphere. Shut off the source of vacuum if it is present. Place a blast shield in front of the apparatus and allow the system to slowly warm to room temperature. It is important that this is an open system to minimize the risk of an explosion upon warming. The sudden boiling of the cryogenic liquid is a significant hazard. Liquid oxygen mixed with organics increases the explosion hazard significantly. Alert researchers in the area and inform DRS.

If skin or the eyes are exposed to cryogens or the cold vapor, use warm water (up to 108 ºF/42 ºC, NOT above 112 ºF/44 ºC) to restore normal body temperature. Do NOT rub the frozen skin. Seek medical attention. Remove or loosen clothing that may restrict blood flow to the frozen area.

In the case of a large spill or rupture of a container, evacuate the area while alerting others. Oxygen deficiency might make the area unsafe to enter. Call 911.

Vacuum implosion

Personal protective equipment should be used when working with systems under vacuum. Protect yourself from potential flying glass or debris by using safety glasses, goggle, face shield, and/or a blast shield when near an apparatus under vacuum.

Emergency shutdown/evacuation procedure

If in an emergency that needs user to evacuate the room immediately, push the evaporator OFF button on the evaporator control panel, and turn the ID3500 (Ion Gun Controller) power switch to OFF. Then leave the room.

What to do if there is a material release or a fault in the process.
Contact Fubo Rao and then follow the emergency shutdown and evacuation procedure that is listed above.

What to do if there is an exposure or injury
Life threatening situations, call 9-1-1.

Storage

Introduction to proper storage

The storage of chemicals goes beyond simply placing bottles on shelves for easy retrieval. Proper storage involves careful separation of incompatible chemicals, examination of containers for integrity, use of appropriately-sized secondary containment and management of time-sensitive or temperature-sensitive chemicals. Improper storage has led to unsafe conditions and incidents.

Special storage requirements

- The tools and razor blades are provided and stored in the labelled boxes.
- Any tool or razor blade for the ion milling system should stay in the room. Users should not take any tool out of the room.
- Users' own lead tools/equipment should be kept separate from other items
- Users should keep any lead-containing samples or evaporation sources in a sealed container, clearly labelled, in their own labs. Do not leave lead-containing samples, evaporation containers, or pellets in the user facility.

Quantity limits and other storage considerations

N/A

Work Practices and Engineering Controls

Introduction to work practices and engineering controls

Safe work practices describe known safe and prudent policies and practices to adhere to in performing the experiment or procedure or in handling the materials. These practices are listed below. Some chemicals are acutely toxic or carcinogenic and require a **designated area** for work with these chemicals. A designated area may be the entire laboratory, an area of the lab, or a containment device such as a fume hood, and if present will be indicated below. Safe work practices may require the use of **engineering controls** and **personal protective equipment** (PPE). **Engineering controls** are methods that are part of the equipment or process to minimize the hazard to the researcher, and if needed should be indicated below. **Personal protective equipment** or PPE refers to protective clothing, attire or garments designed to protect the wearer's body from injury or exposure.

Recommended work practices

- Wear the required PPE
- Clean-up work spaces before and after
- Have a staff member present before starting a lead deposition

Designated area to work with the material or process

- Lead containing materials must only be placed on the Millatron work surface. (insert picture)

Necessary engineering or administrative controls

If necessary, consult the ***Reference*** section for help with controls.

- Place a vent control on the ion mill to prevent from venting too quickly
- Use provided HEPA vacuum to pick up any dust, particles, or flakes.
  - This HEPA vacuum stays inside of 334MRL
- Use "D-Lead" wipes to clean working surfaces before and after using lead materials
- Use provided tools for ion mill work and keep them in 334 MRL
- Place any "D-Lead" wipes, paper towels, and gloves in the provided "lead contaminated waste" bucket

Required Personal Protective Equipment.

If necessary, consult the ***Reference*** section for help with PPE selections.

- Nitrile gloves (doubling the gloves is preferred)
- Safety glasses
- Provided Tyvek lab coats (only for toxic depositions)

Detailed procedures **Introduction**

Successful experiments require adherence to correct procedure. After completion of the experiment, all waste material should be collected in properly labeled containers.

Step-by-step procedures

Lead Deposition/Other Depositions/Ion Milling

SAFETY Before:

- PPE: Gloves, safety glasses, lab coat
- User must have a staff member present when starting a lead deposition
- Use the provided "D-Lead" wipes to clean all work surfaces
- Use ONLY the provided tools

Procedure:

1. About the Error lamp

The "Error" lamp will flash whenever any of the following three problems occurs:

- If the "VENT" and "PUMP DOWN" buttons are illuminated at the same time.
- If "PUMP DOWN" is "ON" and the cryo pump is not under vacuum.
- If "VENT" is "ON" and the ion gauge is "ON".

2. Stop and think

If you see the Error indicator flashing. Don't act impulsively, or you may make things worse. Be sure to release the button you DON'T need, leaving the one you want "ON". The system will immediately act to either VENT or PUMP in response to the illuminated button that remains "ON" when the other is released. Be careful!

Inform staff if the lamps in the illuminated "VENT" and "PUMP DOWN" buttons fail, so we can replace the bulbs. You and your fellow users need these lamps to know what state the buttons are in.

3. Warning

A new set of carbon grids for the ion gun costs more than $10,000. Be forewarned that your research group will be billed for the replacement cost of the grids if you damage them through carelessness or violation of the basic rules of operation. The grids are fragile and easily damaged by

- Touching (careless handling)

- Exposure to air when hot (venting too soon after milling)
- Exposure to water vapor and oxygen in the plasma (poor vacuum while milling).

4. Rules of Operation

- Never touch the carbon grids with any object.
- Do NOT ion mill until the vacuum is better than **$5x10^{-6}$ Torr**.
- After use, allow the system to cool at least **30 minutes** before venting.
- Only authorized users can use this tool.

5. Venting and Pumping the Chamber

****** If loading for a lead (or other toxic) deposition, staff must be present for loading step (but not the entire pump-down).**

****** All lead (or other toxic) depositions must be followed by deposition of a low-toxicity material; remember to load a source for this secondary material. Copper is recommended, unless the user requires a specific secondary layer on their sample.**

******You need to ask staff to verify that you have deposited the low-toxicity material, e.g. Cu, when you are ready to unload you samples after your depositions of lead (or other toxic materials)**

5.1 Venting Chamber

- Gauge controller: turn off the ion gauge by pressing the IG1 button once.

- System controller: If the PUMP DOWN button is illuminated, press the PUMP DOWN button once (if the PUMP DOWN button is not illuminated, skip this step). The light should turn off. You need to hear a loud sound: Hsss…CLUNK! This is the main gate valve closing. If you don't hear this sound, it is <u>not</u> right. Please push the PUMP DOWN button again, and inform staff.

- System controller: Press the VENT button. The button will be illuminated after you press it. You will hear a mild pop as the system starts to vent. There may be a brief delay (10-15 seconds) to assure closure of other valves.

- Wait 5 min until the chamber reaches atmospheric pressure and the door opens.

- System controller: Once you see the door opens up a little. Press the VENT button again to turn off the nitrogen venting gas. The VENT button light should be off.

5.2 Loading samples

- Attach sample to stage, either mechanically (screws) or with vacuum-compatible adhesive. Kapton tape is ideal (avoid bubbles), carbon tape, or very small quantities of vacuum grease are okay. Other adhesives are not acceptable for vacuum.

- If you need to load materials for thermal evaporation, gently remove cylindrical evaporation shield, gripping it by the sides ore bottom (the top "lid" may come off independently). Avoid bumping the crystal monitor and DO NOT BUMP THE MILLING FILAMENT OR GRIDS (upper left).

- Load the evaporation boats – tighten movable electrode first, then stationary electrode. If adding new source material (e.g. pellets, shot or wire), do so *after* boat is secured. Otherwise the material will probably get knocked out while securing the boat. Electrodes are numbered 1 and 2 from left to right.

- **Load Cu pellets to another boat, if lead or other toxic materials are to be evaporated. Ask staff to be present while loading.**

- Replace crystal monitor if necessary, put the evaporation shield back.

5.3 Pumping Down Chamber

- System Controller: press the PUMP DOWN button while pushing the chamber door to help pumping. If the ERROR indicator flashes, make sure the VENT button is not illuminated (if it is, press the VENT button again to release it).

- Wait. After 5-10 minutes, the gate valve should open with a loud noise, allowing the cryopump to continue the pump-down. The ion gauge should be turn ON by itself. Allow another 45min to 1 hour for the cryopump to pump down the chamber.

- If the gate valve does not open after 10 minutes, or if you hear additional valve operations not described here, STOP the pump-down by pressing the PUMP DOWN button again. GET STAFF HELP. Don't try to fix the problem yourself.

6. Ion Milling

- Wait until the chamber pressure is **$2X10^{-6}$ Torr** or better before milling.
- Main chamber: Make sure the shutter is closed, so that your sample is not exposed to the beam while you are setting up the ion gun. Gauge controller: turn off the ion gauge by pressing the IG1 button once.
- Mass Flow Controller: Set display knob, bottom right, to channel #3 for Argon.
- Mass Flow Controller: Toggle switch #3 up to enable flow of Argon using channel 3. The green LED should light.
- System controller: Press the "Argon Gas" switch. You will hear a valve open. The button should light up.
- Wait few seconds, the Ar flow should stabilize at about 3-5 sccm. Turn on the ion gauge. The pressure should be about $3X10^{-4}$ Torr. If the pressure is outside of the range of $2X10^{-4}$ to $5X10^{-4}$ Torr, do not attempt to ion mill. Get staff help.
- System controller: Press the Ion Miller button. The button should light up.
- ID3500 (Ion Gun Controller): Turn all knobs fully counterclockwise. Then turn the power ON.
- ID3500: Push the SOURCE switch (lower left, next to the BEAM switch) up to turn it ON. The “Discharge” LED in the STATUS display will start flashing.
- ID3500: Turn DISCHARGE VOLTAGE up to 120 volts. Here’s how: Under the DISCHARGE knob, push the switch down to select the VOLTAGE display. Observe the upper right digital display (“MONITOR”). Its LED indicator will move to V (Voltage). Turn the DISCHARGE knob until 120V is shown under MONITOR.
- ID3500: SLOWLY turn the CATHODE FILAMENT current up until "Discharge" LED is stable. Do NOT set the current any higher than necessary to make the "Discharge" LED stable. Here’s how: Under the CATHODE knob, push the FILAMENT switch up to FILAMENT. The MONITOR LED will change to A (Amperes) and display about 2.8 A. Turn the CATHODE knob to increase the current until the DISCARGE LED in the STATUS display stops blinking and stays on. The current should be around 6 A.
- ID3500: Adjust DISCHARGE VOLTAGE to 40 V. Here’s how: Under the DISCHARGE knob, push the switch down to VOLTAGE. Under the MONITOR display, the LED indicator will move to V (Voltage). Increase the voltage from about 30 V to 40 V using the DISCHARGE knob.
- ID3500: Push BEAM switch (lower left, next to the SOURCE switch) up to ON.

- ID3500: Turn the BEAM VOLTAGE up to the desired level. Here's how: Under the BEAM VOLTAGE knob, push the switch down to VOLTAGE. The LED under the BEAM display (upper left) will change to V. Increase the voltage from about 47 V to the desired voltage (250 V to 500 V). Do not exceed 500 volts without staff approval.
- ID3500: Turn the ACCELERATOR VOLTAGE up to 10 to 20% of BEAM VOLTAGE. Here's how: Under the ACCELERATOR knob, push the switch down to VOLTAGE. The LED at the MONITOR display will change to V. Adjust the accelerator voltage to 10% to 20% of BEAM VOLTAGE (that is, for 250 V beams, use 25V-50V; for 500 V beams, use 50V-100V).
- ID5300: SLOWLY adjust the CATHODE FILAMENT current to give the desired beam current (50-100mA). Here's how: Turn the CATHODE knob to get 50mA to 100 mA at the BEAM display. Do not exceed 100mA without staff approval.
- ID3500: Adjust the NEUTRALIZER CURRENT to 10 mA above the desired BEAM CURRENT. Here's how: Under the NEUTRALIZER knob, push the switch down to EMISSION and adjust mA value displayed in MONITOR to 10 mA above the desired beam current value, e.g. if desired beam = 63mA, set monitor = 73mA.
- ID5300: With the neutralizer filament now emitting electrons, the BEAM current may be higher than the value you set. Readjust the CATHODE FILAMENT current to give the desired beam current. Here's how: Turn the CATHODE knob to get the desired value at the BEAM display.
- Open the shutter to expose your sample to the ion beam. YOU ARE NOW ION MILLING.

To End Ion Milling

- Main chamber: Close the shutter to stop the exposure of your sample to ion milling.
- ID3500: Turn off the ion source by first pressing Beam OFF, then Source OFF. Then turn all knobs fully counterclockwise. Turn off the Power on ID3500
- Mass Flow Controller: Turn the Ar flow off by pushing the channel #3 switch to the OFF position. The LED should not be illuminated.
- System controller: Press the "Argon Gas" switch. You will hear a valve close. The button should no longer be illuminated.
- Turn off the ion gauge. It will also turn off the Ion Miller button light.
- Wait at least 30 minutes before venting the chamber.

7. Evaporation

***** If depositing lead or other toxic material, staff must be present for initial sample loading and warming of the source.**

***** All lead (or other toxic) depositions must be followed by deposition of 50nm of Cu, BEFORE venting the chamber. If the user does not desire an additional layer on their sample, they may deposit the 50 nm of Cu with the sample shutter closed, or the stage facing away from the evaporation sources.**

- Inficon thickness monitor: Turn on the monitor by pressing the POWER button. Press PG to enter program mode. Set DENSITY, Z-RATIO, and TOOLING FACTOR. Set Tooling Factor = 65%. Press PG to return to the main screen.
- Inficon thickness monitor: To test the lifetime of thickness monitor crystal sensor, press START, then MANUAL, and XTAL. A value will show up for a few seconds at the bottom of the screen (for example, “04% XTAL”). A new crystal gives XTAL = 0%, an old crystal gives XTAL = 100%. If XTAL>90%, ask staff to replace the monitor crystal. If the crystal has failed, a message: XTAL FAIL will be at the bottom of the screen. If "XTAL FAIL" appears, ask staff to replace the monitor crystal.
- Vent the chamber. Load samples, boats and source materials.
- Pump the chamber. Wait until the chamber pressure is $2X10^{-6}$ Torr or better.
- Right Hand Cabinet: Choose the correct boat you wish to heat (#1 or #2) using the large switch below the pump control panel.
- System Controller: Push EVAPORATE ON.
- Inficon thickness monitor: Press START, then MANUAL, and ZERO to reset the thickness monitor.
- Use the Inficon “mouse” (the hand-held control) to adjust % POWER.
- Slowly increase the power. When the rate is reached, open the shutter to begin evaporation. When done, close the shutter, and slowly reduce the power to zero.
- System Controller: Push the EVAPORATE OFF button.
- Repeat these instructions if evaporation from another boat is required.
- Deposit 50 nm of copper with the sample shutter closed.
- Wait at least 30 minutes before venting the chamber.

8. Liquid Nitrogen Cooling of Sample Stage (Optional)

- If desired, the user may cool the sample stage with liquid nitrogen (LN2) during milling or evaporation. Usually, this is intended to avoid sample damage during lengthy ion-milling, esp. of polymers. Nitrogen flow should be setup after reaching base pressure but before milling/evaporating.
- Ensure nitrogen is available in the storage Dewar next to the instrument (check level indicator).
- Attach a length of cryogen-compatible tubing from the storage Dewar to one of the ¼" pipes on the central vacuum feedthrough leading to the sample stage. Secure the end on the feedthrough with a hose clamp.
- Similarly, attach tubing going from the second feedthrough pipe to the lab's exhaust system (if available). If adequate air flow in the lab can be established and a low flow rate is used, nitrogen may be vented directly into the lab. Again, use hose clamps to prevent leaks.
- SLOWLY open the valve on the LN2 storage Dewar. This valve is extremely sensitive – opening it less than 1/8 of a turn is usually sufficient. Listen and watch for any hissing or jets of gas – if any leaks are present, stop cryogen flow and fix them or consult staff.
- Note that as the cryogen tubes cool down, the flow rate may change; throttle it down if necessary. Nitrogen exiting the sample stage should be gaseous; if the exhaust nitrogen is still liquid, the flow rate is way too high. Tubing may become frozen and brittle – do not try to bend it or rapidly warm it.
- Whenever nitrogen flow is no longer required (after milling or evaporation), close the Dewar valve, and wait for any tubing to warm and thaw before removing it.

9. Finish

- **If unloading after a lead (or other toxic) deposition, staff must be present.**
- Wait for milling grids or evaporation sources to cool before chamber is opened.
- If liquid nitrogen cooling was used, the sample should be warmed back to room temperature to avoid condensation on the sample stage during venting. Flow *gaseous* nitrogen through the same ¼" pipes used to cool the stage. The nearby nitrogen blow gun can be used for this purpose. The sample is sufficiently warm when the exhausting gas is no longer cool to the touch.
- Turn off *Pump Down* and listen for gate valve to close. Turn on *Vent*. Wait 5 minutes for chamber to vent, then turn off *Vent*, remove samples, and remove any evaporation sources.

- If the evaporation shield was removed to retrieve sources, make sure to replace it, then *Pump Down* again. The chamber should be left pumping at the end of any session.
- Fill out logbook, all fields are required.
- Cleanup work area (see below for hazardous material handling).

SAFETY After:

- Use the provided "D-Lead" wipes to clean all work surfaces
- Use the provided "D-Lead" wipes to clean the provided tools
- Place the used "D-Lead" wipe, paper towels, and used gloves into the "Lead Contaminated Waste" container
- Make sure razor blades are put back into supplied container

Waste disposal procedure.

If necessary, consult: Division of Research Safety Waste Disposal Guide and Division of Research Safety Chemical Waste Quick Start Guide

For users:

- Place all "D-Lead" wipes, paper towels, and used gloves into the "Lead Contaminated Waste" container
- Dispose of any wanted razor blades in the provided sharps containers

For Staff:

- Complete a waste request through the DRS system
  - Here are the instructions

Reference

Definition of terms

OSHA: Occupational Safety and Health Administration

- Main federal agency charged with the enforcement of safety and health legislation

ACGIH: American Conference of Governmental Industrial Hygienists

- The association for advancing occupation and environmental health

PEL: Permissible exposure limit

- The legal limit for exposure of an employee to a chemical substance or physical agent

TLV: Threshold limit value

- This is the level of a chemical substance to which it is a believed a work can be exposed day after day for a working lifetime without adverse effects

DRS: Division of Research and Safety

- Major resource for accident response, safety programs, training and waste management

PPE: Personal Protective Equipment

- refers to protective clothing, helmets, goggles, or other garments or equipment designed to protect the wearer's body from injury or infection.

Routes of exposure

- Different routes of exposure include inhalation (breathing), ingestion (eating/drinking), and dermal contact (skin)

Carcinogen

- A substance capable of causing cancer in living tissue

Teratogen

- An agent or factor that causes malformation of an embryo

Pneumoconiosis

- A disease of the lungs due to inhalation of dust, characterized by inflammation, coughing, and fibrosis

Fume

- Gas, smoke, or vapor that smells strongly or is dangerous to inhale

Dust

- Fine, dry powder consisting of tiny particles waste matter lying on the ground, surfaces, or carried in the air

Mist

- Tiny water droplets suspended in the air

Tools and resources

Tools for Performing a Lab Risk Assessment

Hazard recognition and identification is the first step to creating a risk assessment for your laboratory procedure. The following links provide guidance in identifying hazards and assessing the risks from the hazards.

American Chemical Society: Hazard Assessment in Research Laboratories

Division of Research Safety: Standard Operating Procedures

Tools for selection of hazard controls

Once the hazards have been identified, control measures aim to eliminate or mitigate (lessen) the risk from each hazard. American Chemical Society: Control Measures

Chemical fume hoods are an important engineering control as they provide protection from vapors, splashes and impacts from chemicals and their reactions: Division of Research Safety: Fume Hoods

PPE should be considered as the last line of defense against exposure to hazardous materials. If used, they should be selected correctly to protect against the hazardous material and fit the wearer. Division of Research Safety: Personal Protective Equipment

Change management

The SOP needs to be reviewed on an annual basis and whenever events or conditions arise that trigger a review, such as:

1. An incident or significant near miss.
2. Modifications to equipment other than replacement in kind.
3. Use of a commercial product for a purpose for which it was not designed.
4. Increased risk beyond what is covered in the SOP.
5. New experiment, equipment, or control software.
6. A change/improvement in a SOP or other program document is discovered.
7. New materials are introduced to an experiment that were not accounted for in the SOP.
8. Changes in essential personnel.

It also helps to maintain a change management document that lists sections or items in the SOP that need to be checked in every review, such as web links or other information that might become outdated.

Reference material

Prudent Practices in the Laboratory. Handling and Management of Chemical Hazards. NRC (National Research Council). National Academy Press: Washington, DC, 2011.

Identifying and Evaluating Hazards in Research Laboratories. ACS (American Chemical Society) 2015.

GHS PICTOGRAMS & HAZARDS

As of June 1, 2015, the Hazard Communication Standard (HCS) will require pictograms on labels to alert users of the chemical hazards to which they may be exposed. Each pictogram consists of a symbol on a white background framed within a red border and represents a distinct hazard(s). The pictogram on the label is determined by the chemical hazard classification.

CORROSION

- Skin Corrosion/Burns
- Eye Damage
- Corrosive to Metals

EXCLAMATION MARK

- Irritant (skin and eye)
- Skin Sensitizer
- Acute Toxicity
- Narcotic Effects
- Respiratory Tract Irritant
- Hazardous to Ozone Layer (Non-Mandatory)

EXPLODING BOMB

- Explosives
- Self-Reactives
- Organic Peroxides

SKULLS & CROSSBONES

- Acute Toxicity (fatal or toxic)

FLAME

- Flammables
- Pyrophorics
- Self-Heating
- Emits Flammable Gas
- Self-Reactives
- Organic Peroxides

GAS CYLINDER

- Gases Under Pressure

ENVIRONMENT

- Aquatic Toxicity

HEALTH HAZARDS

- Carcinogen
- Mutagenicity
- Reproductive Toxicity
- Respiratory Sensitizer
- Target Organ Toxicity
- Aspiration Toxicity

FLAME OVER CIRCLE

- Oxidizers

https://www.osha.gov/dsg/hazcom/pictograms/

Record of changes made to this SOP

Describe the changes made to this document since its creation. Identify the personnel who made the edits or revisions and when the change was made.

Date of change	Changed by	Description of change
9/23/2016	Maisie Swanson/Edward Chainani	Safety aspects for the new SOP/Rough Draft
9/28/2016	M. Swanson, E. Chainani, Fubo Rao, Tao Shang, Adam Weis	Adding operational aspects and emergency response details
10/4/2016	Fubo	Additional information for procedure
10/6/2016	Maisie	Reviewed and made small final changes
6/25/2018	Fubo	Reviewed and made small final changes