Molecular Beam Epitaxy Chamber Maintenance

Standard Operating Procedure

Revision Date: 06/20/22

Laboratory Director (LD) Approval is Required Prior to Performing this Procedure

This standard operating procedure (SOP) covers procedures for opening the molecular beam epitaxy (MBE) chamber any time there is a potential for the release of hydride gas such as arsine or phosphine. Review this document and supply the information required in order to make it specific to your laboratory.

# Description [Provide additional information as it pertains to your research protocol]

Epitaxy refers to the deposition of a crystalline overlay on a crystalline substrate and must take place in highly controlled conditions. Molecular beam epitaxy (MBE) is a method of epitaxy for thin-film deposition of single crystals. The substrate is first, heated and then fired with precise beams of atoms or molecules. When these molecules land on the surface of the substrate, they begin to build up and condense.

## Process [Write the steps for using the chemical in your research protocol]

# Potential Hazards [Provide additional information as it pertains to your research protocol]

Solid arsenic that has coated the interior of the chamber can hydrolyze in the presence of moisture to form arsine gas when the chamber is open. If phosphorus has been used in the chamber, there is a potential for the release of phosphine gas. Solid phosphorus coating on the interior of the chamber also presents the potential risk of fire, which is not covered in this SOP. Any MBE chamber with heavy phosphorus contamination will need to be sent off campus to a company that specializes in this type of decontamination. Contact Environment, Health & Safety (EHS) for assistance if there is any question regarding the ability of the lab to decontaminate the chamber.

Other solid contaminants on the interior of the chamber such as beryllium, gallium, indium, aluminum, nickel, bismuth, silicon etc. present the potential for inhalation or skin contact.

The highest concentration of arsine or phosphine (hydrides) will usually be detected when the chamber is first opened. Typically, after 5 - 15 minutes, the concentration will fall below detectable limits. However, this time period can be lengthened given the amount of humidity in the air, the amount of arsenic or phosphorus present and the surface area involved.

Once the initial oxidation has taken place, activities that will cause additional hydride releases are:

* Working inside the chamber and disturbing the surface layer – usually results in a transient release
* Removing and handling parts from the chamber
* Transferring contaminated parts to the fume hood

Using a portable air sampling instrument throughout the process is critical to monitor for the presence of hydrides in the lab.

## Occupational Exposure Limits (OELs):

Arsine  
ACGIH: **5 ppb, 8-hour** TLV

## Phosphine

ACGIH: **300 ppb, 8-hour** TLV

ACHIH: **1 ppm, 15-minute** STEL

# Engineering Controls [Provide additional information as it pertains to your research protocol]

Ideally there will be a snorkel exhaust available to place at the opening of the chamber. In the absence of a snorkel exhaust, a Nilfisk vacuum with a combination HEPA filter/carbon filter cartridge is to be secured near the opening of the chamber.

MBE tools should only be installed for use in laboratories with a high air change rate and a one pass system – i.e. the air from the lab should not be recirculated back into the building. The lab should be under negative pressure with respect to surrounding areas.

# Work Practice Controls [Provide additional information as it pertains to your research protocol]

Prior to chamber opening, contact EHS to arrange for the use of a TLD (toxic leak detector) portable air sampling instrument, and open-head metal drums for waste collection.

* The contact for the TLD is: EHS Research Health & Safety (734) 647-1143
* The contact for the metal drums for waste collection is EHS Hazardous Materials Management (HMM); (734 763-4568). Specify the size of drum you will need (15, 30 or 55-gallon) and that it needs to be open-head.

EHS will provide initial training on the TLD, which is used to detect hydride gases. When you receive the TLD, check to be sure you also have the following components with it:

* A tape for hydride gases
* An arsine key or phosphine key, whichever is appropriate
* A test card to verify the optical sensor is accurate, along with a sheet of instructions for testing the sensor

Ensure waste drums and poly sheeting to cover the floor are present in the lab before beginning. The drums should be labeled with a hazardous waste label. The contents will be “Solids contaminated with arsenic.” All gloves, booties, disposable suits, poly sheeting and any materials used to clean the area will go into the waste drum. \*\*Prior arrangement with EHS-HMM is necessary for the disposal of any larger components or assemblies from the MBE.\*\* Unless putting waste into the drum, the cover should remain on at all times.

Place poly sheeting to cover the floor around the chamber and under the drum. Place the TLD near the chamber opening.

Ensure anyone not directly involved leaves the lab prior to beginning the work. Start the TLD and once it has gone through its startup sequence, place the sampling tube at the chamber opening. Use tape to secure the tubing and keep it out of the way during work, being careful not to obstruct the opening of the tube. Place the snorkel exhaust or vacuum as close to the chamber opening as possible, but not next to the tubing for the TLD.

Don all PPE (full face cartridge respirator with HEPA/OV cartridges, disposable coveralls, booties, nitrile gloves) and then place the snorkel or Nilfisk vacuum at the chamber opening. Open the chamber and note the reading on the TLD. One person must be designated to observe the reading on the TLD at all times. If the concentration rises above 5 ppb during the course of the work, place the snorkel or vacuum at the chamber opening and leave the lab. Check the reading on the TLD again after a few minutes and if below 5 ppb you can resume working.

Upon completion of work, discard outer pair of gloves and don a new pair. Use disposable towels dampened with water to wipe down the floor and other horizontal surfaces around the chamber where arsenic may have fallen. Discard the towels in the waste drum.

When removing PPE, first remove the outer pair of gloves and discard them in the waste drum. Next remove the suit, then the booties. Remove the second pair of gloves and then remove the respirator; cartridges should be discarded in the waste drum. Respirator cartridges worn during an MBE opening should not be re-used. After all waste is in the drum, seal it and call EHS-HMM for a pick up.

# Personal Protective Equipment [Provide additional information as it pertains to your research protocol]

Full face air purifying respirator with HEPA/OV cartridges, disposable coveralls or lab coat, safety glasses, nitrile gloves (double glove) and booties are required.

Wearing respiratory protection requires participation in the U-M respiratory protection program, which is administered by EHS. The program involves a physical with a physician at Occupational Health Services, and respirator fit testing and training by EHS. Any new group members should contact their EHS Representative to begin the process of obtaining necessary respiratory protection.

# Waste Disposal [Provide additional information as it pertains to your research protocol]

Contact EHS-HMM at (734) 763-4568 for waste containers, labels, manifests, waste collection and for any questions regarding proper waste disposal. Also, refer to the EHS [Hazardous Waste](http://ehs.umich.edu/haz-waste/) Web page for more information.

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# Training of Personnel

All personnel shall read and fully adhere to this SOP when opening a chamber on a MBE.

# Certification

I have read and understand the above SOP. I have received approval from my Lab Director to perform this procedure. I agree to contact my Lab Director if I plan to modify this procedure.

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### Major Revisions (Tracking purposes only -- Do not print as part of SOP)

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| Date | Revision |
| 09-14-18 | EHS name and logo were added, updated the formatting, and revised the content under Exposure/Unintended Content (AKJ). |
| 02-25-19 | Updated Links (DML) |
| 06-20-22 | Reviewed (WBD) |