

EECS 143 Microfabrication Technology

# **1.0 Safety Rules and Chemical Handling**

# 1.1 General Laboratory Rules

You MUST wear safety glasses in the EE143 Lab at all times. For those lab members who wear prescription glasses, prescription safety glasses may be obtained – or you Must wear safety glasses or goggles over your prescription glasses.

Wearing contact lenses in a research laboratory environment is discouraged. While UC does not have a policy forbidding the wearing of contact lenses in combination with safety glasses, it is not recommended. Contact lenses are prone to absorbing or trapping chemicals possibly prolonging or aggravating an accidental exposure. The campus NanoLab does not recommend wearing contact lenses with safety glasses

Sandals, open toe shoes, high heels and bare feet are prohibited in the EE143 Lab. Shorts are only permitted when wearing the full coverall cleanroom suits.

Food and beverages are not allowed in any part of the EE143 Lab

Cell phone usage is not permitted in the EE143 Lab.

Radios, music players and headphones are not allowed in the EE143 Lab.

When in a lab, walk, don't run. Avoid backing up; always look where you are going. Don't rush. Wash your hands after working in the EE143 Laboratory – or any laboratory. Remember that the nitrile gloves must be worn at all times while in the EE143 Laboratory, and are intended to protect equipment and materials as well as your hands. Avoid touching your face with your gloved hands. Check your gloves frequently; put on fresh gloves if they become torn or contaminated.

It is campus wide EH&S policy that gloves are not to be worn outside of laboratories. This is to ensure that any contaminants that might be on your gloves are not transferred outside the laboratory when touching door handles, elevator buttons, etc. Be sure to remove all gowning items including all gloves as you depart the laboratory. It is not acceptable to keep gloves on to "protect the cleanliness" of your samples as you transfer them to another laboratory. Use plastic bags or boxes to transfer your samples. It is best laboratory practice and UCB EH&S recommends that students and researchers wash their hands after leaving any laboratory, even if no known exposure has occurred.

### 1.2 Working With Chemicals

Most chemical handling is done at a fume hood or a wet process station. Working with chemicals outside of a fume hood or wet process station is prohibited. Fume hoods and wet process stations are exhausted, with face velocities greater than 100 feet/minute. Make sure fume hoods equipped with moveable sashes have these sashes adjusted to their indicated position. This assures proper exhaust velocity. Specialized equipment, such as the photoresist dispense and develop tools, have engineering controls (exhaust ducts and additional fans) to handle fumes and prevent exposure.

# 1.2.1 Personal Protective Equipment

You must wear chemically-rated gloves, chemical resistant apron and a face shield when working with any chemicals. The following clarifications to this rule are noted:

- If you are working at a sink, even if you are only handling wafers, chips and DI water, you must have apron and face shield; this is to provide protection from others working at the sink who may be handling acids or heating solvents
- The tri-polymer chemically resistant gloves are not required when handling small amber bottles of photoresist. If you are at a sink and using a spinner, face shield and apron are still required.
- Gloves, apron, and face shield are not required for incidental use of IPA or acetone with squeeze bottles at locations away from the sinks (e.g. when wiping down the sealing surface of a vacuum chamber).

### 1.2.2 Working with and disposing of Acids

At many points in the fabrication process strong acids are used as cleaning solutions or etchants.

Acids used in the EE143 Lab are:

sulfuric acid, H2SO4, in "piranha" cleaning solutions;

nitric acid, HNO3, in the pre-mixed polysilicon etch solution;

hydrofluoric acid, HF, in the polysilicon etch and silicon dioxide etch solutions

Acids can cause severe burns to exposed skin or severe eye injury or blindness if splashed in your eyes. Especially dangerous is hydrofluoric acid (HF acid), a component of most silicon oxide and other glass etchants. HF burns do not hurt immediately on contact, but by the time they do start to hurt, significant injury may already have occurred. The result is a severe burn which is very painful over prolonged periods and is slow to heal, sometimes taking several weeks.

Always wear protective clothing, including a face mask, chemically resistant gloves, and apron when handling corrosive chemicals. Check gloves for pinholes or cracks before putting them on. The protective wear should be stored according to the following rules to avoid contamination: a) Aprons should be returned to the hanging posts with the "front side" (clearly marked) facing into the wall. This is noted by a sticker on the wall. This procedure prevents the potential of chemicals being spilled onto an apron, and then the contaminated side being used as the inside of the apron inadvertently. b) Gloves should be stored in plastic bags. Only ONE pair of gloves belongs in each bag. Please dry the gloves as much as possible prior to storing them following each use. When mixing acids with water, remember to *ADD ACID TO WATER and NOT WATER TO ACID!* A large amount of heat is released when strong acids are mixed with water. Adding more acid releases more heat. If you add water to acid, you form an extremely concentrated solution of acid initially. So much heat is released that the solution may boil violently, splashing concentrated acid out of the container! If you add acid to water, the solution that forms is initially very dilute and the small amount of heat released is not enough to vaporize and spatter the solution. This is a commonly known rule from many high school and university chemistry lab classes that is often summarized as "add the acid last".

It is important to be aware of an exception to the "add acid last" rule. In the EE143 Lab, during several lab sessions, you will prepare a wafer cleaning solution called "Piranha". This solution is a mixture of sulfuric acid and hydrogen peroxide. <u>Piranha solution is made by first pouring the sulfuric acid (H2SO4) and then slowly adding the hydrogen peroxide (H2O2).</u> With this mixture, this is the best method to minimize the chance of spattering. Be aware that reaction between hydrogen peroxide and sulfuric acid is exothermic, producing heat. The container you mix these chemicals in will become very hot. A fresh solution of Piranha can easily reach temperatures above 100°C! Chemical spills have occurred when a lab member has prepared a piranha solution, grasped their beaker or container in order to move it, and dropped it when discovering it was too hot to hold.

Use the chemicals only in the designated area; do not transport chemicals around the room in beakers. Never pour chemicals back into the original container.

Use appropriate containers for your solutions. For instance, do not use glassware for hydrofluoric (HF) acid as HF acid will etch glass. Only some plastics are chemically resistant to HF and other strong acids. Examples of acid compatible plastics are Teflon and high density polyethylene. Some plastics have limited use temperatures; never use a plastic container on a hotplate.

The following materials are used for handling chemicals in the EE143 Lab.

HF acid solutions:	Teflon or high density polyethylene
Sulfuric acid solutions including piranha:	pyrex or quartz
Photoresist developer solutions:	pyrex
Photoresist stripper solutions:	pyrex

Always ask the TA if you are unsure what material to use with which chemical.

#### **Disposing of Acids**

hydrofluoric acid (HF) solution:	ACID WASTE funnel at the acid sink.
silicon etchant (a mixture of nitric acid and HF):	ACID WASTE funnel at the acid sink.
piranha (mixture of H2O2 & H2SO4):	ACID WASTE funnel at the acid sink.
Rinse water:	drain disposal using aspirator

### CAUTION

\*\*\* Never discard any acid solutions into the organic waste funnel at the solvent sink \*\*\* \*\*\* This can result in violent reaction and explosion \*\*\*

# 1.2.3 Working with and Disposing of Organic Solvent Solutions

At many times in the fabrication process, organic solvents are used as cleaning solutions or strippers. Organic solvents can be irritants to the skin and harmful when inhaled. They should be handled only in an appropriately vented area.

Solvents used in the EE143 Lab are:

isopropanol, (CH3)2CHOH, also known as 2-propanol, commonly known as rubbing alcohol;

acetone, (CH3)2CO, commonly found in nail polish remover

N-methyl-2-pyrrolidone, found in many paint strippers, referred to as NMP,

this is the main ingredient in the photoresist stripper known as PRS-3000

### **Disposing of Organic Solvents**

isopropanol:	organic waste funnel at the solvent sink
acetone:	organic waste funnel at the solvent sink
NMP:	organic waste funnel at the solvent sink
Rinse water:	drain disposal using aspirator

# CAUTION

# \*\*\* Never discard any organic solvents into the acid waste funnel at the acid sink \*\*\*

\*\*\* This can result in violent reaction and explosion \*\*\*

# 1.2.4 Working with and Disposing of Photoresist Developer Solutions

At many times in the fabrication process, mild base solutions are used for developing exposed photoresist. Like acids, base solutions, can cause severe burns to exposed skin or severe eye injury or blindness if splashed in your eyes. Especially dangerous are tetramethyl ammonium hydroxide (TMAH) solutions. The chemical structure of TMAH is very similar to that of the neurotransmitter acetylcholine. Therefore, TMAH absorption through the skin not only causes chemical burns but exposure to concentrated TMAH solutions can quickly disrupt respiratory and circulatory signaling causing heart attack and death.

Base solutions used in the EE143 Lab are:

dilute tetramethyl ammonium hydroxide ( <3% TMAH);

this is the primary ingredient in pre-mixed photoresist developer solutions

no solutions >5% TMAH are handled in the EE143 Lab.

# **Disposing of Photoresist Developer Solutions (< 3% TMAH base)**

<3% TMAH solutions: organic waste funnel at the solvent sink

#### ALWAYS ASK WHEN UNSURE OF PROCEDURE!

This is the only way to maintain a safe laboratory for yourself and your fellow students.

### 1.3 Chemical Exposure

If you are exposed to chemicals, you must immediately remove all affected clothing. For all chemical exposures, flush the area with water for at least 15 minutes. If your eyes are exposed, it is critical to flush them with flowing water as quickly as possible. Continue to do so for at least fifteen minutes to ensure good diffusion.

If you are exposed to a chemical while working at a wet process station (especially if splashed in the eyes), utilize the DI water deck hose to flush your eyes instead of trying to make your way to an eyewash station. Hold your eyes open and flush continuously for 15 minutes. The DI deck hoses will often be your fastest response for such an emergency when working at a wet process station or sink.

All eye exposure is regarded as a serious health issue. As a precaution, all exposures to the eye are to be treated as potentially serious and require a visit to an emergency room for a check up, following exposure and initial flushing. Contact a staff member as soon as possible for assistance or call 911.

Special Instructions for HF exposure to the skin:

1. Remove contaminated clothing.

2. Flush with cold water for 15 minutes. Pay particular attention to the area around your fingernails and cuticles.

3. Gently massage calcium gluconate ointment into skin. There is a container of this material at each wet process station in the EE143 Lab.

4. Report any HF exposure or use of calcium gluconate to a staff member as soon as possible for assistance or call 911.

If you are unsure of whether your skin contacted a chemical it is best to calmly remove any affected clothing and insure a thorough (15 minute) rinse. If the suspect exposure was an HF acid solution it is best to also treat the area with the calcium gluconate solution after full rinsing. If you feel discomfort after flushing contact a staff member as soon as possible for assistance or call 911.

# **1.4 Chemical Information**

Students should be aware that the Office of Environment Health and Safety has safety information for all chemicals in the lab (and for any other lab on campus). This information is contained in the Material Safety Data Sheets (MSDS) and is available to any student or employee of the University who works with chemicals.

MSDS sheets for all the chemicals used in the EE143 Lab are available on the shelf of gowning area.

# **2.0 Contamination Control and Cleaning Procedures**

Contamination can be divided into two classes: Chemical and particulate. Examples of chemical contamination are sodium and chloride ions from sweat and oils from your skin. Examples of particulate contamination are dust from the air, exfoliated skin, and hair. Humans are usually the dirtiest objects in a clean room.

Never touch semiconductor specimens, the insides of beakers, the wafer-handling end of tweezers, or photolithographic masks with your fingers. This also applies to furnace boats (especially) or any other equipment which can transfer particles from your skin to the semiconductor devices. Obviously, contaminating a furnace (for example, by touching the boat and then inserting it into the furnace where any contaminants are vaporized) will ruin not only your attempts at device fabrication, but those of all who follow.

#### Cleaning Procedures

General cleaning procedures for preparing your sample follow. They fall primarily into three categories: (1) removal of gross contaminants; (2) removal of organic contaminants; and (3) removal of light and heavy metallic ion contaminants. All instruments used, i.e., beakers, tweezers, etc., must be cleaned before being used to clean wafers.

1. Cleaning Tweezers, Wafer Boxes and Plastic Wafer Holders

Scrub parts in DI water containing several drops of the liquid detergent, "Nova Clean". Rinse well in DI water. Blow dry.

Note: Nova Clean liquid is available in Nanolab.

#### 2. Cleaning Glass and Teflon Beakers

Scrub with Nova Clean diluted in DI water.

Rinse in DI water. Perform the "water break" test to determine if the item is clean: submerge the item in DI water for several seconds and then lift out. Observe the film of water as it drains off the item. On a clean surface, this film will remain unbroken and fairly uniform. Contaminants will cause breaks or sharp irregularities such as "islands" to appear in the color fringes and droplets that remain on the surface.

If necessary, glassware can be further cleaned in dilute (10:1) HF. This removes the outer layer of glass.

Rinse in DI water for 5 minutes.

Let dry top down, on lint-free paper.

3. Correct Wafer Handling Technique

Contamination:

To minimize contamination from tweezers, plastic "holders" are available for handling wafers in solutions, and should be used in lieu of tweezers. Wafer drying is accomplished by drying with the nitrogen guns, or by spinning in special spinner so designated.

#### Avoiding Wafer Breakage:

Wafers can be both quite strong and yet quite brittle. If a wafer is in a typical wafer boat, it is standing on edge in a pair of slots cut into the boat. After processing wafers in this position, they often become lodged tightly into these grooves. To remove the wafer you can use tweezers to pull directly up on the wafer. This is most easily done by looking down on the top edge of the wafer. From this vantage point you can be sure that you are not applying a torque that can easily shatter the wafer. A common mistake is to look at the side of the wafer while you do pull on the wafer. From this vantage point it is difficult to tell whether or not you are pulling directly up on the wafer and so often an excessive amount of torque is generated which breaks the wafer.

You also want to avoid torque when you attempt to remove a wafer from a flat vacuum chuck, such as that in the spinner we use when applying photoresist. If the vacuum is on or if there is a film of liquid beneath the wafer, it may be difficult to get the wafer off the chuck. Using a pair of tweezers to pry and lift up the wafer from an edge will exert torque on the wafer and care must be taken to avoid applying an excessive amount.

4. Cleaning Furnace Apparatus after Contamination

5% HF:H2O for 20 seconds.

Rinse, blow dry.

Bake for 30 minutes.

5. Cleaning Photomasks

This procedure should be performed at least 30 minutes before photo step.

Dip in acetone for 15 minutes.

Rinse.

Scrub with cotton ball dipped in EKC Mask Cleaner. Rinse well.

Soak in 1:1 2-propanol:water for 15 seconds. Soak in 100% 2-propanol for 15 seconds. Blow dry.

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