

# Metal Assisted Etching for Silicon

## Standard Operating Procedure

**Lab Manager: Dr. James Vlahakis**

**Faculty Supervisor: Prof. Robert White, Mechanical Engineering (x72210)**

**Tufts Emergency Medical Services are at x66911.**

For more information on metal assisted etching:

Jae-Hong Lim. "Three-dimensional Hierarchical Te-Si Nanostructures", *Nanoscale*, 2014, **6**, 11697-11702

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**Warning:** Hydrofluoric Acid (HF) is an extremely dangerous material. Because of the ability of HF to produce severe *delayed* tissue damage without necessarily producing pain, any contact with HF should receive immediate first aid and medical evaluation, even if the injury appears minor or no pain is felt. Work carefully in the hood with full personal protective equipment, including apron, face shield and trionic gloves.

If HF does get on your skin, rinse the affected area with water for 5 minutes, and then apply calcium gluconate gel. The calcium gluconate is on the wire shelves in the lithography room. Have someone in the lab call **Tufts EMS at x66911**.

**Purpose:** Metal assisted etching is a process used to etch patterns into a silicon substrate. A metal pattern is deposited and then the substrate is placed in a solution of oxidized acid. The metal acts as a catalyst and material under the metal is removed. If, for example, a square of metal is deposited, a square depression will be etched into the substrate. The metal is not consumed in the process.

### **1. Material Requirements:**

**1.1 Equipment:** Three polyethylene beakers or polymethylpentene screw cap jars, PTFE tweezers or stainless steel 4" wafer tweezers, PTFE wafer handling tools

*Warning: HF attacks glass! You cannot put it in a glass container. Polymethylpentene and polyethylene are fine to use. Polystyrene (plastic Petri dishes are often polystyrene!) is not considered compatible with long-term HF exposure.*

**1.2 Chemicals:** Hydrofluoric Acid 50% (HF), Hydrogen Peroxide 30% (H<sub>2</sub>O<sub>2</sub>), Ethanol

#### **1.2.1 Hazards associated with these chemicals:**

**HF:** liquid or vapors are extreme health hazards; cause severe burns and bone loss, which may not be immediately painful or visible. Significant exposure (100 mL) to HF can kill directly. Please use extreme caution; HF is very hazardous, both acutely and long term.

**H<sub>2</sub>O<sub>2</sub>:** Liquid or vapors are serious health hazards; and can cause severe burns. Waste jugs require a ventilated cap, ensure one is available before proceeding. Mixing with solvents can produce very energetic reactions

(explosions), use care.

Ethanol: A volatile, flammable solvent. Avoid heat sources. Do not breath fumes. Conduct processing in the fume hood.

**1.3 Engineering Controls**: Conduct procedure in ventilated fume hood. Store bottles of HF (sealed tightly) in the acid cabinet with secondary containment. Hydrogen peroxide is stored in the bases cabinet. Ethanol is stored in the solvents cabinet. Work area should contain an eye wash and safety shower.

**1.4 Personal Protective Equipment**: Trionic gloves on top of nitrile gloves, apron, goggles, and face-shield. Never work with HF without all this equipment.

## **2.0 Procedure**:

For information on preparing a photoresist mask, see the SOPs on Standard Lithography, the OAI Aligner, and the Spinner. For information on metal deposition, see the SOP on NSC 3000 Sputter Deposition. For information on Piranha Clean, see the SOP on Piranha Clean. For Gold Etching, see the SOP on Wet Etching of Gold.

### **Complete all processes in the acid process fume hood.**

#### **2.1 Metal Deposition:**

**2.1.1** Perform a Piranha clean (see Piranha Clean SOP) on a silicon substrate.

**2.1.2** Perform lithography (see Standard Lithography SOP).

**2.1.3** Deposit 20nm of gold on a clean silicon substrate (see NSC 3000 Sputter Deposition SOP). Make a positive pattern of the desired etching area. If, for example, a square of metal is deposited, a square depression will be etched into the substrate.

#### **2.2 Oxidized Acid Etch, variable time**

**Warning: HF etches glass. Therefore, you must do HF processing in polyethylene or polymethylpentene containers only.**

**2.2.1** Place fab wipes in a pile in the hood. Get a polyethylene beaker or polymethylpentene jar that will fit your samples for processing (you should find one labeled "HF" on the shelves). Put it on the fab wipes in the hood.

**2.2.2** Measure the following chemicals and pour, in this order, into a polyethylene beaker or polymethylpentene jar. To measure the HF, use a small graduated cylinder and a funnel (to prevent spilling); *graduated cylinder and funnel must be made of polyethylene or polymethylpentene.*

120ml Ethanol

12ml HF

2ml H<sub>2</sub>O<sub>2</sub>

**2.2.3** Transfer the sample into the solution carefully with tweezers or PTFE wafer tools.

**2.2.4** Let the sample etch for the required length of time (depending on etch depth). Expect approximately 1 micron per hour etch rate.

### **2.3 DI Water Rinse, 3 x 5 min:**

- 2.3.1 While the sample is etching, fill the first rinse beaker (polyethylene beaker or polymethylpentene jar) with enough DI water to cover the sample.
- 2.3.2 When the etch is complete, transfer the sample carefully to the first rinse beaker with tweezers or PTFE wafer tools.
- 2.3.3 Leave the tweezers/tools (which have HF on them now) to soak in the DI water beaker with the sample.
- 2.3.4 Let the sample and tweezers soak for 5 mins.
- 2.3.5 Fill the second rinse beaker with DI water.
- 2.3.6 If you do not plan to do another etch, dump the used solution into a dedicated waste bottle labeled with all chemical names. (Bring the waste bottle into the hood to pour it! There is a polyethylene funnel you should use. The waste bottle must also be made of polyethylene.) Refill the etching container with DI water and again dump it into the waste container.
- 2.3.7 Rinse the etching container two more times, but now dump the rinse water into the “dilute acid/base waste” 5 gallon HDPE dilute water waste container. Put the clean etching container aside to dry.
- 2.3.8 After the 5 minutes are up, transfer the sample to the second rinse beaker. Dump the first rinse beaker into the waste bottle.
- 2.3.9 Refill the first rinse beaker or jar with DI water while the sample is in the second rinse beaker.
- 2.3.10 After the sample has soaked for 5 more minutes in the second rinse beaker, transfer it back to the first rinse beaker (which now has fresh DI water in it). Let it soak in there for 5 more minutes.
- 2.3.11 Dump the water from the two final rinse beakers into the dilute water waste container.
- 2.3.12 Rinse both beakers two more times with DI water and dump them into the dilute water waste container.

### **2.4 Sample Dry:**

- 2.4.1 After the third waste rinse is finished, remove your samples and blow them dry with the air gun.
- 2.4.2 After removing the majority of the water with the air gun, you can further dry your wafers on a hotplate or in the convection oven if needed.

### **2.5 Metal Removal:**

The metal pattern is not consumed in the etching process and will remain at the bottom of the etched features.

- 2.5.1 Remove the gold using gold etchant (see Wet Etching of Gold SOP).

## **3.0 Cleanup**

- 3.1 Return all labware to its proper location. The beakers can drip dry on fab wipes in the hood or on the bottom shelf of the storage shelving.
- 3.2 Wipe up any drips in the area with chemical wipes and dispose in either acid trash or solvent trash as appropriate. If you don't know what the drips are, dispose in acid trash.

#### **4.0 Storage:**

- 4.1 Hydrofluoric acid should be stored in the acid cabinet in a tightly capped polyethylene bottle.
- 4.2 Hydrogen peroxide is stored in the bases cabinet.
- 4.3 Ethanol is stored in the solvents cabinet.

#### **5.0 Waste Disposal:**

##### 5.1 Etching Mixture waste:

- 5.1.1 Wipes are disposed of in the acid trash can.
- 5.1.2 Liquid waste is collected in the dedicated waste container and stored in the satellite storage area with secondary containment.
- 5.1.3 Dilute water waste from second and third rinses goes into the dilute water waste 5 gallon HDPE tank.

##### 5.2 HF waste:

- 5.2.1 HF wipes are disposed of in the acid trash can.
- 5.2.2 Liquid waste is collected in the HF waste container and stored in the satellite storage area with secondary containment.
- 5.2.3 Dilute water waste from second and third rinses goes into the dilute water waste 5 gallon HDPE tank.

##### 5.3 Hydrogen Peroxide:

- 5.3.1 Wipes should go in the acid / base waste bin.
- 5.3.2 Liquid waste should go in the Standard Clean-1 Etch waste bottle. This container can be glass or HDPE.

##### 5.4 Isopropanol:

- 5.4.1 Solid waste should go in the solvent/photoresist trash.
- 5.4.2 Liquid waste should go in the solvent/photoresist liquid waste bottle.

#### **6.0 Accident Procedures:**

Read MSDS prior to working with any chemical to familiarize yourself with the symptoms of exposure and recommendations for treatment.

##### 6.1 HF Acid:

- 6.1.1 Skin contact: Rinse affected area with water for 5 minutes, removing contaminated clothing during the rinse. Apply generous amounts of calcium gluconate gel to the area. **Get immediate medical attention. Don't be shy. Call the medical center if you got HF on your skin. Tufts Emergency Medical Services are at x66911.**
- 6.1.2 Eye contact: Immediately flush with water for 20 minutes while holding the lids open. Do not apply calcium gluconate. **Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.**
- 6.1.3 Ingestion: Do not induce vomiting. **Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.**

- 6.1.4** Inhalation: Remove to fresh air. Resuscitate if necessary. Take care not to inhale any HF released from the victim's lungs. **Get immediate medical attention. Call Tufts Emergency Medical Services at x66911.**
- 6.2 Ethanol:**
  - 6.2.1** Skin contact: Wash with soap and water.
  - 6.2.2** Eye contact: Flush eyes with flowing water for at least 15 minutes.
  - 6.2.3** Ingestion: Wash out mouth with water. Contact physician.
  - 6.2.4** Inhalation: Remove to fresh air. If breathing is difficult, contact emergency personnel.
- 6.3 Hydrogen Peroxide:**
  - 6.3.1** Skin contact: Remove contaminated clothing, rinse affected area with water for 10 minutes. If there is a visible burn, get immediate medical attention.
  - 6.3.2** Eye contact: Immediately flush with water for 20 minutes while holding the lids open. Get immediate medical attention.
  - 6.3.3** Ingestion: Do not induce vomiting. Get immediate medical attention.
  - 6.3.4** Inhalation: Remove to fresh air. Resuscitate if necessary.
- 6.4 Spill:**
  - 6.4.1** If a small, contained spill occurs, such as inside the hood, wipe it up with chemical wipes and dispose of in the appropriate trash container (acid trash).
  - 6.4.2** If a large spill occurs that you are not comfortable cleaning up, evacuate the lab and notify the Tufts emergency services (x66911) immediately. Clean up should only be performed by authorized personnel according to MSDS guidelines. Notify the faculty advisor.

**If at any time you feel a situation is dangerous, do not hesitate to call the safety office (x73246) or the faculty supervisor (x72210, Robert White).**

**Report all accidents (injuries, major spills, fires) to the safety office at x73246 and the faculty supervisor at x72210 (Robert White).**

**For emergencies, call Tufts Emergency Services at x66911.**