

# Titanium Etching

## Standard Operating Procedure

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

Safety Office: Peter Nowak x73246 (Just dial this directly on any campus phone.)  
(617)627-3246 (From off-campus or from a cell phone)

Tufts Emergency Medical Services are at x66911 (on campus) or 617 636 6911 (off campus or cellphone).

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*HF is extremely hazardous to your health. It can kill with limited skin contact on the order of hundreds of milliliters. Exposure to small quantities of HF can cause chronic bone damage.*

### 1. Material Requirements:

**1.1 Equipment:** One polymethylpentene (PMP) graduated cylinder, three polyethylene beakers or polymethylpentene screw cap jars, stainless steel 4" wafer tweezers

*Note: HF attacks glass. You cannot put it in a glass container. Polymethylpentene and polyethylene are fine to use. Polystyrene is not considered compatible with long-term HF exposure.*

**1.2 Chemicals:** HF (hydrofluoric acid), Hydrogen Peroxide

**1.2.1 Hazards associated with chemicals:**

**1.2.1.1** HF: liquid or vapors are extreme health hazards; causes severe burns, which may not be immediately painful or visible. Please use caution; HF is very hazardous, both acutely and long term.

**1.2.1.2** Hydrogen Peroxide: Strong oxidizer. Liquid or vapors are serious health hazards; and cause severe burns.

**1.3 Engineering Controls:** Conduct procedure in ventilated fume hood. Store bottles of chemicals (sealed tightly) in cabinets with secondary containment. Work area should contain an eye wash, safety shower and appropriate fire extinguisher.

**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:

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

#### 2.1 Prepare etchant solution (1:1:20 HF:H<sub>2</sub>O<sub>2</sub>:DI Water)

- 2.1.1 Add 400 mL of DI water to the process container (PMP screw cap jar).
- 2.1.2 Add 20 mL of HF (49% HF – straight out of the bottle) to the process container using the PMP graduated cylinder.
- 2.1.3 Rinse the graduated cylinder once with DI water, dispose of the rinse water in HF contaminated waste (HDPE bottle labeled for HF waste).
- 2.1.4 Add 20 mL of H<sub>2</sub>O<sub>2</sub> (30% hydrogen peroxide – straight out of the bottle) to the process container using the PMP graduated cylinder.
- 2.1.5 Rinse the graduated cylinder twice with DI water, dispose of the rinse water in HF and Hydrogen Peroxide contaminated waste (HDPE bottle labeled for HF and Hydrogen Peroxide waste).

#### 2.2 Titanium Etch

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

- 2.2.1 Before starting the etch, prepare the two rinse containers (PMP screw cap jars or polyethylene beakers) by filling them with DI water.
- 2.2.2 Transfer the sample to be etched into the etching solution. Stainless steel tweezers and PTFE wafer handlers or dipper baskets may be used. After loading the sample into the etch, put the sampling handling tool into the first rinse and leave it there for the duration of the etch.
- 2.2.3 Let the sample etch for the required length of time (depending on feature size). Expect approximately 10 nm/sec etch rate, although this may vary with the size of your sample and amount of material being etched.
- 2.2.4 Transfer the sample to the first rinse. Rinse in DI water for 3 minutes.
- 2.2.5 Transfer the sample to the second rinse. Rinse in DI water for 3 minutes.
- 2.2.6 Make sure that the sample handling tool has also been rinsed for at least 3 minutes in DI water.
- 2.2.7 Remove the sample, and blow dry with the air gun.

#### 2.3 Cleanup

- 2.3.1 If you plan to do another etch in the near future, save the etchant by screwing the cap onto the polymethylpentene jar, labeling the lid with your name, “Titanium Etch – HF, Hydrogen Peroxide,

Water 1:1:20”, and the date, and leaving the jar in the hood. Do not save etchant in a jar for more than 1 week.

- 2.3.2 If you do not plan to do another etch in the near future, use the acid pump to pump the etchant *carefully* into an HF waste container. The waste container should be a HDPE bottle, labeled with “Hydrofluoric Acid”, “Hydrogen Peroxide”, and “water”. The waste can be mixed with other HF contaminated waste, including HF/Nitric, and Buffered HF.
- 2.3.3 Pour the first rinse beaker into the process container, swirl around a bit, and dump or pump that into the HF waste as well.
- 2.3.4 Pour the second rinse beaker into the first rinse beaker, then into the process container, and dump or pump that into the HF waste as well.
- 2.3.5 Finally, rinse all containers one last time with fresh DI water, and pour the rinse water into the dilute acid/base waste (5 gallon HDPE jug).

2.4 Return all labware to its proper location.

2.5 Wipe up any drips in the area with chemical wipes and dispose in acid trash.

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### **3.0 Storage:**

3.1 Hydrofluoric acid should be stored in the acid cabinet.

3.2 Hydrogen peroxide should be store in the base/oxidizer cabinet.

### **4.0 Waste Disposal:**

4.1 Acid waste:

4.1.1 HF/Peroxide wipes are disposed of in the acid trash can.

4.1.2 HF/Peroxide waste is collected in a HDPE bottle, labeled for HF and Hydrogen Peroxide waste and stored in the satellite storage area with secondary containment.

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### **5.0 Accident Procedures:**

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

5.1.1 HF Acid or mixed HF/Hydrogen Peroxide/Water solution:

5.1.1.1 Skin contact: Remove contaminated clothing, rinse affected area with water for 5 minutes. 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 (campus phone) or 617 636 6911 (off campus or cellphone).**

5.1.1.2 Eye contact: Immediately flush with water for 20 minutes while holding the lids open. **Get immediate medical attention. Call Tufts Emergency Medical Services are**

at x66911 (campus phone) or 617 636 6911 (off campus or cellphone).

**5.1.1.3** Ingestion: Do not induce vomiting. **Get immediate medical attention. Call Tufts Emergency Medical Services at x66911 (campus phone) or 617 636 6911 (off campus or cellphone).**

**5.1.1.4** 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 (campus phone) or 617 636 6911 (off campus or cellphone).**

## **5.1.2 Hydrogen Peroxide (no HF)**

**5.1.2.1** Skin contact: Remove contaminated clothing, rinse affected area with water for 10 minutes. **If there is a visible burn, get immediate medical attention. Don't be shy. Call the medical center if you got Piranha on your skin. Tufts Emergency Medical Services are at x66911.**

**5.1.2.2** Eye contact: Immediately flush with water for 20 minutes while holding the lids open. **Get immediate medical attention. Call Tufts Emergency Medical Services at x66911.**

**5.1.2.3** Ingestion: Do not induce vomiting. **Get immediate medical attention. Call Tufts Emergency Medical Services at x66911.**

**5.1.2.4** Inhalation: Remove to fresh air. Resuscitate if necessary. Take care not to inhale any fumes released from the victim's lungs. **Get immediate medical attention. Call Tufts Emergency Medical Services at x66911.**

## **5.2 Spill:**

**5.2.1** 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 (solvent or acid).

**5.2.2** If a large spill occurs that you are not comfortable cleaning up:

**5.2.2.1** 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, Peter Nowak) or the faculty supervisor (x72210, Robert White).

**Report all accidents (injuries, major spills, fires) to the safety office at x73246 (Peter Nowak) and the faculty supervisor at x72210 (Robert White). For emergencies, call Tufts Emergency Services at x66911.**