MEMS Device Release

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.

For more information on glass etching with HF see:

G. Spierings. "Wet Chemical Etching of Silicate Glasses in Hydrofluoric Acid Based Solutions", Journal of Materials Science, **28** (1993), pp. 6261-6273.

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1. Material Requirements:

1.1 Equipment: Two glass beakers (1 chemical, 1 rinse), one polymethylpentene graduated cylinder (if planning to dilute the HF), two polyethylene beakers or polymethylpentene screw cap jars, PTFE tweezers, 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: Acetone, HF (hydrofluoric acid), IPA (isopropanol), Methanol

- **1.2.1** Hazards associated with chemicals:
  - **1.2.1.1** Acetone, IPA, and Methanol: extremely flammable; keep away from sparks and flames, in case of fire, use water spray, alcohol foam, dry chemical or carbon dioxide. Harmful if swallowed or inhaled, causes irritation.
  - **1.2.1.2** 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.

1.3 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 Acetone soak 5 min:

2.1.1 Pour enough acetone into a glass beaker to cover the sample.
2.1.2 Place the sample into the acetone with the tweezers.
2.1.3 Let it soak for 5 minutes.

2.2 Isopropanol soak (IPA) 5 min:

2.2.1 Into a second glass beaker, pour in just enough IPA to cover the sample.
2.2.2 After the 5 minute acetone soak is over, transfer the sample directly to the IPA with tweezers (try not to let it air dry while transferring).
2.2.3 Let it soak for 5 minutes.
2.2.4 While waiting, dump the acetone from the acetone beaker into the waste solvent container.
2.2.5 Spray off the inside of the empty acetone beaker twice with deionized (DI) water from the DI spray bottle and dump it into the solvent waste container.

2.3 Hydrofluorice Acid (HF) etch: variable time

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

2.3.1 If a lower concentration of HF is required, measure the desired amount of DI water into a polyethylene beaker or polymethylpentene jar using the polymethylpentene graduated cylinder. Lower concentrations of HF will etch more slowly (see Spiering's paper listed at the top of this document).
2.3.2 In the polyethylene beaker or polymethylpentene jar, pour in enough HF to cover the samples. *Note: Always Add Acid to Water*. So, if you are diluting the HF, put the water in **first** then add HF to the water.
2.3.3 Transfer the sample from the IPA to the HF carefully but rapidly without allowing the surface to dry. This will help reduce etching problems due to air bubble formation on the sample surface.
2.3.4 Let the sample etch for the required length of time (depending on feature size). Expect approximately 2 microns/minute etch rate, although this will vary with glass composition (again, see Spiering's paper, listed at the top of this document).
2.3.5 While waiting, dump the IPA from the chemical beaker into the waste solvent container.

2.3.6 Spray down the inside of the IPA beaker twice with DI water from the DI squirt bottle and dump it into the solvent waste container.

2.4 DI Water rinse: 10 min:

2.4.1 While the sample is etching, fill a second polyethylene beaker or polymethylpentene jar with enough DI water to cover the sample.
2.4.2 When the HF etch is complete, transfer the sample carefully to the rinse beaker with tweezers. (The MEMS structure will now be released and fragile.)
2.4.3 Leave the tweezer tips (which have HF on them now) to soak in the DI water beaker with the sample.
2.4.4 Let the sample and tweezers soak for 10 mins.
2.4.5 If you plan to do another etch in the near future, save the HF by screwing the cap onto the polymethylpentene jar, labeling the lid with your name, "HF", and the date, and leaving the jar in the hood. Do not save HF in a jar for more than 1 week.
2.4.6 If you do not plan to do another HF etch in the near future, dump the HF *carefully* into the concentrated acid waste container. Use a funnel. Refill the HF container with DI water and dump it into the concentrated acid waste container.
2.4.7 Repeat this rinse two more times, but now dump the rinse water into the large dilute waste water container rather than into the acid waste container.

2.5 IPA rinse 5 min:

2.5.1 Pour just enough IPA into a glass beaker to cover the sample.
2.5.2 Transfer the sample carefully to the IPA beaker.
2.5.3 Let it soak for 5 minutes.
2.5.4 While waiting, dump the water rinse beaker into the large dilute waste water container.
2.5.5 Refill the rinse beaker with DI water and dump it into the large dilute waste water container.

2.6 Methanol rinse:10 min.

Note: We do a methanol rinse last in order to reduce stiction problems. Methanol has the lowest surface tension of the common solvents, and will produce less stiction problems as the parts dry than water. However, large, flexible structures will still have problems with stiction. If stiction is causing problems, there are other options such as critical point drying, vapor-phase release, bumping, self-assembled monolayers (SAM), etc.

2.6.1 Pour just enough methanol into a glass beaker to cover the sample.
2.6.2 Transfer the sample from the IPA to the rinse beaker. Do not allow the sample to air dry during transfer.

2.6.3 Let the sample soak in methanol for 10 minutes.
2.6.4 While waiting, dump the IPA into the waste solvent container.
2.6.5 Spray down the inside of the IPA beaker with DI water twice and dump it into the solvent waste container.

2.7 Air dry:

2.7.1 After the methanol soak is finished, transfer the sample to a chemical wipe (towel) to air dry.
2.7.2 Dump the methanol from the glass beaker into the waste solvent container.
2.7.3 Spray down the methanol beaker with DI water twice and dump it into the solvent waste container.

2.8 Return all labware to its proper location.
2.9 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.

3.0 Storage:

3.1 All three solvents should be stored in an appropriate flammable liquid storage cabinet.
3.2 Hydrofluoric acid should be stored in an appropriate storage cabinet in a tightly capped polyethylene bottle.

4.0 Waste Disposal:

4.1 Solvent waste:
4.1.1 Acetone, isopropanol, and methanol wipes should be disposed of in the solvent trash can.
4.1.2 Waste acetone, isopropanol, and methanol are collected in the solvent waste container and stored in the satellite storage bin with secondary containment.
4.2 Acid waste:
4.2.1 HF wipes are disposed of in the acid trash can.
4.2.2 HF waste is collected in the acid waste container and stored in the satellite storage area with secondary containment.

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 Solvents:
5.1.1.1 Skin contact: Flush with water.
5.1.1.2 Eye contact: Flush with copious amounts of water for 15 minutes.
5.1.1.3 Ingestion: Do not induce vomiting. Give large volumes of water.

5.1.1.4 Inhalation: Remove to fresh air. Resuscitate if necessary.

5.1.2 HF Acid:

5.1.2.1 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.**

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 are at x66911.**

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

5.1.2.4 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 are 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 If it is a **solvent** spill, make sure that there are no ignition sources (open flames, hot filaments… most likely there will not be any), evacuate the room, close the door, and allow the solvent to evaporate. Notify the Tufts emergency services (x66911) immediately. Also notify the lab manager and faculty advisor.

5.2.2.2 If it is a large **HF** spill, 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.