

# GaSb cleaning using HF/Nitric/Acetic Acid (40:18:2) and Nitric Acid/Hydrochloric acid (1:30)

## 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:** Three glass beakers (1 chemical, 2 rinse), one polymethylpentene or polyethylene 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.*

### **2.0 Chemicals:**

- 2.1 Hydrofluoric Acid (HF) (49%):** 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. Compatible with polymethylpentene (PMP), polyethylene (PE), and Teflon (PTFE).
- 2.2 Nitric Acid (70%):** Acute burns to skin and eyes. Severe respiratory irritant. Can get hot when reacting with water, or any base. Compatible with glass, HDPE, polymethylpentene (PMP), polyethylene (PE), and Teflon (PTFE).
- 2.3 Glacial Acetic Acid (>98%):** Acute burns to skin and eyes. Severe respiratory irritant. Can get very hot when reacting with water, or any base. Compatible with glass, HDPE, polymethylpentene (PMP), polyethylene (PE), and Teflon (PTFE).
- 2.4 Hydrochloric Acid (37%):** Corrosive. Explodes on contact with ethyl hydroperoxide.
- 2.5 Engineering Controls:** Conduct procedure in ventilated fume hood. Store bottles of chemicals (sealed tightly) in cabinets with secondary containment.
- 2.6 Work area** should contain an eye wash, safety shower and appropriate fire extinguisher.
- 2.7 Personal Protective Equipment:** Trionic gloves on top of nitrile gloves, apron, goggles, and face-shield. Never work with HF without all this equipment.

### 3.0 Procedure:

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

#### 3.1 Acetic acid, Nitric acid, Hydrofluoric acid soak (40:18:2) 40 sec.

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

**3.1.1** Place some fab wipes in the hood. Get three polyethylene beakers or polymethylpentene jars that will fit your samples for processing (you should find containers labeled "HF", "HNA", "HF Rinse" and "HNA Rinse" on the shelves... any of these are acceptable). Put them on the fab wipes in the hood. Try to use the smallest containers that will work for your sample size to reduce waste.

**3.1.2** Use graduated cylinders to measure the following chemicals into the first container.

**3.1.2.1** *Use a glass graduated cylinder for the acetic acid and nitric acid. Use a polyethylene or polymethylpentene graduated cylinder for the HF.*

**3.1.2.2** Add the acetic acid first, then the nitric acid, and finally the HF. Use the following ratio:

40 mL Glacial Acetic Acid (>98%)

18 mL Nitric Acid (70%)

2 mL Hydrofluoric Acid (49%)

**3.1.3** Cap the bottles and put them away.

**3.1.4** Fill the second and third process containers (also polyethylene or polymethylpentene containers) with deionized water.

**3.1.5** Transfer the sample into the HNA mixture carefully with tweezers or PTFE wafer tools.

**3.1.6** Let the sample etch for 1 min.

#### 3.2 DI Water rinse: 2 x 5 min:

**3.2.1** When the time is up, transfer the sample carefully to the first rinse beaker with tweezers or PTFE wafer tools.

**3.2.2** Leave the tweezers/tools (which have HNA solution on them now) to soak in the DI water with the sample.

**3.2.3** Let the sample and tweezers soak for 5 mins.

**3.2.4** Transfer the sample and tools to the second rinse beaker, and soak for 5 minutes.

#### 3.3 Hydrochloric acid, Nitric Acid soak (30:1) 1 min:

**3.3.1** Place some fab wipes in the hood. Get three glass beakers that will fit your samples for processing (you should find containers labeled "HCl", or "HCl/Nitric Clean" on the shelves... any of these are acceptable). Put them on the fab wipes in the hood. Try to use the smallest containers that will work for your sample size to reduce waste.

**3.3.2** Use a glass graduated cylinder to measure the following chemicals into the first container.

**3.3.2.1** Add the hydrochloric acid first, then the nitric acid. Use the following ratio:

30 mL Hydrochloric Acid (37%)

1 mL Nitric Acid (70%)

**3.3.3** Cap the bottles and put them away.

**3.3.4** Fill the second and third process containers (also glass beakers) with deionized water.

**3.3.5** Put your sample in the cleaning solution and let it soak for 1 minute.

**3.4 DI Water rinse: 2 x 5 min:**

**3.4.1** When the time is up, transfer the sample carefully to the first rinse beaker with tweezers or PTFE wafer tools.

**3.4.2** Leave the tweezers/tools (which have HNA solution on them now) to soak in the DI water with the sample.

**3.4.3** Let the sample and tweezers soak for 5 mins.

**3.4.4** Transfer the sample and tools to the second rinse beaker, and soak for 5 minutes.

**3.4.5** Dry sample with N<sub>2</sub> gun.

**3.4.6** Return all labware to its proper location.

**3.4.7** 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.

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**4.0 Cleanup**

**4.1 HNA (HF/Nitric/Acetic)**

**4.1.1** If you do plan to do another HNA etch in the near future (next 2 days), cap the etchant container or cover it with aluminum foil, label it with "HF/Nitric/Acetic", your name, and the date, and place it in the back of the hood.

**4.1.2** If you do not plan to do another HNA etch in the near future, dump the used HNA solution into the HF waste bottle. Refill the HF container with DI water and again dump it into the HF waste container. The waste container should be an HDPE bottle. Use a plastic funnel.

**4.1.3** Rinse the HNA container with DI water again and dump it into the HF waste bottle.

**4.1.4** Dump the first water rinse container into the HF waste bottle.

**4.1.5** Dump the water from the second rinse beaker into the dilute acid/base water waste container. (5 gallon jug)

**4.1.6** Rinse all the containers one more time with DI water and dump them into the dilute acid/base water waste container. (5 gallon jug)

**4.1.7** Return all labware to its proper location. The beakers can drip dry on fab wipes in the hood or on the storage shelving.

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

**4.2 HCl/Nitric Acid**

**4.2.1** Rinse the HCl/H<sub>2</sub>SO<sub>4</sub> container with DI water again and dump it into the HCl acid waste bottle.

**4.2.2** Dump the first water rinse container into the HCl acid waste bottle.

**4.2.3** Dump the water from the second rinse beaker into the dilute acid/base water waste container. (5 gallon jug)

**4.2.4** Rinse all the containers one more time with DI water and dump them into the dilute acid/base water waste container. (5 gallon jug)

**4.2.5** Return all labware to its proper location. The beakers can drip dry on fab wipes in the hood or on the storage shelving.

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

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**5.0 Storage:**

**5.1** Hydrofluoric acid, nitric acid, hydrochloric, and glacial acetic acid should all be stored in the acid cabinet in tightly capped polyethylene bottles.

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## **6.0 Waste Disposal:**

### **6.1 Acid waste:**

- 6.1.1** HNA and HCl/Nitric contaminated fab wipes are disposed of in the acid trash can.
- 6.1.2** HNA waste is collected in the HF waste container (HDPE bottle) and stored in the satellite storage area with secondary containment.
- 6.1.3** HCl/Nitric waste is collected in the HCl waste container (HDPE bottle) and stored in the satellite storage area with secondary containment. Do NOT mix HCl waste and sulfuric acid waste.
- 6.1.4** Dilute water waste from second and third rinses goes into the dilute water waste 5 gallon HDPE tank.

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## **7.0 Accident Procedures:**

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

### **7.1.1** Solvents:

- 7.1.1.1** Skin contact: Flush with water.
- 7.1.1.2** Eye contact: Flush with copious amounts of water for 15 minutes.
- 7.1.1.3** Ingestion: Do not induce vomiting. Give large volumes of water.
- 7.1.1.4** Inhalation: Remove to fresh air. Resuscitate if necessary.

### **7.1.2** HF Acid:

- 7.1.2.1** 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.**
- 7.1.2.2** 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.**
- 7.1.2.3** Ingestion: Do not induce vomiting. **Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.**
- 7.1.2.4** 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.**

### **7.2** Spill:

- 7.2.1** 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).
- 7.2.2** If a large spill occurs that you are not comfortable cleaning up:
  - 7.2.2.1** 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.
  - 7.2.2.2** 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.**