

Aluminum Nitride Etch

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.

Revised: October 19, 2017

Saravanan, S., et al. "Surface micromachining process for the integration of AlN piezoelectric microstructures." in: SAFE 2004, 7th Annual Workshop on Semiconductor Advances for Future Electronics, 25-26 Nov 2004, Veldhoven, the Netherlands (pp. 676-681).

Warning: Avoid contact with skin and eyes. Do not ingest. Do not breathe the vapors. Vapors are moderately irritating to the mucous membranes and respiratory track and can cause excessive tearing. Work carefully in the hood with goggles, apron, face shield and trionic gloves.

1. Material Requirements:

- 1.1 Equipment: One hot plate, thermometer, one glass crystallizing dish (125 mm diameter, 65 mm high), two 1000 mL glass beakers (for rinse), 150 mm diameter watch glass, stainless steel tweezers, PTFE (Teflon) wafer holders or sample holders.
- 1.2 Chemicals: Phosphoric acid 85%
 - 1.2.1 Hazards associated with chemicals:
 - 1.2.1.1 This solution is seriously corrosive to the eyes, skin and mucous membranes and causes irritation and burns. Vapors **severely** irritate the respiratory track.
 - 1.2.1.2 Do not mix with strong oxidants or organic materials.
- 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 and safety shower.
- 1.4 Personal Protective Equipment: Trionic gloves on top of nitrile gloves, apron, goggles, and face-shield.

2.0 Procedure:

Complete all processes in the fume hood.

2.1 Phosphoric acid (85%) : 20nm/min at 120°C (Saravanan)

- 2.1.1 Get two water rinse beakers which will fit your samples (A 1000 mL beaker works for a single 4" wafer). **Do this first.** If something goes wrong, you want the water available to quench the reaction.
- 2.1.2 Place the rinse beakers on a few fab wipes in the hood, and fill them with deionized water such that the water level will cover the entire sample.
- 2.1.3 Bring and place a hotplate in the hood. Do not turn it on yet.
- 2.1.4 Place a couple of fab wipes in a pile in the hood. Get a glass 125 mm crystallizing dish that will fit your samples for processing (you should find one labeled "Aluminum Nitride Etchant" on the shelves). Put it on the fab wipes in the hood.
- 2.1.5 Carefully pour Phosphoric acid (85% concentration, which should be what is stocked in the stock bottle) into the dish such that it is a little over half full.

- 2.1.6 **This step should be done very carefully** : move the dish with Phosphoric acid onto the hot plate prepared in 2.1.3.
- 2.1.7 Cover the dish using a 150 mm diameter glass concave dish (“watchglass”) convex side down, to condense the vapor, returning it to the container. This prevents severe concentration change caused by evaporation during heating of the solution.
- 2.1.8 Set the temperature of the hotplate. The target temperature of the Phosphoric acid solution is 120°C. The temperature of the bath will be at least 10°C lower than the temperature of the hotplate. Measure the solution temperature using thermometer and adjust hotplate temperature as needed to stabilized at 120°C.
- 2.1.9 Calculate the etch time for your sample. You will need to know the thickness of your aluminum nitride layer. At 120°C, the AlN will etch at a rate of approximately 20nm/min. Etch rate depends on the temperature of the solution.
- 2.1.10 Put your wafer into the heated Phosphoric acid for the approximate amount of time calculated in the previous step. Stirring the solution is not recommended for your own safety.

DI water rinse: 10 mins

- 2.1.11 When the etch is complete, transfer the sample carefully to the first DI water rinse beaker. You may choose to use PTFE wafer holder to hold the wafer or sample once in the rinse.
- 2.1.12 If you used tweezers to move the sample, make sure you leave them in the rinse beaker to rinse it as well.
- 2.1.13 Let the sample and tools soak in DI water for 10 mins.
- 2.1.14 Transfer the sample to the second DI rinse beaker, and rinse for another 10 mins.

2.2 Sample dry :

- 2.2.1 After the water rinse is finished, remove your samples, and blow and dry with the air gun.
- 2.2.2 After getting most of the water off, you can dry the samples more in an oven at 120°C or on a hotplate at 150°C.
- 2.2.3 Inspect wafer for traces un-etched aluminum nitride. If features are small, use an optical microscope. If more etch time is required, place wafer back into the Petri dish with the etchant for another 30 seconds. Repeat rinse and drying procedure.

2.3 Cleanup

- 2.3.1 Turn off the hotplate and cool down the heated Phosphoric acid solution below 30°C.
- 2.3.2 The etchant may be used for multiple etches. For temporary storage (<1 day), cover the dish with Parafilm in the back of the hood. Make sure the dish is clearly labeled “Phosphoric acid” and has your name and the date on it.
- 2.3.3 When you are finished using the etchant, check the temperature of Phosphoric acid solution. If the temperature is below 30 °C, dispose of it in a HDPE or glass bottle, label “Phosphoric acid” with the red hazardous waste tag. The phosphoric acid can be mixed with other strong acids including nitric acid, acetic acid, HF, HCl, HNA, and Cr etchant. Keep the bottle in the satellite accumulation area (under the hood). If a waste bottle already exists, use that one, otherwise start a new one.
- 2.3.4 Rinse the dish and glass concave dish once with DI water, and dump it into the phosphoric acid waste bottle.
- 2.3.5 Dump the first DI rinse beaker into the process dish, and then into the phosphoric acid waste bottle.
- 2.3.6 Dump the second DI rinse beaker into the first rinse beaker, then into the process container, and finally into the 55 gallon HDPE “Dilute Acid Waste” drum.

- 2.3.7 Rinse all glassware one final time and dump into the 55 gallon HDPE “Dilute Acid Waste” drum.
- 2.3.8 Return all labware to its proper location. The dish, watch glass and beakers can drip dry on lab wipes in the hood or on the shelves.

3.0 Storage:

- 3.1 Store phosphoric acid in the “Acid” cabinet.

4.0 Waste Disposal:

- 4.1 Phosphoric acid waste :
 - 4.1.1 Solid waste should go in the acid waste bin
 - 4.1.2 Liquid waste should go in the mixed acid waste bottle. This container can be glass or HDPE. The acid waste can be mixed with nitric acid, acetic acid, HF, HCl, nitric acid, acetic acid, HNA, and Cr etchant. Add “Phosphoric acid” to the hazardous waste tag.

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 Phosphoric acid :
 - 5.1.1.1 Skin contact : immediately flush with water. Remove contaminated clothing, wash skin with soap and water. **If there is any irritation, get medical attention. Don’t be shy. Call Tufts Emergency Medical Services are at x66911.**
 - 5.1.1.2 Eye contact : Immediately flush with water for at least 15 minutes while lifting upper and lower eyelids occasionally. Get immediate medical attention. **Call Tufts Emergency Medical Services are at x66911.**
 - 5.1.1.3 Ingestion : Do not induce vomiting. **Call Tufts Emergency Medical Services are at x66911.**
 - 5.1.1.4 Inhalation : remove to fresh air. Resuscitate if necessary. Take care not to inhale any fumes released from the victim’s lungs. **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.
 - 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 at 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.