

GaAs wafer prep(Piranha followed by Hydrochloric)

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: July 7, 2009

Warning: Piranha is highly reactive, hot, and attacks organic materials. It will burn you badly if it gets on your skin. Under no circumstances let it get in your eyes. Do not breath the vapors. Work carefully in the hood with full personal protective equipment.

1. Material Requirements:

- 1.1 **Equipment:** Three glass beakers (2 chemical), stainless steel tweezers, PTFE (Teflon) wafer holders or sample holders.
- 1.2 **Chemicals:** Sulfuric Acid (H_2SO_4 96%), Hydrogen Peroxide (H_2O_2 30%), Hydrochloric Acid (HCl), Deionized Water (H_2O). Note: These percentages are the dilutions as they arrive in the bottles from the chemical supplier. Just use the solution straight out of the bottle.
 - 1.2.1 **Hazards associated with chemicals:**
 - 1.2.1.1 Sulfuric Acid: Liquid or vapors are serious health hazards; and cause severe burns. Sulfuric acid is much more viscous than water, be prepared for this when you pour it.
 - 1.2.1.2 Hydrogen Peroxide: Liquid or vapors are serious health hazards; and cause severe burns.
 - 1.2.1.3 Hydrochloric Acid: Corrosive. Explodes on contact with ethyl hydroperoxide. Reacts with water or steam to produce toxic and corrosive fumes.
- 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 Sulfuric Acid and Hydrogen peroxide dilute clean (1:8:500 96% H_2SO_4 : 30% H_2O_2 : H_2O): 30 sec

- 2.1.1 Place 5 fab wipes in a pile in the hood. Get two beakers that will fit your samples for processing. Put it on the fab wipes in the hood. A 1000 mL beaker works for a single 4" wafer.
- 2.1.2 Put your samples in the empty beaker, and see how much solution you will need to cover them. If the samples come within 10% of the top of the container, get a larger container.
- 2.1.3 Leave your samples in the beaker, and pour in deionized H_2O .
- 2.1.4 Pour in the Hydrogen peroxide.
- 2.1.5 Lastly pour in the Sulfuric Acid. This solution is a dilute "piranha" clean.
- 2.1.6 Allow the solution to sit for 30 sec.

2.2 DI Water rinse: 5 min:

- 2.2.1 Fill second and third glass beakers with enough DI water to cover the sample.
- 2.2.2 When the piranha clean is complete, transfer the sample carefully to the rinse beaker with tweezers.
- 2.2.3 Leave the tweezer tips (which have piranha on them now) to soak in the DI water beaker with the sample.
- 2.2.4 Let the sample and tweezers soak for 5 mins.
- 2.2.5 Transfer to the second DI water rinse beaker. Soak for 5 mins.
- 2.2.6 If you do not plan to do another piranha clean in the near future, dump the piranha *carefully* into the piranha acid waste container (a *glass* bottle). Use a glass funnel. Refill the piranha container with DI water and dump it into the piranha acid waste container.
- 2.2.7 Repeat this rinse two more times, but now dump the rinse water into the 5 gallon HDPE dilute acid/base waste water container rather than into the piranha waste container.
- 2.2.8 The first rinse beaker should be dumped into the piranha waste bottle.
- 2.2.9 The second rinse beaker should be dumped into the 5 gallon HDPE dilute acid/base waste water container.
- 2.2.10 Both rinse beakers should be rinsed once more with DI water and dumped into the 5 gallon HDPE dilute acid/base waste water container.

2.3 Hydrochloric dilute clean (1:1 HCl and H₂O): 2 min:

- 2.3.1 When the DI water rinse is complete, transfer the sample carefully to the hydrochloric acid clean beaker.
- 2.3.2 Pour in the deionized water to cover half of your samples.
- 2.3.3 Pour in the hydrochloric acid.
- 2.3.4 Let the sample and tools soak in clean for 2 mins.

2.4 DI Water rinse: 5 min:

- 2.4.1 Fill second and third glass beakers with enough DI water to cover the sample.
- 2.4.2 When the HCl clean is complete, transfer the sample carefully to the rinse beaker with tweezers.
- 2.4.3 Leave the tweezer tips (which have HCl on them now) to soak in the DI water beaker with the sample.
- 2.4.4 Let the sample and tweezers soak for 5 mins.
- 2.4.5 Transfer to the second DI water rinse beaker. Soak for 5 mins.
- 2.4.6 If you do not plan to do another HCl clean in the near future, dump the HCl *carefully* into the HCl acid waste container. Use a funnel. Refill the HCl container with DI water and dump it into the concentrated HCl 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.
- 2.4.8 The first rinse beaker should be dumped into the HCl waste bottle.
- 2.4.9 The second rinse beaker should be dumped into the 5 gallon HDPE dilute acid/base waste water container.
- 2.4.10 Both rinse beakers should be rinsed once more with DI water and dumped into the 5 gallon HDPE dilute acid/base waste water container.

2.5 Sample dry:

- 2.5.1 After the HCl clean is finished, remove your samples and blow them dry with the N₂ gun.

2.6 Cleanup

- 2.6.1 Dispose of the waste and rinse water as described above.

- 2.6.2 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.
- 2.6.3 Wipe up any drips in the area with chemical wipes and dispose in the acid trash

2.7 Storage

- 2.7.1 Sulfuric acid should be stored in the acid cabinet.
- 2.7.2 Hydrochloric acid should be stored in the acid cabinet.
- 2.7.3 Hydrogen peroxide should be stored in the base/oxidizer cabinet.

3.0 Waste Disposal:

3.1 Piranha waste:

- 3.1.1 Solid waste should go in the acid waste bin.
- 3.1.2 Liquid waste should go in the "Piranha" (that is, Sulfuric Acid and Hydrogen Peroxide) *glass waste bottle* in the hazardous waste accumulation area.

3.2 Hydrochloric Acid waste:

- 3.2.1 Solid waste should go in the acid waste bin.
- 3.2.2 Liquid waste should go in the Hydrochloric Acid waste. This should be an HDPE or glass waste bottle in the hazardous waste accumulation area. Do NOT mix HCl waste and sulfuric acid waste.

4.0 Accident Procedures:

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

4.1.1 Piranha mixture, straight sulfuric acid, straight hydrochloric acid, or straight hydrogen peroxide:

- 4.1.1.1 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.**
- 4.1.1.2 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.**
- 4.1.1.3 Ingestion: Do not induce vomiting. **Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.**
- 4.1.1.4 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 are at x66911.**

4.2 Spill:

- 4.2.1 If a small, contained spill occurs, such as inside the hood, wipe it up with chemical wipes and dispose of in the acid trash container.
- 4.2.2 If a large spill occurs that you are not comfortable cleaning up:
 - 4.2.2.1 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.