

Silver 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: May 29, 2009

Warning: Ingestion of Nitric Acid may cause irritation to mouth and stomach. Prolonged exposure may cause skin burns.

1. Material Requirements:

1.1 Equipment: Three glass Petri dishes (the Cr/Au rinse dishes can be used), stainless steel tweezers, PTFE (Teflon) wafer holders or sample holders.

1.2 Chemicals: Nitric Acid

1.2.1 Hazards associated with chemicals:

1.2.1.1 Oxidizer. Decomposes in storage under conditions of excessive heat causing release of nitrogen oxides and oxygen that supports combustion. Decomposition could form a high temperature melt.

1.2.1.2 Inhalation: Vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract.

1.2.1.3 Do not mix with organic solvents or strong reducing agents

1.3 Engineering Controls: Store bottles of chemicals (sealed tightly) in cabinets with secondary containment. Work area should contain an eye wash and safety shower. All processing should be performed in the chemistry fume hood.

1.4 Personal Protective Equipment: Trionic gloves on top of nitrile gloves, apron, face shield, and safety glasses.

2.0 Procedure:

Complete all processes in the fume hood

2.1 Silver Etch

2.1.1 Get two water rinse beakers or petri dishes which will fit your samples. **Do this first.** If something goes wrong, you want the water available to quench the reaction.

2.1.2 Fill the rinse beakers with deionized water such that the water level will cover the entire sample.

2.1.3 Place a couple of fab wipes in a pile in the hood. Get a glass Petri dish that will fit your samples for processing (you should find one labeled "Silver Etch" on the shelves). Put it on the fab wipes in the hood.

- 2.1.4 Create a 1:1 solution of Nitric Acid by adding 60mL of concentrated nitric acid to 60mL of deionized water in the petri dish. Nitric acid is much denser than water, making the resulting mixture much more concentrated at the bottom of the petri dish. Stir the solution to dissolve the acid in the water.
- 2.1.5 Calculate the etch time for your sample. You will need to know the thickness of your copper layer. At 20°C, the silver will etch at a rate of approximately 400 nm/min. This is neither exact nor constant! Testing it for yourself is a good idea. The etch rate will slow down dramatically as the concentration of AgNO₃ in solution increases.
- 2.1.6 Put your wafer into the etchant and soak for the appropriate amount of time calculated in the previous step. Careful swirling of the Petri dish will improve uniformity. Be sure to keep the petri dish and wafer level, or the concentration gradient in the solution will cause uneven etching.

2.2 DI water rinse:10 min:

- 2.2.1 When the etch is complete, transfer the sample carefully to the first DI water rinse beaker
- 2.2.2 If you used tweezers to move the sample, make sure you leave them in the rinse beaker to rinse as well.
- 2.2.3 Let the sample and tools soak in DI water for 5 mins.
- 2.2.4 Transfer the sample to the second DI rinse beaker, and rinse for another 5 mins.

2.3 Sample dry:

- 2.3.1 After the water rinse is finished, remove your samples and blow them dry with the gun.
- 2.3.2 Inspect wafer for traces un-etched silver. 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 10-20 seconds while swirling. If un-etched silver still remains, it most likely because the etchant has reached capacity. Add 10mL more of nitric acid to the solution to increase the etch capacity. Alternatively, you may heat the etchant to 50°C. If these two measures fail to complete the etch, remake the etch solution. Do not add more nitric acid or increase the temperature further. Repeat rinse and drying procedure.

2.4 Clean-up:

- 2.4.1 The etchant may be used for multiple etches. However, the etch capacity of the etchant is limited. For temporary storage, place the top of the Petri dish over the etchant and store on fab wipes in the back of the hood. Make sure the dish is clearly labeled "Silver Etchant"
- 2.4.2 When you finish using the etchant, dispose of it in a HDPE or glass bottle, label "Nitric Acid" with the red hazardous waste tag. The waste is compatible with both the Chromium Etchant and

Aluminum Etch waste bottles. 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.

- 2.4.3 Rinse the Petri dish once with DI water, and dump it into the waste bottle.
- 2.4.4 Dump the first DI rinse beaker into the Ag Etch waste bottle.
- 2.4.5 Dump the second DI rinse beaker into the Ag Etch waste bottle
- 2.4.6 Rinse all three containers a second time with DI water, again dumping them into the Ag waste bottle.
 - 2.4.6.1 Do not put any nitric acid waste into the 5 gallon dilute acid/waste container.
- 2.4.7 Return all lab ware to its proper location. The Petri dish and the beaker can drip dry on fab wipes in the hood or on the shelves.

3.0 Storage:

- 3.1 Keep container tightly closed. Store on corrosion-proof area.

4.0 Waste Disposal:

- 4.1 Chemical: Nitric Acid
 - 4.1.1 Solid waste for chemicals should go in the acid waste bin.
 - 4.1.2 Liquid waste for chemicals should go in the Ag/Cr Etch waste bottle. This container can be glass or HDPE.

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 Chemical: Nitric Acid
 - 5.1.1.1 CALL A PHYSICIAN. If swallowed do not induce vomiting; If conscious, give water, milk or milk of magnesia.
 - 5.1.1.2 If inhaled remove to fresh air. If not breathing give artificial respiration. If breathing is difficult give oxygen.
 - 5.1.1.3 In case of contact immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before re-use.

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 If it is a **chemical** spill, do this. Notify the Tufts emergency services (x66911) immediately. Also 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.