Copper Etch #2: 1:1:18 Acetic Acid:Hydrogen Peroxide:Water

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 8, 20155

Warning: Acetic acid and hydrogen peroxide will both cause burns and irritation if they contact the skin, eyes, or mucous membranes. Wear personal protective equipment. Avoid exposure.

Hydrogen Peroxide – the waste is an explosive hazard, all waste containing peroxide must use a vented cap. Do not mix with solvents, energetic chemical reactions (explosions!) may result. Ensure you work area is free of solvent contamination and do not use a waste container that includes solvent waste.

Notes: This etch is intended as an isotropic wet etch for copper. This etch *does not* attack nickel, gold, chromium, glass, or silicon. Copper etch rates are on the order of 200 nm/min (you should confirm the etch rate with your own measurements). The etch *does* slowly etch titanium. Other materials have not been tested to date at Tufts. To date it is not known how long a photoresist mask will stand up to the etch.

1. Material Requirements:

1.1 Equipment: One glass Petri dish, two 1000 mL glass beakers (for rinse), graduated cylinder (either glass or polyethylene/polypropylene), 100 mL beaker (for chemical transfer), stainless steel tweezers, PTFE (Teflon) wafer holders or sample holders.

1.2 Chemicals: glacial acetic acid, hydrogen peroxide

1.2.1 Hazards associated with chemicals:

1.2.1.1 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).

1.2.1.2 Hyrdogen Peroxide: Liquid or vapors are serious health hazards; and cause severe burns.

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: Nitrile gloves and safety glasses.

2.0 Procedure:

Complete all processes in the fume hood

2.1 Prepare rinse beakers

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 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 "Acetic:Peroxide:Water" on the shelves). Put it on the fab wipes in the hood.

2.2 Mix the Etchant: (1:1:18 by volume glacial acetic acid : 30% hydrogen peroxide: deionized water)

2.2.1 First, get >180 mL of water from the DI water source in the small beaker. Measure 180 mL accurately using the graduate cylinder, and pour into the process petri dish. Use multiple steps if necessary (if you are using a 100 mL beaker or 100 mL graduate cylinder, for example).

2.2.2 Next, pour more than 10 mL of glacial acetic acid from the stock bottle into the small 100 mL beaker. Use the graduated cylinder to accurately measure 10 mL of glacial acetic acid from the small beaker, and add this to the process dish.

2.2.3 Dump the remaining glacial acetic acid from the small beaker into an HDPE waste bottle labeled for acetic acid, hydrogen peroxide, and water. Rinse the beaker with DI water twice and dump into the waste bottle.

2.2.4 Finally, pour more than 10 mL of hydrogen peroxide (30%) from the stock bottle into the small 100 mL beaker. Use the graduated cylinder to accurately measure 10 mL of hydrogen peroxide from the small beaker, and add this to the process dish.

2.2.5 Dump the remaining hydrogen peroxide from the small beaker into an HDPE waste bottle labeled for acetic acid, hydrogen peroxide, and water. Rinse the beaker with DI water twice and dump into the waste bottle.

2.2.6 Return the small beaker to the shelves.

2.3 Perform the etch

2.3.1 Place the sample to be etched into the petri dish. Expect approximately 200 nm/min copper etch rate (this should be confirmed if critical!).

2.3.2 Allow the sample to etch for the desired time.

2.3.3 *Note:* You may notice the evolution of bubbles at the etch surface as the etch progresses. These bubbles could cause micro-masking of the etch. You may find it helpful to gently agitate your sample to detach the bubbles periodically.

2.4 DI water rinse:6 min:

2.4.1 When the etch is complete, transfer the sample carefully to the first DI water rinse beaker

2.4.2 If you used tweezers to move the sample, make sure you leave them in the rinse beaker to rinse as well.

2.4.3 Let the sample and tools soak in DI water for 3 mins.

2.4.4 Transfer the sample to the second DI rinse beaker, and rinse for another 3 mins.

2.5 Sample dry:

2.5.1 After the water rinse is finished, remove your samples and blow them dry with the gun.

2.5.2 Inspect wafer for traces of un-etched copper. If features are small, use an optical microscope. If more etch time is required, place wafer back into the Petri dish with the etchant as required. Repeat rinse and drying procedure.

2.6 Clean-up:

2.6.1 When you finish using the etchant, dispose of it in a HDPE or glass bottle, label "Acetic Acid, Hydrogen Peroxide" with the red hazardous waste tag. 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.

2.6.2 Rinse the Petri dish once with DI water, and dump it into the Cu Etch waste bottle.

2.6.3 Dump the first DI rinse beaker into the Cu Etch waste bottle.

2.6.4 Dump the second DI rinse beaker into the 5 gallon HDPE "Dilute Acid Waste" container.

2.6.5 Rinse all three containers a second time with with DI water. This time, dump them into the 5 gallon HDPE "Dilute Acid Waste" container.

2.6.6 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 in corrosion-proof area.

4.0 Waste Disposal:

4.1 Chemical: Acetic Acid and Hydrogen Peroxide mixture

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 Cu Etch waste bottle. This container can be glass or HDPE. This waste container must have a vented cap. Ensure this waste is not exposed to any solvents, an explosion may result.

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: Glacial Acetic Acid, Hydrogen Peroxide, or mixture

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.