Aqua Regia 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

Aqua Regia is a mixture of hydrochloric acid, nitric acid and water. This particular formulation is dilute aqua regia mixed as 2 parts water:3 parts 37% HCl:1 part 70% HNO_3. Those percentages (37%, 70%) are the concentration of the HCl and HNO_3 stocked by the lab – so the stock bottles have 37% HCl and 70% HNO_3 in them – you should mix the solution using 2:3:1 of the constituents as poured out of the stock bottles.

The mixture is notable in its ability to etch noble metals. This etch attacks molybdenum (650 nm/min), palladium (400 nm/min), gold (700 nm/min), copper (600 nm/min), nickel (100 nm/min), and aluminum (600 nm/min). It can be masked with standard photoresists. It does not attack silicon or glass. The solution may also etch platinum at elevated temperatures, although that process is not covered by this SOP.

Williams, Kirt R., Kishan Gupta, and Matthew Wasilik. "Etch rates for micromachining processing-Part II." *Microelectromechanical Systems, Journal of* 12.6 (2003): 761-778.

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 glass Petri dish, two 250 mL glass beakers, 100 mL polypropylene graduate cylinder for acid, two 1000 mL glass beakers (for rinse), stainless steel tweezers, PTFE (Teflon) wafer holders or sample holders.

1.2 Chemicals: DI water, Hydrochloric Acid, Nitric Acid.

Aqua Regia is a mixture of water, Hydrochloric acid, and Nitric acid (mixed at 2:3:1 by volume in that order). The ratio is the actual volume you need to mix using the acids from the bottles stored in the cleanroom. The lab stocks Hydrochloric acid at 37%, and Nitric acid at 70%.

1.2.1 Hazards associated with chemicals:

- **1.2.1.1** This solution is **extremely corrosive to the eyes, skin and mucous membranes** and causes serious irritation and burns.
- **1.2.1.2** Vapors severely irritate the respiratory track.
- **1.2.1.3** Do not mix with strong oxidants or organic materials.
- **1.2.1.4** Do not mix with alkaline solutions.

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 DI water, Hydrochloric acid, and Nitric acid mixed at 2:3:1

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 Stand 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 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 "Aqua Regia (DI water, Hydrochloric acid, and Nitric acid mixed at 2:3:1)" on the shelves. Put it on the fab wipes in the hood.

2.1.4 Get two 250 mL glass beakers labeled for nitric acid and hydrochloric acid.

2.1.5 Pour approximately 100 mL of 37% hydrocholoric acid into the hydrocholoric acid beaker.

2.1.6 Pour approximately 50 mL of 70% nitric acid into the nitric acid beaker.

Note: Mixing acids & water is an exothermic reaction therefore always slowly add acid to water. The large volume of water absorbs the heat and prevents boiling/splashing. For this reason we will start with water, then add the more dilute HCl, then finally add the more concentrated HNO_3.

2.1.7 Measure out 50 mL of DI water from the DI water supply using the 100 mL HDPE graduated cylinder labeled for "DI water".

2.1.8 Pour the water into your process container (glass petri dish).

2.1.9 Measure out 75 mL of 37% hydrochloric acid by pouring from the 250 mL beaker into the HDPE graduated cylinder labeled "acid".

2.1.10 Pour the 75 mL of hydrochloric acid slowly into your process container.

2.1.11 Measure out 25 mL of nitric acid by pouring from the 250 mL beaker into the HDPE graduate cylinder labeled "acid".

2.1.12 Pour the 25 mL of nitric acid slowly into your process container.

2.1.13 The solutions will self heat to approximately 30 ºC.

2.1.14 Wait for 30 min to let the solution cool down near room temperature.

2.1.15 Put your wafer into the Aqua Regia. Stirring the solution is **not** recommended for your own safety.

Expected etch rates (users should verify by experiment):

molybdenum (650 nm/min)	palladium (400 nm/min)
gold (700 nm/min)	copper (600 nm/min)
nickel (100 nm/min)	aluminum (600 nm/min)

Does not etch: glass, silicon, SiO_2, Si_3N_4, Ti, Ta, Cr.

DI wafer rinse: 10 mins

2.1.16 When the etching 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.

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

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

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

2.2 Sample dry :

2.2.1 After the water rinse is finished, remove your samples and blow them 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 ºC or on a hotplate at 150 ºC. Note that remaining metal patterns in the wafer may be oxidized at the elevated temperature, and you will need to dip into BOE to eliminate metal oxide.

2.2.3 Inspect wafer for traces un-etched metal. 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 The etchant should **not** be used for multiple etches. Williams, *et al* report that the HCl evaporates off, so the solution should be prepared immediately before etching.

2.3.2 Dispose of the etchant in mixed acid waste HDPE bottle. Make sure the label includes "Hydrochloric acid" and "Nitric acid" on the hazardous waste tag. The acid can be mixed with other strong acids including acetic acid, HF, 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.

2.3.3 Pour any excess HCl and HNO_3 from the 250 mL beakers into the mixed acid waste bottle as well.

2.3.4 Rinse each of the 250 mL beakers once with water and dump that into the mixed acid waste.

2.3.5 Rinse the acid graduated cylinder twice with water, disposing of the rinse water in the mixed acid waste bottle.

2.3.6 Rinse the Petri dish once with DI water, and dump it into the acid waste bottle.

2.3.7 Dump the first DI rinse beaker into the acid waste bottle.

2.3.8 Dump the second DI rinse beaker into the 55 gallon HDPE "Dilute Acid Waste" container.

2.3.9 Rinse all glassware a second time, dumping the water into the 55 gallon HDPE "Dilute Acid Waste" container.

2.3.10 Return all labware to its proper location. The Petri dish and beakers can drip dry on fab wipes in the hood or on the shelves.

3.0 Storage:

3.1 Store Hydrochloric acid and Nitric acid in the "Acid" cabinet.

4.0 Waste Disposal:

4.1 Hydrochloric acid, and Nitric acid waste :

4.1.1 Solid waste should go in the acid waste bin

4.1.2 Liquid waste should go in mixed acid waste bottle. This container can be glass or HDPE. The acid waste can be mixed with HCl, HNO3, acetic acid, HF, HNA, and Cr etchant. Label "Hydrochloric acid and Nitric acid" with 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 Hydrochloric acid and Nitric acid :

5.1.1.1 Skin contact : immediately rinse 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.