

RCA Clean for Silicon

Standard Operating Procedure

Lab Manager: Dr. James Vlahakis

Faculty Supervisor: Prof. Robert White, Mechanical Engineering (x72210)

Tufts Emergency Medical Services are at x66911.

Revised: July 8, 2015

Warning: Hydrofluoric Acid (HF) is an extremely dangerous material. Because of the ability of HF to produce severe *delayed* tissue damage without necessarily producing pain, any contact with HF should receive immediate first aid and medical evaluation, even if the injury appears minor or no pain is felt. Work carefully in the hood with full personal protective equipment, including apron, face shield and trionic gloves.

Do not use HF without proper training.

If HF does get on your skin, rinse the affected area with water for 5 minutes, and then apply calcium gluconate gel. The calcium gluconate is on the wire shelves in the lithography room. Have someone in the lab call **Tufts EMS at x66911**. Any contact with HF requires immediate medical attention.

Purpose: RCA is a three stage cleaning process for silicon substrates that removes organic and ionic contaminants and the native oxide layer, leaving a pure silicon surface.

1. Material Requirements:

- 1.1 Equipment:** Three 125mm x 65mm glass dishes for SC1 and SC2 cleaning. (These are wide enough for a 4" substrate and deep enough to contain a very active chemical reaction. Do not use shallow dishes. Bubbling can cause the acid mixtures to increase to several times the original volume.) Three 500ml polymethylpentene jars for the HF dip. Fluorocarbon (CTFE) tweezers.

Note: A full set of dishes have been set aside for use with RCA cleaning of silicon substrates. Do not use these dishes for any other purpose. Clean these dishes thoroughly before returning them to their storage bin (they should look like new). RCA Clean is used for processes that require absolute cleanliness. Please do your part to maintain that cleanliness.

Warning: HF attacks glass! You cannot put it in a glass container. Polymethylpentene and polyethylene are fine to use. Polystyrene (plastic Petri dishes are often polystyrene!) is not considered compatible with long-term HF exposure.

- 1.2 Chemicals:** Hydrofluoric Acid 50% (HF), Hydrochloric Acid (HCL), Ammonium Hydroxide 30% (Ammonia), Hydrogen Peroxide 30% (H2O2)

1.2.1 Hazards associated with these chemicals:

HF: Liquid or vapors are extreme health hazards; cause severe burns and bone loss, which may not be immediately painful or visible. Significant exposure (100 mL) to HF can kill directly. Please use extreme caution; HF is very hazardous, both acutely and long term.

Hydrochloric Acid: Liquid or vapors are serious health hazards; and cause severe burns. Hydrochloric acid is much more viscous than water, be prepared for this when you pour it.

Ammonium Hydroxide: Liquid is extremely basic and corrosive. Exposure can cause severe burns.

Hydrogen Peroxide: Liquid or vapors are serious health hazards; and can cause severe burns. Peroxide waste is an explosive hazard and vented caps must be used for these waste containers. Ensure these caps are available *before* proceeding.

1.3 Engineering Controls: Conduct procedure in ventilated fume hood. Store bottles of HF and HCL (sealed tightly) in the acid cabinet with secondary containment. Hydrogen peroxide is stored in the bases cabinet. Ammonium Hydroxide is stored in the base cabinet. 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. Never work with HF without all this equipment. Wear full protective gear for entire RCA procedure.

2.0 SC1 Clean: (H2O2:Ammonia:H2O = 1:1:5)

Complete all processes in the acid process fume hood. Wear full protective gear for entire RCA procedure.

These cleaning mixtures do not depend on precise measurements. Safe handling is more important than precise quantities.

2.1 Half-fill two glass rinse dishes with DI water (about 400ml).

2.2 Using a 250ml beaker, measure 125ml DI water, 25ml H2O2, and 25ml ammonia (in that order) into the third glass dish.

2.3 Place the dishes on fab wipes.

2.4 Place the wafer into the SC1 solution carefully with tweezers. Leave the tweezers in the first rinse dish if they contacted the SC1 mixture.

2.5 Wait at least **20 minutes**.

2.6 Transfer the wafer carefully to the first rinse dish with tweezers.

2.7 Leave the tweezers to soak in the DI water dish with the wafer.

2.8 Let the wafer and tweezers soak for at least 5 mins.

2.9 Transfer the wafer carefully to the second rinse dish with tweezers.

2.10 Let the wafer and tweezers soak for at least 5 mins.

2.11 Dump the used solution into a dedicated glass waste bottle properly labeled for hydrogen peroxide and ammonium hydroxide. Bring the waste bottle into the hood to pour.

- 2.12 Pour the first rinse water into the container used for the SC1 solution, then dump into the waste bottle.
- 2.13 Fill the two empty dishes with water. Dump into the waste bottle.
- 2.14 Rinse the empty dishes one more time, but now dump the rinse water into the “dilute acid/base waste” 5 gallon HDPE dilute water waste container.
- 2.15 Rinse the empty beaker twice, dump into the waste bottle.

3.0 SC2 Clean: (H₂O₂:HCL:H₂O = 1:1:5)

Complete all processes in the acid process fume hood. Wear full protective gear for entire RCA procedure.

These cleaning mixtures do not depend on precise measurements. Safe handling is more important than precise quantities.

- 3.1 Half-fill a glass rinse dish with DI water (about 400ml).
- 3.2 Using a 250ml beaker, measure 125ml DI water, 25ml H₂O₂, and 25ml HCL (in that order) into the third glass dish.
- 3.3 Place the containers on fab wipes.
- 3.4 Transfer the wafer into the SC2 solution carefully with tweezers. Leave the tweezers in the first rinse dish if they contacted the SC2 mixture.
- 3.5 With the wafer removed, you can now dump the second rinse into the waste bottle. Rinse one more time, but now dump the rinse water into the “dilute acid/base waste” 5 gallon HDPE dilute water waste container. Fill with DI water.
- 3.6 Wait at least **20 minutes**.
- 3.7 Transfer the wafer carefully to the first rinse dish with tweezers.
- 3.8 Leave the tweezers to soak in the DI water dish with the wafer.
- 3.9 Let the wafer and tweezers soak for at least 5 mins.
- 3.10 Transfer the wafer carefully to the second rinse dish with tweezers.
- 3.11 Let the wafer and tweezers soak for at least 5 mins.
- 3.12 Dump the used solution into a dedicated glass waste bottle properly labeled for hydrogen peroxide and hydrochloric acid. Bring the waste bottle into the hood to pour.
- 3.13 Pour the first rinse water into the container used for the SC2 solution, then dump into the waste bottle.
- 3.14 Fill the two empty dishes with water. Dump into the waste bottle.
- 3.15 Rinse the empty dishes one more time, but now dump the rinse water into the “dilute acid/base waste” 5 gallon HDPE dilute water waste container.
- 3.16 Rinse the empty beaker twice, dump into the waste bottle. Rinse the beaker one more time, but now dump the rinse water into the “dilute acid/base waste” 5 gallon HDPE dilute water waste container.

4.0 HF Dip: (HF:H₂O = 1:100)

Complete all processes in the acid process fume hood. Wear full protective gear for entire RCA procedure.

Warning: HF etches glass. Therefore, you must do HF processing in polyethylene or polymethylpentene containers only.

These cleaning mixtures do not depend on precise measurements. Safe handling is more important than precise quantities.

- 4.1 Half-fill two polymethylpentene rinse jars with DI water (about 250ml).
- 4.2 Use a 250ml polymethylpentene beaker to measure 200ml of DI water into the third polymethylpentene jar.
- 4.3 Pour a small amount of HF into the empty beaker. Use a disposable pipette (LDPE) to add 2ml HF to the 200ml of water. The pipettes currently stocked by the lab have a 5-8 mL bulb, so approximately half a bulb full of HF will be sufficient. The measurement does not need to be particularly precise.
- 4.4 Place the three jars on fab wipes.
- 4.5 Transfer the wafer into the HF solution carefully with CTFE tweezers.
- 4.6 Wait **one minute**.
- 4.7 Transfer the wafer carefully to the first rinse jar with CTFE tweezers.
- 4.8 Leave the tweezers (which have HF on them now) to soak in the rinse jar with the wafer.
- 4.9 Let the wafer and tweezers soak for at least 5 mins.
- 4.10 Transfer the wafer carefully to the second rinse jar with CTFE tweezers.
- 4.11 Let the wafer and tweezers soak for at least 5 mins.
- 4.12 Remove the substrate. Carefully blow dry with nitrogen. Store the wafer in a clean case.
- 4.13 Use the wafer as quickly as possible. The native oxide will begin to regrow immediately.
- 4.14 Dump the used solution into a dedicated HDPE waste bottle properly labeled for hydrofluoric acid. Bring the waste bottle into the hood to pour it! There is a polyethylene funnel you should use. The waste bottle must also be made of polyethylene (HDPE).
- 4.15 Pour the first rinse water into the jar used for the HF solution, then pour the second rinse water into the jar for the first. Dump both into the waste bottle.
- 4.16 Fill the three jars with water. Dump all into the waste bottle.
- 4.17 Rinse the jars two more times, but now dump the rinse water into the “dilute acid/base waste” 5 gallon HDPE dilute water waste container.
- 4.18 Rinse the empty beaker twice, dump into the waste bottle. Rinse the beaker one more time, but now dump the rinse water into the “dilute acid/base waste” 5 gallon HDPE dilute water waste container. With each rinse, flush out the pipette. Dispose of the pipette in the acid trash.
- 4.19 Inspect the jars and beaker to be sure they are perfectly clean. Blow dry with nitrogen. Return to storage bin.

5.0 Cleanup

- 5.1 Inspect all labware. Everything must be perfectly clean and dry. Reclean as needed.
- 5.2 Return all labware to its proper location.
- 5.3 Wipe up any drips in the area with chemical wipes and dispose in acid trash.

6.0 Storage:

- 6.1 Hydrofluoric acid should be stored in the acid cabinet in a tightly capped polyethylene bottle.
- 6.2 Hydrochloric acid is stored in the acid cabinet.
- 6.3 Hydrogen peroxide is stored in the bases cabinet.
- 6.4 Ammonium hydroxide is stored in the bases cabinet.

7.0 Waste Disposal:

7.1 HF waste:

- 7.1.1 HF wipes are disposed of in the acid trash can.
- 7.1.2 Liquid waste is collected in the HF waste container and stored in the satellite storage area with secondary containment. This container must be polymethylpentene or polyethylene (HDPE).
- 7.1.3 Dilute water waste from third rinses goes into the dilute water waste 5 gallon HDPE tank.

7.2 Hydrogen Peroxide and Ammonium Hydroxide:

- 7.2.1 Wipes should go in the acid / base waste bin.
- 7.2.2 Liquid waste should go in a waste bottle labeled for hydrogen peroxide and ammonium hydroxide. This container must be glass and a vented cap *must* be used.
- 7.2.3 *Do not mix peroxide waste and solvents, an explosion may result.*

7.3 Hydrochloric Acid and Ammonium Hydroxide:

- 7.3.1 Wipes should go in the acid / base waste bin.
- 7.3.2 Liquid waste should go in a waste bottle labeled for hydrochloric acid and ammonium hydroxide. This container must be glass.

8.0 Accident Procedures:

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

8.1 Hydrofluoric Acid (HF):

- 8.1.1 Skin contact: Rinse affected area with water for 5 minutes, removing contaminated clothing during the rinse. Apply generous amounts of calcium gluconate gel to the area. **Get immediate medical attention. Don't be shy. Call the medical center if you got HF on your skin. Tufts Emergency Medical Services are at x66911.**
- 8.1.2 Eye contact: Immediately flush with water for 20 minutes while holding the lids open. Do not apply calcium gluconate. **Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.**
- 8.1.3 Ingestion: Do not induce vomiting. **Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.**
- 8.1.4 Inhalation: Remove to fresh air. Resuscitate if necessary. Take care not to inhale any HF released from the victim's lungs. **Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.**

8.2 Hydrochloric Acid:

- 8.2.1 Skin contact: Remove contaminated clothing, rinse affected area with water for 10 minutes. If there is a visible burn, get immediate medical attention.

- 8.2.2 Eye contact: Immediately flush with water for 20 minutes while holding the lids open. Get immediate medical attention.
- 8.2.3 Ingestion: Do not induce vomiting. Get immediate medical attention.
- 8.2.4 Inhalation: Remove to fresh air. Resuscitate if necessary.
- 8.3 Ammonium Hydroxide:
 - 8.3.1 Skin contact: Remove contaminated clothing, rinse affected area with water for 10 minutes. If there is a visible burn, get immediate medical attention.
 - 8.3.2 Eye contact: Immediately flush with water for 20 minutes while holding the lids open. Get immediate medical attention.
 - 8.3.3 Ingestion: Do not induce vomiting. Get immediate medical attention.
 - 8.3.4 Inhalation: Remove to fresh air. Resuscitate if necessary.
- 8.4 Hydrogen Peroxide:
 - 8.4.1 Skin contact: Remove contaminated clothing, rinse affected area with water for 10 minutes. If there is a visible burn, get immediate medical attention.
 - 8.4.2 Eye contact: Immediately flush with water for 20 minutes while holding the lids open. Get immediate medical attention.
 - 8.4.3 Ingestion: Do not induce vomiting. Get immediate medical attention.
 - 8.4.4 Inhalation: Remove to fresh air. Resuscitate if necessary.
- 8.5 Spill:
 - 8.5.1 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 (acid trash).
 - 8.5.2 If a large spill occurs that you are not comfortable cleaning up, 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) or the faculty supervisor (x72210, Robert White).

Report all accidents (injuries, major spills, fires) to the safety office at x73246 and the faculty supervisor at x72210 (Robert White).

For emergencies, call Tufts Emergency Services at x66911.