University of California, Berkeley



Lab Manual

Marvell NanoLab

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Lab Manual Contents

<u>MercuryWeb</u>

#### **Revision History**

Chapter 2.0

# **General Cleaning Procedures and Sink Summary**

(General Cleaning Procedure - Labwide)

### 1.0 <u>Title</u>

General Cleaning Procedure and Sink Summary

### 2.0 Purpose

This General Cleaning Procedure chapter gives overview information for all sinks in the NanoLab.

### 3.0 <u>Scope</u>

This chapter provides summary of baths at all sinks; provides an overview of cleaning procedures for tweezers, labware and dummy wafers; provides exclusions and critical cross contamination control policies supplemented by the Material and Process Compatibility chapter 1.7.

#### 4.0 Introduction

All cleaning procedures must be performed correctly, otherwise wafers and labware are likely to become contaminated. Never touch a wafer with your hands, and always use clean tweezers. Use stainless steel tweezers for non-acid work and Teflon<sup>®</sup> holders for acid work. Use DI water only for any cleaning and rinsing in the lab. See section 4.0 and beyond for pre-furnace cleaning required for furnaces. See section 9.0 for sink summary and section 10.0 for bottle wash and dumbwaiter operation.

### 5.0 Cleaning Laboratory Ware

Correct and thorough cleaning of Pyrex, Teflon<sup>®</sup> or polypropylene ware (beakers, Petri dishes, wafer boxes, thermometers, stirring rods, etc.), is very important. All cleaning is to be done in the Wet Chemical Room of 582A of the Marvell Nanolab at either Msink16 or Msink18.

- 5.1 Scrub all items to be used in the lab in **Nova Clean NC-1** with a brush.
- 5.2 Rinse thoroughly in running DI water for two minutes.
- 5.3 Perform the **water break** test: Submerge the item in DI water and observe the film of draining water.

On a clean surface it will remain unbroken and fairly uniform. Contaminants will cause breaks or sharp irregularities such as **islands** to appear in the color fringes and droplets remain on the surface.

5.4 Allow to drain and dry chemical ware in the VWR or Vacuum oven on an aluminum foil sheet, under an IR lamp or with a nitrogen gun. Do not place any of the furnace boats or related quartz ware in any oven, as these boats need to be kept super clean, and or normally handled by staff.

# 6.0 <u>Cleaning Tweezers</u>

#### 6.1 Degrease

Degrease all new stainless and Teflon<sup>®</sup> tweezers as per follows, prior to cleaning:

- ▶ 5 minutes rinse in acetone
- ▶ 1 minute rinse in running DI water

# 6.2 Cleaning

# 6.2.1 Metal and Teflon<sup>®</sup> Tweezers - Removal of Organics

Use acetone as per Section 3.1, to remove residual organic contaminants.

# 7.0 Wafer Handling & Cleaning

To minimize contamination from the tweezers, never allow the rinse or dip to touch the tweezers before it touches the wafer. Rinse or blow-dry toward the tweezers.

## 7.1 Standard Wafer Cleaning in the VLSI Area

This section explains about mandatory pre-furnace wafer cleaning designed to remove possible residual organic and metal contaminants from wafers before going into Tystar furnaces. Standard pre-furnace clean steps are as per follows:

- 7.1.1 Piranha clean for 10 minutes at Msink6.
- 7.1.2 DI water rinse your wafers: Carefully lift the cassette out of the piranha bath, holding it above the bath until it stops dripping, and then place the cassette in the quick dump rinse station and start the DI rinse. Make sure proper resistivity level is reached ( ≥10 Mega Ohm-cm), before moving the cassette/wafers into the HF tank, next.
- 7.1.3 Short Hydrofluoric (HF) dip for 1 minute in the 25:1 or 10:1 HF bath, available at MSink6 (room temperature, bath ~ 25°C). Please note some process sequences cannot tolerate HF dip, in which case it can be skipped in the overall pre-furnace clean process, just receiving piranha in such cases will suffice.
- 7.1.4 Rinse wafers in the Msink6 QDR (quick dump/rinse), which is currently set for four dump/rinse cycles.
- 7.1.5 Final rinse until water resistivity reaches 10 Mega Ohm-cm.
- 7.1.6 Place cassette/wafers in the Msink6 SRD (spin-rinse/dryer) for a final rinse and dry cycle. Water resistivity should reach 12 or more after this step.

### Important Note:

An additional Msink8 cleaning (Piranha + short HF dip) prior to Msink6 cleaning is required for wafers that have just had photoresist stripped on them (MOS or non-MOS). This extra Msink8 clean is also required for non-MOS (MEMS) type processes, any time they arrived at furnace station, regardless of having had photoresist removed from them or not. MEMS (non-MOS clean) wafers always need to first receive Msink8 clean, then the Msink6 cleaning before they can enter any of the Tystar furnaces in the Marvell lab.

# 7.2 Thin Oxide Film Removal

Dip in 10:1  $H_2O$ :HF until front and back of wafer dewet. Pulling the wafer from the water leaves an absolutely water free surface with only a drop or two on the lowest edge. If this test fails, reclean.

**7.3 RCA Clean** (widely used in industry) and on contaminated Teflon cassettes and handles here in Marvell Nanolab, and as an additional measure to ensure cleanliness at our VLSI acid sinks, performed on an as needed basis (by staff). These procedures as an additional cleaning step to our standard piranha/HF clean are considered optional; therefore, need to get justified, set up, and performed by a member who may be interested in performing the RCA clean. Please, consult with staff when RCA clean is needed.

RCA-1 - Removes organic residues

5 H<sub>2</sub>O (DI Water) 1 NH<sub>4</sub>OH (Ammonium Hydroxide) 1 H<sub>2</sub>O<sub>2</sub> (Hydrogen Peroxide) RCA-2 - Removes metallic residues

- 6 H<sub>2</sub>O (DI Water)
- 1 HCl<sub>2</sub> (Hydrochloric Acid)
- 1 H<sub>2</sub>O<sub>2</sub> (Hydrogen Peroxide)

## Process

5 min. in RCA-1 at 70°C 5 min. in DI water rinse 5 min. in RCA-2 at 70°C DI water rinse to 10 megohm-cm

# 8.0 Quartzware Cleaning Procedure

Hydrofluoric acid (HF) is extremely dangerous. Always wear chemical-resistant gloves on top of your nitrile gloves, chemical resistant apron and eye protection (safety goggles plus face shield) when you work with it. Test your gloves for leaks by inflating them with the N2 gun to twice their size and immersing in a water bath. If any bubbles of escaping gas are observed throw the glove away.

- 8.1 For all quartzware, it is essential that oils (from fingerprints) or other organic contaminants be removed by wiping with trichloroethane followed by acetone.
- 8.2 The cleaning solution consists of 5% HF : 95% DI water.
- 8.3 Rinse well in DI water. Dry under IR lamp or in oven.

# 9.0 <u>Sink Summary</u>

<b>Sink Name</b> (Maximum Wafer Size)	Processes Available	Marvell Lab Location	Important Comments
msink1 (8")	PR strip, tank develop, pre-furnace metal clean	382	Note 2
msink2 (6")	PR strip, tank develop all metals allowed	382	Metal contaminated sink - Note 1
msink3 (8")	Manual Resist Processing	382	
msink4 (6")	TMAH & KOH etch	382	Staff Review required before wafers processed at this sink returning to msink6, msink7, msink8.
msink5 (8")	HF, BHF etch special project etch (check labels at tanks)	382	Metal contaminated sink, PR coated wafers allowed- see Note 1
msink6 (8")	Final Piranha - MOS clean sink	386	No metals allowed - MOS clean sink
msink7 (6")	Hot phosphoric, HF, silicon etch	386	No metals, PR coated wafers allowed
msink8 (8")	Piranha clean, BHF, aluminum etch	386	No metals except aluminum in the aluminum etch tank, no PR coated wafers
msink16 (N/A)	General clean - beaker process/part clean	582A	Metal contaminated sink - Note 1
msink18 (N/A)	General clean - beaker process/part clean	582A	Metal contaminated sink - Note 1

**Note 1** - Wafers processed at this sink are metal contaminated, ARE NOT ALLOWED to return to msink6, msink7, msink8.

Note 2 - Gold, copper and or other highly diffusive metals ARE NOT ALLOWED at this sink.

# 10.0 <u>Transferring & Disposing of Used (Emptied) Chemical Bottles</u>

# 10.1 Chemical Bottle Usage and Transfer

In general, make sure to check the station for available chemicals (existing stock), before checking in a new bottle or opening a new bottle to use.

To transfer bottles across the lab or between the two floors, plastic bottle carriers are to be used (strictly required). Bottle carriers are stored in front of the dumbwaiter at the North end of the lab. Up to two bottles can be carried up/down in these carriers by a member in stairwell. Up to nine bottle can be transferred in dumbwaiter between the two floors of the Marvell lab. See section 8.5 for dumbwaiter (elevchem) operation.

Chemical bottles that have been opened cannot be returned to the chemical storage cabinets in the chemical/gas vault; there are chemical storage cabinets for opened bottles throughout the lab. Partial bottles can also be stored in the storage carts (compartments) under the wet sinks. Empty chemical bottles must be transferred to Wet Chemical Room 582A, to the bottle wash station for disposal. Do not leave empty bottles by any equipment or at the sinks, as they must be promptly rinsed (flushed) properly, X marked and placed in recycle bins. This means all empty acid and solvent bottles need to first get thoroughly rinsed off in the bottle washer before discarding them in the recycle bins. Check the next section regarding the operation of bottle washer (bottlewash).

### **10.2 Bottle Washer and Disposal**

Empty chemical bottles need to be taken to the allocated area of the lab, currently the wet lab (582A) to get thoroughly rinsed off in the bottle washer machine, and their labels X-ed out with a marking pen to indicate the bottle has been rinsed, then disposed into the proper receptacle in this room.

Acid and water looks alike, wear proper acid protection attire and use the techni-cloths provided in the holders at each room to wipe and dry down work surfaces for any liquid that might have been left behind on surfaces (PH can be checked by acid paper). In case of small amount of acid spill, dilute the puddle with DI water, using the sink deck hose first, and then wipe it up clean with techni-cloth and discard the cloth.

# **10.3 Bottle Wash Station Operation**

- 10.3.1 No equipment enable is required for this bottle washer on the Mercury, equipment name "bottlewash' Shown in Figure1.
- 10.3.2 Always wear protective face shield, chemical resistant (yellow) gloves and apron during operation of this tool and/or handling of chemical bottles.
- 10.3.3 The bottlewash is a 2-step rinse/dry operation.
- 10.3.4 Open the top cover and double check no bottles are left inside (Figure2).
- 10.3.5 Take bottle caps off and flip the bottle/s one at the time upside down right above the wash compartment, in case there are acid or solvents left behind in the bottles. Lower the bottles over the wash nozzles (rods) one at the time inside the rinse compartment. Up to four bottles can be loaded/washed at any given time(Figure3).
- 10.3.6 Close the cover. Make sure the cover is sealed properly, as there are no sensors installed on this tool (there is no electricity connected for safety reasons).
- 10.3.7 Step on the blue pedal to begin the rinse sequence. Rinse for at least 45 seconds.
- 10.3.8 Next, step on the red pedal to dry. It will take approximately 12-15 seconds to clear water out of the line. High pressure N2 will flow through the system. Note: Only the inside of the bottle will be dried.
- 10.3.9 To retrieve bottles, lift cover and remove bottles one by one. Dry outside of bottle with a Teri\_Towel, X-out label with a marker, then dispose of bottle into the proper receptacle

Note: empty H2O2 bottles (hydrogen peroxide) rinsed, then get disposed into a dedicated blue bin (marked as H2O2 bottles) currently available to the right of the bottlewasher. These bottles are saved to be used for organic waste collection due to their special caps.

## 10.4 Dumbwaiter (elevchem)

A dumbwaiter is available to transfer full and empty chemical bottles between the two floors at the North end of the Marvell Nanolab, connecting bays 386 and 586. Lab members can proceed through the stairs or use this small elevator to transfer chemical bottles between the two floors. The tool is easy to use as per following instructions.

- 10.4.1 Equipment "elevchem" on Mercury (does not require enable).
- 10.4.2 Make sure the dumbwaiter is in standby mode (Figure 4). Check the IN USE lamp on top of the control panel is off.
- 10.4.3 If the CAR HERE status lamp is on, the elevator is ready to load/unload (Figure 5). Press UP/DOWN buttons once to move elevator to the correct floor and wait until CAR HERE lamp turns on to proceed.
- 10.4.4 To open front door, press the OPEN button once.
- 10.4.5 The elevator can accommodate up to 9 standard size bottles at a time. The plastic piece that is holding the first row of the bottles can be removed easily, and in case a bigger size item needs to get transported.
- 10.4.6 To close the door, press and hold the CLOSE button until doors have completely closed. The elevator won't start until the doors are fully closed.
- 10.4.7 Press UP/DOWN to transfer your items to your floor destination. Once the elevator has arrived and IN USE light is on, open the door as described above. When finished, make sure the doors are closed properly by holding the CLOSE button until doors closes up/stops.

### 11.0 <u>Tystar Dummy Cleaning Procedure</u>

### 11.1 Wafer Identification

All dummy wafers are specific to a furnace and should be scribed on the back with the furnace number. Each furnace deposits a different film as follows:

- tystar9 and tystar17: silicon nitride
- tystar11 and tystar12: doped and undoped silicon dioxide
- tystar10 and tystar16: doped and undoped polysilicon

Each film requires a different method of removal as described below. If you are unsure what film is on a dummy wafer, contact Sia Parsa, Process Engineering Manager.

### 11.2 Tystar9 (Silicon Nitride)

- 11.2.1 Make sure that the dummies are scribed on the back with the Tystar furnace name. If not, scribe TYSTAR9 on the back.
- 11.2.2 Load wafers into a Msink7 cassette and dip in 5:1 HF for about 15 seconds. (If necessary, fill the left non-MOS bath at Msink7 with the 5:1 BHF. Rinse the wafers thoroughly in the Msink7 non-MOS clean QDR.
- 11.2.3 Place cassette in phosphoric acid bath located in the right side heated bath of Msink7.
- 11.2.4 Turn the heater on and check that the set point is at 160°C.
- 11.2.5 Leave the cassette in the bath for about 24 hours. (Longer may cause surface damage to wafer characterized by a clouding of the smooth surface and recognizable polish marks. Damaged wafers should be thrown out.)
- 11.2.6 Remove, rinse, and dry the wafer cassette.

- 11.2.7 At this point the wafers may be stored for piranha cleaning in the original tystar dirty box. If so, label the box accordingly.
- 11.2.8 Standard piranha clean in Msink8 then Msink6.
- 11.2.9 Place clean, stripped dummies in clean tystar9 dummy box.

### 11.3 Tystar10 and tystar16 (Polysilicon)

- 11.3.1 If the dummies are new, dummies must be scribed on the back with the Tystar name. If not, scribe them.
- 11.3.2 Dummies to be reworked must be sorted. Those with obvious stress lines should be discarded.
- 11.3.3 Make special silicon etch if not made already 2200ml Water : 2200ml Nitric Acid : 90ml HF. This should be made in the left heated bath at Msink7. If the etch is already made up, add 15ml HF prior to etching.
- 11.3.4 Turn on the heater. The setpoint should be around 80-85°C. The heater greatly increases the etch rate. Place cassette of dummies in etch. The time of the etch will vary but it should never exceed 7 minutes. The sacrificial nitride layer may etch away resulting in surface damage. Surface damage is characterized by a clouding of the smooth surface and recognizable polish marks. The etch is very nonuniform so look out for thinning of the nitride layer. The etch is done when the wafers are brilliantly colored purple or yellow.
- 11.3.5 Remove, rinse, and dry the cassette. Inspect the wafers. If the wafer color is a light blue or the shade of a bare wafer, set wafer aside to be coated with nitride.
- 11.3.6 At this point the wafers may be stored for piranha cleaning in the original tystar10 or tystar16 dirty dummy box. If so, label the box accordingly.
- 11.3.7 Standard piranha clean in Msink8 then Msink6.
- 11.3.8 Place clean, stripped dummies in the clean tystar10 or tystar16 dummy box.
- 11.3.9 After a standard clean, dummies not previously coated with nitride or those that need to be recoated, should be coated in tystar17 (tystar10 dummies only!) or tystar18 (tystar16 dummies only!) using the recipe SNITC with a deposition time of 50 minutes Place dummies in the CLEAN tystar11 or tystar16 dummy box.

### 11.4 Tystar11 and tystar12 (LTO & PSG)

- 11.4.1 Make sure that the dummies are scribed on the back with the tystar name. If not, scribe them.
- 11.4.2 Dip in 5:1 BHF (Msink8) until wafers dewet. This usually takes at least 30 minutes. The time may be longer if the wafers have a very thick film on them. (The etch rate of fresh 5:1 BHF is about 1000A/min.)
- 11.4.3 Remove and rinse the cassette.
- 11.4.4 At this point the wafers may be stored for piranha cleaning in the original tystar12 or tystar20 dirty dummy box. If so, label the box accordingly.
- 11.4.5 Standard piranha clean in Msink8 then Msink6.
- 11.4.6 Place clean, stripped dummies in CLEAN tystar11 or tystar12 dummy box.
- **Note:** If you do not have the time to clean the dummies in both Msinks 8 and 6, do not just clean them in Msink8 and put them back into the dirty box. This defeats the cleaning you just did. If you find it necessary to split up the cleanings, use a separate clean box. Do not use the clean tystar box.

## 11.5 Tystar17 (Low Stress Nitride)

- Note: Tystar17 dummies are to be replaced every 4 weeks. Calendar mail will be sent as a reminder.
- 11.5.1 Retrieve new dummies.
- 11.5.2 Scribe the tystar number on the back of the dummies.
- 11.5.3 Standard piranha clean in Msink8 then 6.
- 11.5.4 Place clean dummies in CLEAN tystar17 box or directly into tystar17.

# 12.0 <u>Dummy Unload Procedure</u>

### 12.1 Furnace Status

Check the status of the furnace. It must be in the standby mode before you can unload the dummies. You can check the status at the furnace itself by pressing the STATUS (7) key, or by accessing the tycom on the wand under Equipment Communication--Tystar furnace interface. For tystar9 the standby program is called **9STNBYA**. For tystar11, it is called **11STNBYA**, for tystar12 the standby program is called **12STNBYA**, and for tystar16 it is called **16STNBYA** tystar20 and tystar18 are not monitored through the tycom, but have their own display panels. (See the tystar18 and tystar20 manual, Chapter 5.9 for procedures.)

### 12.2 Venting the Furnace

The furnaces will be under vacuum in the standby mode, and will need to be vented to atmosphere in order to open them. This can be done at the furnace by pressing the ACK (acknowledge) button on the furnace control panel. For tystar9 the ACK button must be pressed twice, for the other two furnaces only once. You should here a loud **squealing** sound from the back of the furnace as the furnace begins to vent. You can also check that the furnace is back-filling (venting) by pressing the STATUS button until the time countdown is displayed. The backfill step counts down for 00:07:00 (7 minutes), except for tystar16 where it backfills for 00:04:00Soon after the furnace begins venting, a loud alarm will sound. This is silenced by pushing the appropriate ALARM ACK button below the furnace control panels. When the seven minutes are up, the furnace will alarm to indicate the END of the process, and you can silence this alarm by pressing the ACK button. Located on the TYSTAR Furnace Controller Panel are the controls for retracting and sending in the cantilever holding the wafer boats. This is labeled BOAT CONTROL and has 3 buttons labeled IN, OUT, and STOP Press the OUT button to bring the cantilever out of the furnace.

### 12.3 Unloading the Dummies

For tystar9, the wafers are held in quartz cassettes. Remove all the dummies using the vacuum wand and place in the quartz cassette at the furnace until the dummies cool. When the dummies are cool, place them in the box labeled for dirty dummies. These can now be cleaned according to the Dummy Clean Procedure. For tystar11, tystar12, and tystar16 the wafers are held in quartz cages. The top portion of the cage must be removed using the quartz-tipped fork- like tool found at the furnaces. Place the tool with the lid in the metal plate holder located at the furnaces. The dummies can now be removed and placed in the quartz cassettes to cool. When they have cooled, place them in the appropriately labeled dirty dummy box, and later clean them according to the procedures outlined for dummy cleaning. Replace all dummies with clean ones.

### 12.4 Returning the Furnace to STANDBY

Press the IN button on the BOAT LOAD CONTROL panel to return the cantilever to the furnace (make sure that the cantilever is in completely). Press the RUN button on the furnace control panel to start the standby program. Press the STATUS (7) button until the panel displays the standby recipe name (9STNBYA for tystar9, 11STNBYA for tystar11, 12STNBYA for tystar12

16STNBYA for tystar16). Report on the WAND in the comments section for each furnace that clean dummies have been installed in the furnaces.



Figure 1 - Bottle Wash

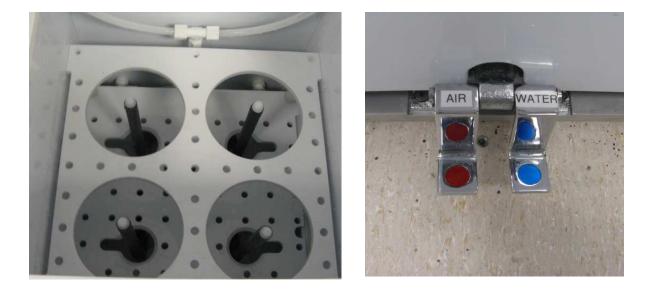


Figure 2 - Bottle Wash shower nozzle

Figure 3 - Bottle Wash foot pedals



Figure 4 - Elevchem (dumbwaiter)

Sia Parsa/Marily Kushner 05/22/12

Figure 5 - Elevchem (dumbwaiter) Buttons