Chapter 2.4

**msink4 Operation**

(msink4 - 382)

1.0 **Title**

msink4 Operation

2.0 **Purpose**

This document specifies information about msink4, the isotropic silicon etch sink.

3.0 **Scope**

Msink4 is assigned for isotropic silicon etching, which houses TetraMethyl-Amonium Hydroxide (TMAH) and Potassium Hydroxide (KOH) solutions.

Msink4 consists of two baths, one QDR, a general drain area, a deck hose, and a N2 gun (see an overview picture and other images shown in the Figures & Schematics section of this manual).

4.0 **Applicable Documents**

Publications/references:


5.0 **Definitions & Process Terminology**

**Quick Dump Rinse (QDR)** – DI water fills the dump rinse tank followed by a quick dump to get rid of excess acid and/or contaminants. This cycle may get repeated several times based on the program loaded on the unit (staff).

**Silence** – Alarm indicates sink problem.

6.0 **Safety**

For details, see MSDS sheets for KOH and TMAH. Do not drain etching solution above 60°C.

For safety reason, temperature high limit is set at 95°C. Do not heat baths above high limit temperature.

Rinse the beakers and tools you used to mix the solution.
EPO red button (Figure 1): Cuts power to the sink in case of emergencies. Report promptly on FAULTS.

Wipe down the KOH and TMAH contaminated surface you worked on.

Never touch any surface while wearing chemical-resistant gloves that other lab members may come into contact with, such as the table tops, door handles, computer keyboards, face shields, aprons, etc. If you need to step away from the sink at any time, rinse off the gloves, dry with techni-cloths, and put away in your drawer until you are ready to resume your work at the sink.

6.1 Tetramethyl-Amonium Hydroxide (TMAH)

Flammable (in 100% solution).

Concentrated TMAH poses significant chemical hazards. TMAH (Tetramethylammonium hydroxide) is widely used as a photoresist developer (2-3%) and for the anisotropic etching of silicon (10-15%). TMAH is corrosive, a strong base and hazardous by ingestion, inhalation, skin (dermal) exposure and eye contact. In addition to alkalinity-related chemical burn, dermal exposure to concentrated (>15%) TMAH may also result in respiratory failure and/or cardiac arrest. Fatalities have been reported after exposure to 25% TMAH to only 7% body surface area for less than a minute.

This chemical is stored in the tall yellow cabinet (381-C2) in chase 381. Open under fume hood in msink4 only. Labmembers must wear chemical resistant gloves, a chemical resistant apron, and face shield provided at msink4 at all times. A dedicated carrier for TMAH is located in chase 381 to carry TMAH from the cabinet over to msink4. Return carrier to chase 381 after transfer.

6.2 Potassium Hydroxide (KOH)

Concentrated KOH also poses a significant chemical hazard. KOH is corrosive, a strong base and hazardous by ingestion, inhalation, skin (dermal) exposure and eye contact.

7.0 Statistical/process Data

7.1 Problems and Comments Section under Equipment Section of the Mercury.

7.2 KOH etch results listed below have been generated by staff (01/2014). Please note the KOH percentages here are volume percentages. The weight of the solute (KOH palettes) divided by the volume of the solution (A mixture of dissolved KOH palettes in DI water) x100. See appendix section of this manual for more detail.

7.2.1 24% by volume KOH etch rate is the fastest and shows smoothest etched Si surface. The etch rate for this concentration was ~ 81.733 um/hr. (1.362 um/min.) with 1-0-0 type Si wafers.

7.2.2 28% by volume KOH etch rate is ~ 78.33 um/hr. at 80C with 1-0-0 P-type Si wafer and shows a smooth etched Si surface.

7.2.3 50% by volume KOH etch rate is ~73 um/hr. at 80C with 1-0-0 P-type Si wafer and shows a rougher etched Si surface plane.

8.0 Available Processes, Gases, Process Notes

Chemically resistant gloves should be worn on top of the nitrile gloves, plus a face shield and apron must be worn at all times while working at Msink4 and/or handling KOH and TMAH solutions.

<table>
<thead>
<tr>
<th>Bath</th>
<th>Chemical</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>Left-Heated</td>
<td>KOH</td>
<td>80°C</td>
</tr>
<tr>
<td>Right-Heated</td>
<td>TMAH</td>
<td>80°C</td>
</tr>
</tbody>
</table>
Both available silicon etchants at Msink4 etch silicon 100 planes at a relatively high rate to other crystal planes. For the plane (111) etch rate as a reference rate ‘1’, the (100) is 300-400, and (110) is 600 typically. Silicon Nitride is a good mask material for both etchants, and Silicon Dioxide may be used as a mask for TMAH. KOH solutions can produce smoother etch surfaces, yet the free radicals in KOH may affect device performance on wafers with fabricated devices. Solutions can have dramatically reduced etch rates based on doping, and n and p doped regions may be selectively etched with an applied bias during etching. Additives to TMAH can improve the selectivity to metals. See noted references for details.

Please check out KOH pellets or TMAH from staff (Kim or Marilyn). You will be charged for the amount of KOH pellets or bottles of TMAH you check out. Please plan ahead because KOH pellets or TMAH will not be available for check out during the non-business hours. The Nanolab will still stock small amount of TMAH and 500 gram bottles for general use and beaker scale preparation of KOH baths.

9.0 Equipment Operation

The sink operation is relatively easy—labmembers invoke the dump rinse cycle at the QDR and heated baths from a keypad mounted on the face of the station. Msink4 has two sets of cassettes to be used exclusively at this sink: one 4” and one 6” cassette with locking handles stenciled “TMAH” and one 4” and 6” cassette with locking handles stenciled “KOH”.

9.1 Control Key Description

There are four control/displays at this station. Two SOLO 4848 (Figures 1, 2 and 3) temperature control panels for two heated baths. The Microkleen Dump Rinser (Figure 1) control panel is for the quick dump rinse (QDR) station. The QDR is currently set up for two dump rinse cycles. Wafers are initially showered with DI water followed by two DI fill-dump cycles. These cycles end with wafers submerged in the water for operator to extract and dry with the N2 gun. See Appendix 1 for more details. The station performs an automatic self-cleaning, every 60 minutes by one QDR cycle. The Resistivity Monitor panel is not used at this sink. EPO MPC-901 (Figure 1) control panel controls the main power on msink4. There are also two Drain switches at this sink as described below.

9.1.1 Power Control Key Description for "SOLO 4848" Temperature Controller

<table>
<thead>
<tr>
<th>Key</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>SET</td>
<td>Set heating bath power to on and off (Figures 2 and 3).</td>
</tr>
<tr>
<td>^ and v</td>
<td>Change temperature set point.</td>
</tr>
</tbody>
</table>

9.1.2 Control Key Description for the quick dump rinse station (QDR)

<table>
<thead>
<tr>
<th>Key</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>START</td>
<td>Activates dump rinse cycle / Reactivates dump rinse cycle.</td>
</tr>
<tr>
<td>RESET</td>
<td>- Silences alarm.</td>
</tr>
<tr>
<td></td>
<td>- Deactivates the quick dump rinser.</td>
</tr>
<tr>
<td></td>
<td>- Automatically reset itself in preparation for another run.</td>
</tr>
<tr>
<td></td>
<td>- Exits program mode.</td>
</tr>
<tr>
<td>OPEN</td>
<td>- Manually dumps water in the tank.</td>
</tr>
<tr>
<td>SETUP</td>
<td>- Parameters to be written in the EEPROM memory.</td>
</tr>
</tbody>
</table>

9.1.3 Control Key Functions for Overall Sink Operations

<table>
<thead>
<tr>
<th>Key</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>EPO</td>
<td>Big red button for emergency stop. This will cut the power off to the entire sink. A red LED light will light up on the EPO status key, when the red EPO button is pushed in (activated).</td>
</tr>
<tr>
<td>MAIN</td>
<td>Power on when LED is on.</td>
</tr>
<tr>
<td>SYS</td>
<td>System is ON when LED is on.</td>
</tr>
<tr>
<td>POWER ON</td>
<td>Turns main power ON located at top of control panel.</td>
</tr>
<tr>
<td>POWER OFF</td>
<td>Turns main power OFF located at top of control panel</td>
</tr>
</tbody>
</table>
ALARM  Silences any alarm at this sink.

9.1.4 KOH and TMAH Tank Drain Switches

There are two drain switches, one for each of the two heated baths in Msink4. Do not drain chemicals at high temperature. Turn off the heater to drop the temperature at or below 60°C, before draining the chemicals. This will protect the drains pipes from melting.

**STATIC BATH SWITCH ON**  Switch in up position will drain the respective tank.

**STATIC BATH SWITCH OFF**  Switch in down position will close the drain valve.

9.2 Quick Dump Rinse Operation (Figure 1)

9.2.1 Place wafers in the tank; tank initially should be full of DI water.

9.2.2 Press **START** button to activate the dump rinse cycle (Figure 1). It will cycle down from 2 to 1 then show 0 in the display window. At the end of the two cycles a beeping alarm will sound.

9.2.3 Press **RESET** to silence the alarm.

9.2.4 Press **RESET** to reset itself in preparation for another run.

9.2.5 Press **OPEN** to manually dump the tank.

9.2.6 Upon completion of two rinse cycles remove wafers dry with N2 gun.

9.3 Etching

Mix TMAH bath on the right side and KOH on the left side:

KOH etches silicon dioxide, so it is not a suitable masking material for long etches. Silicon nitride is the preferred masking material. Additional information is available in Chapter 1.9.

9.3.1 Rinse the beaker thoroughly, dump water and make sure it is clean before processing if you need to use it. KOH pallets are available in 500 gram and 3000 gram (3Kg) bottles. Only two 500 gram bottles available in the chase behind Msink4, as well as Msnk16&18 in 582A to make small etch bath. Members must obtain larger amount as a check out item (to make a full bath of KOH solution at Msink4). Labmembers will need to plan ahead for doing their work on non-business hours. This means the lab members must check out the KOH pallets ahead of time and during business hours. Follow KOH and TMAH instructions listed below.

9.3.2 KOH - With the cassette out of the tank, add DI water until the 12.5 liter mark has reached and pour the whole container of 3 kg KOH pallets into the KOH tank. This should result in 24% by volume KOH (3Kg KOH/12.5Kg of DI water). Mix the new solution and wait until your etch temperature set point has stabilized. Flip the toggle switch up on the HEATED BATH-1 O2 BUBBLER switch for the KOH bath. This is our standard concentration which is the simplest, fastest and most cost effective way of preparing a full KOH bath for 6” wafer batch processing at Msink4. This also gives the smoothest Si surface that we tested. Please note this concentration is different from Kirt William’s report in our VLSI etchant chapter1.9. See appendix section of this manual for more information.

**Note:**  The KOH tank requires ~12.5 liters of solution to cover 6” cassette and to activate the level sensor. 12.5 liter mark is ~ ¼” below the bottom white fitting or 2 ½” from the rim of the KOH tank.

9.3.3 TMAH solution is commonly stocked in a 25 % concentration. Calculate the amount of 25% solution and DI water to make the desired etchant concentration (often 3%-15%). Under the hood, open TMAH container, and add desired amounts of solution and DI water to bath. Make enough solution to reach ½ inch above the metal flowing cup contact
nut. This amount should be sufficient to cover a 6” wafer cassette. Mix the new solution and wait until your etch temperature set point has stabilized. Flip the toggle switch up on the HEATED BATH-2 N2 BUBBLER OFF/ON switch for the TMAH bath. Note: Mixed etchant solution must be high enough to activate metal flowing cup for heater to turn on.

9.3.4 Make sure the Main Power button is on the MPC-901 controller on the sink.
9.3.5 Make sure the SOLO 4848 Temperature Controller LED is lit at the top of the sink.
9.3.6 Press v or ^ button on the left SOLO 4848 temperature controller to choose the desire KOH temperature on the top panel and press SET to set the desire temperature for KOH etchant (Figure 2). Press v or ^ button on the right SOLO 4848 temperature controller to choose the desire TMAH temperature on the top panel and press SET to set the desire temperature for TMAH etchant (Figure 3).
9.3.7 Typically the bath should set to 80ºC. The temperature will reach the set point in 30-60 minutes for the bath. During the temperature stabilization, a visible interface may form between the hot and cold (KOH and water) liquids. Load an empty cassette with a handle as a stirrer, this interface may be broken to allow the mixture to stabilize faster.
9.3.8 Remove the lid. Careful when removing the lid because solution condenses on it. Carefully tilt the lid at an angle to drain off the condensed liquid back into the heated tank and place the lid on the bench top between the tanks. Load wafers to be etched in the cassette and place it in the bath when bath temperature reached what you want.
9.3.9 Replace the lid and start timing the etch. For critical runs, the wafer can be rotated during the run (Remove the cassette of wafers from the heated bath, rinse the cassette of wafers in the QDR, pull the cassette of wafers out of the water, rotate the wafers 180° and place the cassette of wafers back into the heated bath). The etch rate is temperature dependant (a factor of 2 to 3 for every 10ºC for KOH), and temperature significant temperature gradients may develop in the solution. The etch rate is approximately 1.36 um per minute for 24% by weight of KOH (3 kg and fill with D. I. water to 12.5 liter mark) and ~1 um per minute for TMAH (5%) at 80ºC. Check the liquid level every hour and add D. I. water to the level where you started.
9.3.10 When the etching is completed, remove the lid and cassette. Let the wafers cool briefly before rinsing by partially dipping the wafers in the QDR (Quick Dump Rinse) to avoid breaking membranes. Initiate the rinse cycle. For gentle rinse, use deck hose instead of QDR rinse.
9.3.11 If you have finished etching, shut off the heater. Press v button until SOLO 4848 Temperature Controller displays 10C, press SET to set the temperature to cool down the KOH or TMAH bath. Flip the toggle switch down to turn off the HEATED BATH-1 O2 BUBBLER or HEATED BATH-2 N2 BUBBLER OFF/ON controller on the KOH or TMAH bath. Drain the solution at 60ºC or lower, rinse the bath, drain the bath and repeat the process 3 times with DI water if needed. Disable the sink afterwards. Report on Mercury, if for some reason the drain switch does not drain the solution. Make sure you explain which tanks and which switch is not functioning properly.

10.0 Troubleshooting Guidelines
10.1 If the sink does not power up, then check Photohelic Differential Pressure reading (Figure 1) and makes sure it is within acceptable range (between the two red bars): Consult with staff to check house exhaust pressure, if it is outside the limits set by the two red bars.

10.2 Sink System Status Indicators (displayed on MPC-901 located on top sink panel)

**LOW PURGE**  Red indicator light will come on when there is low air purge to cool off electronics for the sink.

**DRAIN**  KOH and TMAH switches up to drain tanks.
SILENCE: If it sounds, push the SILENCE button to silence the alarm on the sink, and notify the process staff and post the problem on Faults.

10.3 If EPO is pushed, follow instructions:

10.3.1 Pull out EPO button.

10.3.2 Open head case pull down breaker switch and then lift up.

10.3.3 Push button on front of sink that says power on.

11.0 Figures & Schematics

Figure 1
Figure 2

Figure 3

Figure 4

12.0 Appendices
12.1 DECK HOSE Instructions

The de-ionized (DI) water deck hose for the sinks is **ALWAYS** available for emergencies; it provides a good safety backup in the event of exposure to chemicals.

12.2

<table>
<thead>
<tr>
<th>Temperature Controller</th>
<th>Dump Rinser</th>
</tr>
</thead>
<tbody>
<tr>
<td>Left SOLO 4848 KOH 80ºC</td>
<td>CY 2</td>
</tr>
<tr>
<td>Right SOLO 4848 TMAH 80ºC</td>
<td>Fp 20</td>
</tr>
<tr>
<td></td>
<td>dp 10</td>
</tr>
<tr>
<td></td>
<td>Sd 0</td>
</tr>
<tr>
<td></td>
<td>Ad 0</td>
</tr>
<tr>
<td></td>
<td>n2 n</td>
</tr>
<tr>
<td></td>
<td>Ac 0</td>
</tr>
<tr>
<td></td>
<td>Pc 5</td>
</tr>
<tr>
<td></td>
<td>dc 1</td>
</tr>
<tr>
<td></td>
<td>Pn 0</td>
</tr>
<tr>
<td></td>
<td>nb 10</td>
</tr>
<tr>
<td></td>
<td>SL 1</td>
</tr>
</tbody>
</table>

12.3 KOH etch test discovery:

12.3.1 We test etched 1-0-0 P-type test wafers with O2 bubbler activated at 80C with three different concentration of KOH for two hours. It was concluded that the 24% by volume KOH etch had the fastest etch rate which etched 81.733 um/hr. (1.362 um/min.) This KOH concentration also had the smoothest etched Si surface. 28% by volume KOH etch rate was ~ 78.33 um/hr. (1.305 um/min) and showed a fairly smooth etched Si surface. 50% by volume KOH etch rate was ~73 um/hr. (1.257 um/min.) and showed a rougher etched Si surface plane than the other two KOH concentration. Below are the pictures and data that support the result.
24% by volume KOH etched Si surface  28% by volume KOH etched Si surface

50% by volume KOH etched Si surface

24% by volume KOH etched Si surface roughness in height is ~0.231 um
28% by volume KOH etched Si surface roughness in height is ~1.065 μm
50% by volume KOH etched Si surface roughness in height is \(\sim 1.257\ \text{um}\)

12.3.2 KOH bath concentrations were tested as follow:

12.3.2.1 **24% by volume KOH method is our standard bath for etching silicon wafers which proved to provide the smoothest Si surface.** Add D. I. water until the 12.5 liter mark in the clean KOH tank. Add One 3 kg bottle of KOH pallets, mix well, heat it up to 80C, and turn on the O2 bubbler before etch. The 24% recipe described that it yields faster etch rate, smoother surface and saves chemicals.

12.3.2.2 Mix 28% by volume KOH for etching silicon wafers. Add D. I. water until the 12.5 liter mark in the clean KOH tank. Add One 3 kg bottle and one 500 gram KOH pallets, mix well, heat it up to 80C and turn on the O2 bubbler before etch. 28% etched Si surface didn’t yield as smooth as the 24% etch method.

12.3.2.3 Mix 50% by volume KOH (this is the original concentration which Kirt Williams (IEEE 2003 publication) used for etching silicon wafers. **See staff if you plan to prepare 50% KOH using the outdated method of 5.5 kg KOH pallets to 11 liters D. I. H2O.**

Note: The KOH data in Kirt William’s report calculate the KOH concentration by weight using following formula:
Weight of the solute (KOH pallets) / weight of the solvent (DI) + weight of the solute (KOH pallets).