Potassium Hydroxide (KOH) Anisotropic Silicon Etch

Standard Operating Procedure

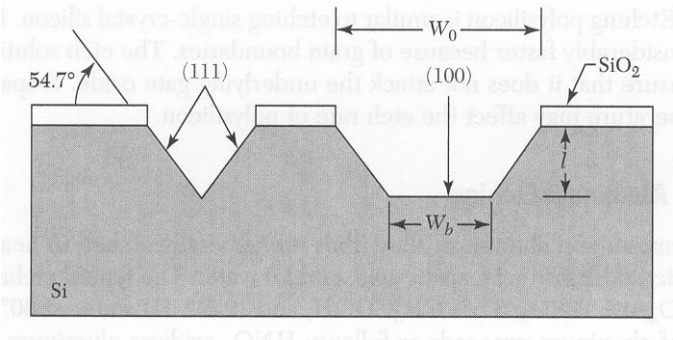
Faculty Supervisor: Prof. Robert White, Mechanical Engineering (x72210)
Safety Office: Peter Nowak x73246 (Just dial this directly on any campus phone.)
(617)627-3246 (From off-campus or from a cell phone)
Tufts Emergency Medical Services are at x66911.

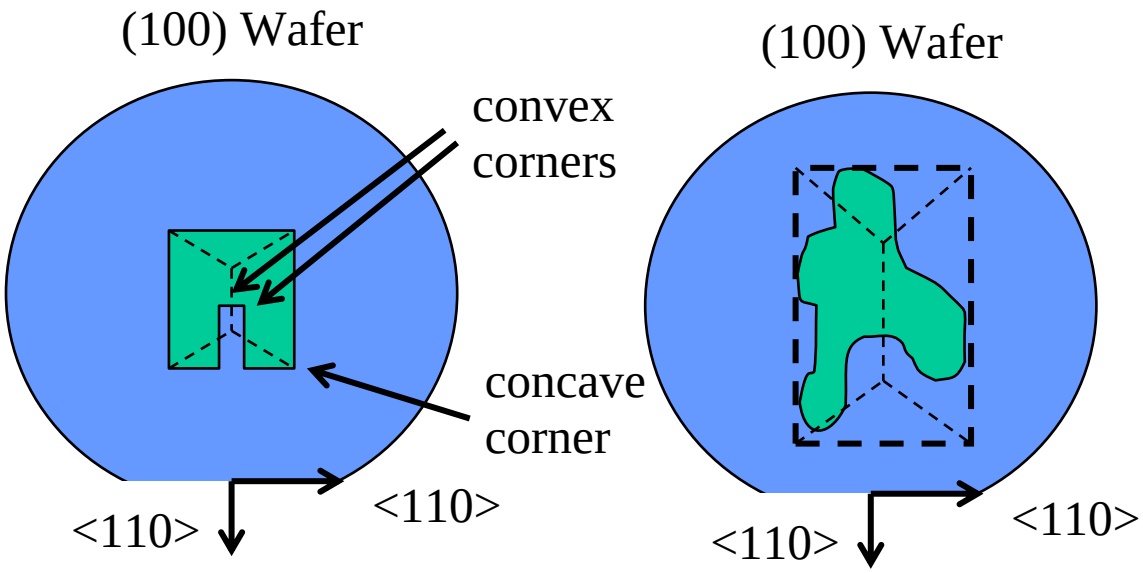
Revised: September 7, 2011

Warning: Potassium Hydroxide (KOH) is corrosive and causes severe eye and skin burns. Causes severe digestive and respiratory tract burns. Harmful if swallowed.

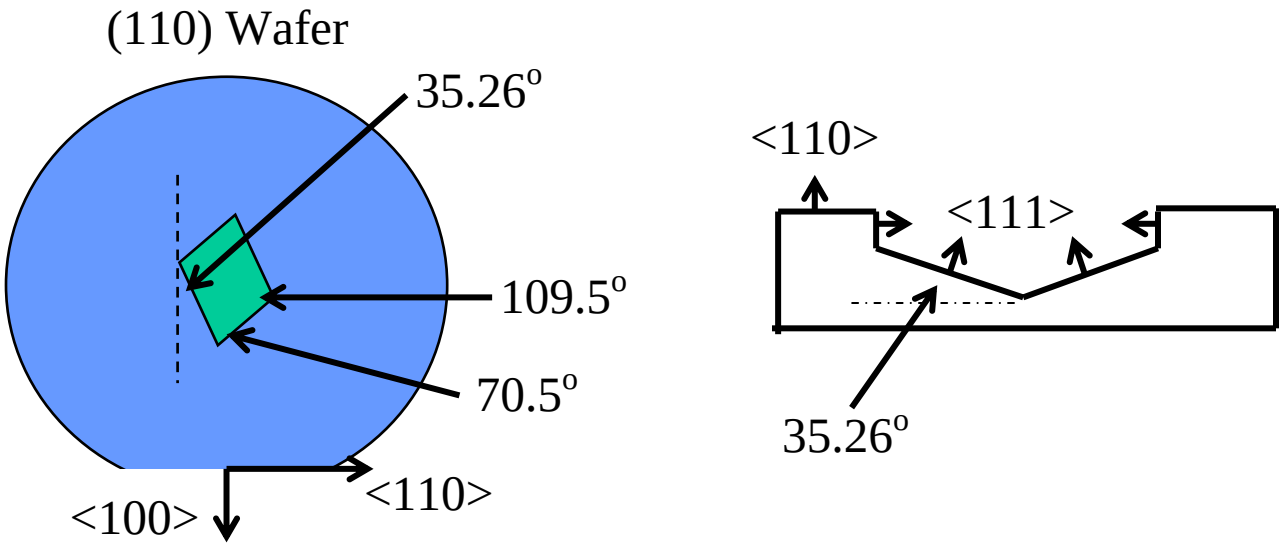
Notes: The 30% KOH solution at 80° C described in this lab will etch silicon <100> planes at approximately 1.5 μm/min. It will etch <111> planes at a much lower rate (not measured, Madou suggests 600:1 ratio of etch rates). Silicon nitride is an excellent masking material for this etch (literature suggests less than 1 nm/min, not measured in our lab yet). SiO_2 can be used as a mask, but will etch slowly (etch rate measured for thermal oxide ~ 10 nm/min, agrees with literature). Photoresist **can not** be used as an etch mask.

<100> oriented wafers will etch into characteristic v-grooves and pyramidal pit shapes. Convex corners will be undercut. The pyramidal pits have top edges oriented parallel and perpendicular to the <110> direction (the major flat). Clamped-clamped beams can be created in <100> oriented wafers by angling them at 45° with respect to the major flat. They will be undercut.





(110) oriented wafers will etch deeper trenches with initially vertical sidewalls, but will eventually stop on <111> planes creating a slanted base to the trench.



Potassium (K+) is an extremely fast-diffusing alkali metal ion, and a lifetime killer for MOS devices. Do not bring samples that have been processed using KOH into a processing environment where MOS devices are being fabricated; you may contaminate that facility and seriously disrupt its ability to produce functioning MOS devices!

1. Material Requirements:

1.1 Equipment: One stainless steel beaker (1000 mL), two glass rinse, graduated cylinder. PTFE (Teflon) wafer or sample carriers, PTFE coated thermometer, hotplate, digital scale, stainless steel tweezers.

1.2 Chemicals: Potassium Hydroxide (KOH) pellets, isopropanol

1.2.1 Hazards associated with chemicals:

1.2.1.1 Isopropanol (IPA): extremely flammable; keep away from sparks and flames, in case of fire, use water spray, alcohol foam, dry chemical or carbon dioxide. Harmful if swallowed or inhaled, causes minor skin irritation.

1.2.1.2 Potassium Hydroxide: Causes severe eye and skin burns. Causes severe digestive and respiratory tract burns. Heats upon dissolution in water.

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 Mix 30% KOH solution: mix from 100 g KOH pellets:200 mL DI water: 2 mL IPA

2.1.1 Measure desired weight of KOH pellets on the digital balance using a polystyrene Petri dish (labeled "KOH weighing") as a weigh boat. Choose desired weight to produce final required KOH solution volume: 100 g of KOH pellets per 200 mL of DI water.

2.1.2 Put weighed KOH pellets in the stainless steel beaker.

2.1.3 Add required amount of water (200 mL of DI water for every 100 g of KOH pellets) using a graduated cylinder.

2.1.4 Add 1% (by volume… so 1 mL IPA per 100 mL DI water) isopropanol to the solution if desired (this will give a smoother etch surface by reducing surface tension so that H_2 bubbles formed during etching will be smaller and detach more easily).

2.1.5 Place the stainless steel beaker on a tinfoil covered hotplate.

2.1.6 Put the PTFE coated thermometer in the solution.

2.1.7 Put a Pyrex watch glass on top of the beaker, convex side down, to catch most of the evaporating solution and return it to the beaker. If you do not use the watch glass, you will get a lot of spots of dried KOH solution in the hood and on the walls behind the beaker!

2.1.8 Turn on hotplate and set temperature so that final solution will stabilize at 80°C. It was observed that (without a watch glass!!) for 200 mL of KOH solution, hotplate temperature needs to be set at 170°C for the solution to reach a stable final temperature of 80 °C. It took about 30 mins to heat up to final temperature. For higher volumes, higher temperatures may be required. **Be careful with this step… keep careful watch on the solution temperature as it heats up, do not let it exceed 90 °C**.

2.2 Etch:

2.2.1 Fill the DI water rinse beaker with enough DI water to cover your sample, and have it ready before you begin the etch so if something goes wrong you can move your sample out of the etch and quench it immediately.

2.2.2 After the 30% KOH solution has reached equilibrium at 80°C, place the sample to be etched in the solution using PTFE sample handling tools.

2.2.3 Etch for the desired length of time. Expect approximately 1.5 μm/min etch rate of silicon in the <100> direction, and undercut rate at convex corners of about 3 μm/min.

2.2.4 When etch time is complete, remove the sample from the KOH and place it gentle into the DI water rinse. Soak in DI water for 5 mins.

2.2.5 Turn off the hotplate.

2.2.6 Transfer to a second DI water rinse for 5 mins.

2.2.7 An alternative to using the second DI water rinse is to transfer to a 5 minute isopropanol rinse followed by a 5 minute methanol rinse. This may be particularly useful if you are using the KOH etch to undercut MEMS structures which are now released; by doing your final rinse in methanol, you will have less stiction problems than if your final rinse is water.

2.3 Cleanup:

2.3.1 After turning off the hotplate, allow the KOH solution to cool to less than 30 °C before proceeding with cleanup.

2.3.2 The watch glass used to cover the KOH etch beaker, the PTFE coated thermometer, and any other tools used in the KOH etch process will have deposits of KOH on them. These must be carefully rinsed off using the deionized water squirt bottle. Rinse into the KOH etch beaker until all deposits have been removed.

2.3.3 Dispose of the KOH solution in a HDPE bottle labeled "Potassium Hydroxide" waste.

2.3.4 Rinse the KOH beaker twice more with deionized water and dump into the potassium hydroxide waste.

2.3.5 Dump the first DI rinse beaker into the potassium hydroxide waste.

2.3.6 Dump the second DI rinse beaker into the 5 gallon HDPE "dilute acid/base waste" container.

2.3.7 Rinse all the containers again with DI water and dump into the 5 gallon HDPE "dilute acid/base waste" container.

2.3.8 Remove the tinfoil from the hotplate and dispose of it in the acid/base trash can.

2.3.9 Wipe down the hood and the walls of the hood near the KOH etch beaker with deionized water and fab wipes; the KOH etch tends to deposit white spots of KOH everywhere, these need to be wiped up with water and thrown away in the acid/base trash.

2.3.10 If any solvents were used for final rinses, these should be disposed of in the mixed solvents waste bottle. All DI water rinses of the solvent containers should be dumped into the solvent waste bottle as well.

2.4 Storage

2.4.1 Potassium Hydroxide (KOH) pellets should be stored in the "Base" cabinet.

2.4.2 Isopropanol should be stored in the "solvents" cabinet.

3.0 <u>Waste Disposal:</u>

3.1 KOH waste:

3.1.1 Solid waste should go in the acid/base trash bin.

3.1.2 Liquid waste should go in the potassium hydroxide waste bottle.

4.0 <u>Accident Procedures:</u>

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

4.1.1 KOH solution:

4.1.1.1 Skin contact: Remove contaminated clothing, wash skin with soap and water. **If there is any irritation, get immediate medical attention. Don't be shy. Tufts Emergency Medical Services are at x66911.**

4.1.1.2 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.**

4.1.1.3 Ingestion: Do not induce vomiting. **Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.**

4.1.1.4 Inhalation: Remove to fresh air. Resuscitate if necessary. Take care not to inhale any fumes released from the victim's lungs. **Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.**

4.2 Spill:

4.2.1 If a small, contained spill occurs, such as inside the hood, wipe it up with chemical wipes and dispose of in the acid trash container.

4.2.2 If a large spill occurs that you are not comfortable cleaning up:

4.2.2.1 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, 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.