KOH and TMAH Etching of Bulk Silicon

Recipes, Tricks, What is Possible, and What is Impossible

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February 11, 2010

1) General:

KOH and TMAH are anisotropic silicon etches, used to make V-grooves, membranes, and holes through wafers, as well as other devices. This is an ‘old’ technology for MEMS (30 years +), it is used a fair amount, but many advanced applications are still not possible, even after the 100’s of papers published on the etch processes.

KOH and TMAH are close relatives. KOH is simply the hydroxide of potassium. TMAH is a organic hydroxide and stands for Tetramethyl ammonium hydroxide. KOH for etching comes in bottles of 45% by weight concentration and TMAH comes as 25% by weight concentration. Out of all the hydroxides, KOH etches the nicest.

2) Wafers

When etching into Silicon using KOH or TMAH, always use prime wafers. If you are doing a shallow etch, single sided polished is fine, but to go through a wafer or remove a large part of a wafer, double sided is needed to do the alignment.

The reason why prime wafers are needed is that the ‘history’ of the wafer is critical to the quality or the etch (or if it will etch at all). The cheaper wafers (coin rolled and test), are lower quality with often doped areas that can cause etch rate changes or the flats on the wafers are cut at the wrong angles and the devices can not be formed. Prime wafers have a known history and are high quality, so these issues are less of a problem.

3) (100) plane Etching and Masking Layers

Most often KOH and TMAH etches are used to etch the (100) plane, forming the membranes, V-grooves and other structures. The side walls of the etch are defined by the (111) planes. The angle between the sidewalls and the (100) plane is 54.7 degrees. Any MEMS textbook is a good resource for more details.

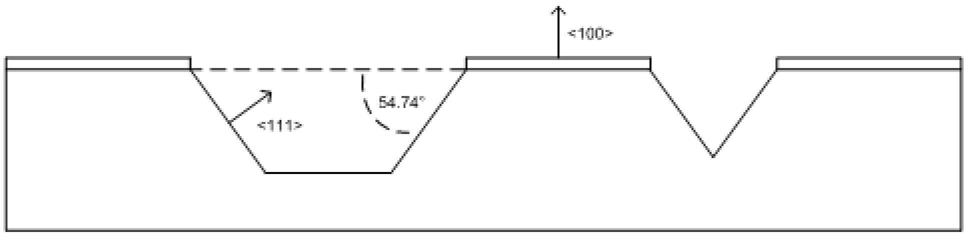

Figure 1. Typical side profile after anisotropic etch

The major flat on the wafer is aligned carefully (via the microscope) to the opening in the mask. This ensures the etched areas are bounded by the (111) planes. With care you can align to ~0.2 degrees and the specification on the wafers is that the major flat is with in 0.5 degrees of the (111) planes.

Keep in mind that KOH and TMAH will etch any exposed silicon. A masking layer may need to be applied to the backside of the wafer or a carrier used that will protect the backside.

a) Shallow KOH etch:

- Etch solution is 32% KOH by weight
- Heat to 95 degrees C with stir bar slowly stirring
- Add wafer to be etched
- Etch rate ~1.0um/min
- Masking layers: SiO_2 (thermal) or SiN_x
- KOH etches SiO_2, mask will only last for <50um deep Si etch
- SiO_2 masking layer can be removed with BOE (buffered oxide etch)

b) Deep KOH etch

- SiN_x mask will survive etch a deeper etch (through wafer - 50um)
- SiN_x masking layer can be removed by dry etch (fast) or by a long soak in BOE (>8hrs)

c) TMAH etch (shallow or deep)

- Use 25% TMAH (no dilution)
- Heat to 95 degrees C
- Wafers must be BOE dipped for 30 seconds to remove native oxide in opened areas (TMAH will NOT etch SiO_2, even nanometers of native oxide)
- Etch rate: ~0.6um/min
- Mask: SiO_2 (Thermal)

4) Smoothness of (100) surface

The normal surface after a KOH etch often looks like an orange peel. A TMAH etched surface can not only have the orange peal surface, but can also have pyramids left. These are areas that did not etch due to either local masking by dirt, or SiO_2 that wasn’t removed or that is found within the wafer.

To smoothen the (100) surface, there are a number of ‘tricks’ that can be used:

1. Add IPA to the solution. Ensure that a layer of IPA remains on top of the KOH or TMAH solution during the entire etch. The etch rate of KOH and TMAH will drop significantly.
2. Tilt the wafers to make it easier for the hydrogen bubbles to come off the surface (this is a cause of the roughness)
3. Add a surfactant. There are recent papers that gives the best surfactants for KOH and TMAH. The papers are in the journal “Sensors and Actuators A”, as well as the latest work in this field

5) (111) to (100) ratio …. The Undercut

The ratio of (100)/(111) etch rates vary in the literature from 20:1 to 200:1. In my experience, 25:1 to 35:1 are realistic. This ratio describes the undercutting of the masking layer as the (100) plane etches. The higher the ratio; less the undercut. Most applications would like no undercut, but that does not seem possible.

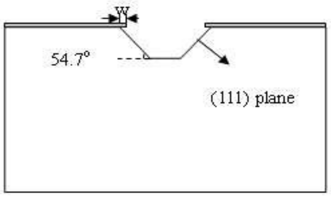

Figure 2: Undercut in anisotropic etching (W)

The reason for the large variation in the literature is not clear and not investigated. My personal opinion is that it is related to the accuracy to which the sidewalls of the mask layer are parallel to the (111) plane. The closer the edge is to parallel, the lower the rate. However, given the 0.5 degree unknown in the flat to (111) plane in the wafer and 0.1-0.2degrees in the alignment of the mask to the major flat, gives plenty of room for a significant non parallel mask edge to the (111) plane. Another reason could be a non straight mask edge, but rather a rough mask edge, due to poor lithography, which allows for greater undercut.

6) Etching structures that require higher order planes (points, flat areas, compensation structures)

Etching structures that use the higher order planes (i.e. features that are left above the (100) etched surface), this is an area of very little solid knowledge. Which plane that appears after the etch is not certain. The (411), (114), (311), (5,5,12) and many others have been identified in the literature.

It appears that the higher order planes that appear depend on the contaminates in the KOH and TMAH baths (HCO_3, Na, B, and others), mask placement, mask shape, age of bath, and others. The only advice I can give after etching 100’s of wafers is that if you need compensation structures or sharp AFM tips, is to try it a few times and determine what works best to achieve desired results.

Standard Operating Procedure: **Isotropic Silicon Etch (HNA)**

Principle of Operation

For isotropic wet etching of silicon and polysilicon. Etch rates are on the order of 1-5 µm/min depending on the ratio of chemicals used.

Material Requirements

Equipment: substrate, two polypropylene containers, extra polypropylene beakers for measuring and PTFE tweezers

> *Warning: HF attacks glass, so do not put it in a glass container.*

Chemicals: Hydrofluoric Acid (HF 48-51%), Nitric Acid (HNO_3 60%) and Acetic Acid ($C_2H_4O_2$ 99.5%)

- Hydrofluoric Acid Hazards
    - 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. Use extreme caution, HF is very hazardous, both acutely and long term.
- Nitric Acid and Acetic Acid Hazards
    - Liquid or vapors are serious health hazards and cause severe burns.

Personal Protective Equipment: Trionic gloves on top of nitrile gloves, apron, safety glasses and face-shield

Procedure

Estimated Time:

Note: This etch is intended for etching silicon and polysilicon, but it will also attack silicon dioxide and some metals (aluminum, titanium, etc.). You can mask an HNA etch with photoresist for a short etch (~1 minute). Use a 40 minute, 120°C hardbake on your resist. If you etch longer, the photoresist will start to peel off.

HNA Etch

1. Rinse both beakers with DI water prior to beginning the process.
2. Stand the beaker to be used for rinsing on a few fab wipes in the hood and fill it with DI water so that the water level will cover the entire substrate.
3. Get a container that will fit your samples for processing. Put it on fab wipes in the hood.

4. Determine the ratio of acids you want to use and the required volumes of each to completely submerge your sample.
5. Measure out the necessary volumes of acetic acid, nitric acid and HF and add them to the HNA container.
6. Calculate the etch time for you sample. You will need to know what etch rate you expect with the ratio of chemicals you are using.
7. Put your sample into the etchant and soak for the appropriate amount of time calculated in the previous step.

DI Water Rinse

1. When the etch is complete, transfer the sample carefully to the DI water rinse beaker.
2. If you used tweezers to move the sample, make sure you leave them in the rinse beaker as well.
3. Let the sample and tools soak in DI water for 5 minutes.
4. Remove the sample from the rinse container.
5. Rinse the sample with DI water in the hood sink.

Sample Dry

1. After the water rinse is finished, blow the sample dry with the N_2 gun.
2. After getting most of the water off, you can dry the samples more in an oven or on a hotplate if allowable for your sample.

Cleanup

1. The etchant may be used for multiple etches. For temporary storage (<1 day), place the top of the Petri dish over the etchant and store on fab wipes in the back of the hood. Make sure the dish is clearly labeled “HF/Nitric/Acetic”.
2. Dump the etchant waste into the carboy designated for HF acid.
3. Rinse the container once with DI water, and dump it into the same carboy.
4. Rinse the beakers used for measuring with DI water and dump them into the HF or acids carboy depending on what they were used to measure.
5. Pour the rinse water containers into the HF acid carboy.
6. Rinse all the containers again with DI water in the hood sink.
7. Return all labware to its proper location. The containers can drip dry on fab wipes in the hood; however, remember to move them back to their storage location once dry.
8. Wipe up any drips in the area with chemical wipes and dispose in the trash.
9. Store the HF in the HF cabinet, the Nitric Acid in the Nitric Acid cabinet and the Acetic Acid in the Acids cabinet.
10. Inspect all of the PPE to ensure it did not come in contact with the etchant before returning it to its storage location.

Accident Procedure

Hydrofluoric Acid Contact

- Skin: Rinse affected area with water for 5 minutes, removing contaminated clothing during rinse. Apply generous amounts of calcium gluconate gel to the area. **Get immediate medical attention.**
- Eye: Immediately flush with water for at least 20 minutes while lifting upper and lower eyelids occasionally. Do not apply calcium gluconate. **Get immediate medical attention.**
- Ingestion: Do not induce vomiting. **Get immediate medical attention.**
- 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.**

Nitric Acid and Acetic Acid Contact

- Skin: Rinse affected area with water for 5 minutes, removing contaminated clothing during rinse. **If there is a visible burn, get immediate medical attention.**
- Eye: Immediately flush with water for at least 20 minutes while lifting upper and lower eyelids occasionally. **Get immediate medical attention.**
- Ingestion: Do not induce vomiting. **Get immediate medical attention.**
- 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.**

Spills

If a small, contained spill occurs, such as inside the hood, wipe it up with chemical wipes and dispose of them in the proper trash container. If a large spill occurs, evacuate the area and notify the cleanroom staff.

Revision History: