KOH Si Wet Etch Review

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1. INTRODUCTION

Potassium Hydroxide (KOH) is an alkali hydroxide used in an anisotropic wet-etch technique and is one of the most commonly used silicon etch chemistries for micromachining silicon wafers. Anisotropic etches attack the substrate preferentially. That is, they etch at a higher rate in certain directions than they do in others, whereas isotropic etches (such as HF) attack in all directions. The KOH process is used because of its repeatability and uniformity in fabrication while maintaining a low production cost [1]. Isopropanol (IPA) is often added to the solution to change selectivity from the {110} walls to the {100} walls and improving surface smoothness.

Both oxide and nitride etch slowly in KOH. Oxide can be used as an etch mask for short periods in a KOH etch bath. For longer periods, nitride is a better mask since it etches much more slowly in KOH. Another alternative (and useful for many other functions) is doping the wafer with boron. It will also reduce etch rate, in effect stopping the etching of the boron rich silicon.

Etch rate increases as KOH concentration increases for concentrations up to 18wt% at 80°C. As KOH concentration increases over 18wt%, etch rate decreases. The chemical reactions that take place during etching are the result of this change. This is discussed more in depth later. Concentration of maximum etch rate is not typically used, however, because surface smoothness improves as concentrations increase (noticeably above 30%). The etch rate of Si{100} in KOH can be calculated using the following equation:

 $R = [H_2O]^4 [KOH]^{1/4} exp(-E_A/kT)$ Equation 1: {100} Etch Rate

Where concentration is in mol/liter, $k0 = 413 \mu m/min * (mol/liter)^{-4.25}$, and $E_A = 0.595 \text{ eV}$. The density and molecular weight of KOH are 2.055 g/cm³ and 56.11 g/mol, respectively, and 1.00 g/cm³ and 18.02 g/mol for water.

There are a few disadvantages to using KOH etching. The most problematic is the creation of H_2 gas bubbles during etching. These H_2 bubbles act as a pseudomask. This increases roughness and potentially damages microstructures. Another issue associated with KOH etching is the fact that KOH contains alkali ions. KOH is a lifetime killer for MOS devices. It is essential that those using KOH be careful not to contaminate any other processes. KOH also etches aluminum, which can be a concern for on-chip circuitry [10][11].

2. Basic KOH Etching Characteristics

I. Anisotropic KOH Etch Rates versus Orientation

The etch rate of a KOH etch is heavily affected by the crystallographic orientation of silicon. This is because of its anisotropic nature. The following table relates the silicon orientation with KOH concentration at 70°C. Table 1 is taken directly from [2], which is also given in [1]. The numbers in parentheses are the normalized values relative to (110).

Crystallographic	Rates at different KOH Concentration			
Orientation	30%	40%	50%	
(100)	0.797 (0.548)	0.599 (0.463)	0.539 (0.619)	
(110)	1.455 (1.000)	1.294 (1.000)	0.870 (1.000)	
(210)	1.561 (1.072)	1.233 (0.953)	0.959 (1.103)	
(211)	1.319 (0.906)	0.950 (0.734)	0.621 (0.714)	
(221)	0.714 (0.491)	0.544 (0.420)	0.322 (0.371)	
(310)	1.456 (1.000)	1.088 (0.841)	0.757 (0.871)	
(311)	1.436 (0.987)	1.067 (0.824)	0.746 (0.858)	
(320)	1.543 (1.060)	1.287 (0.995)	1.013 (1.165)	
(331)	1.160 (0.797)	0.800 (0.619)	0.489 (0.563)	
(530)	1.556 (1.069)	1.280 (0.989)	1.033 (1.188)	
(540)	1.512 (1.039)	1.287 (0.994)	0.914 (1.051)	
(111)	0.005 (0.004)	0.009 (0.007)	0.009 (0.010)	

 Table 1: Etch Rate of Silicon Depending on Crystallographic Orientation and KOH Concentration [2]

The (110) plane is the fasted etching primary surface and the (111) plane is by far the slowest etching plane. This is believed to be due to the ideal (110) surface having a more uneven atomic structure than the (100) and (111) primary surfaces; whereas the (111) is tightly packed, having only a single dangling-bond per atom and is overall atomically flat [2].

II. KOH Etch Rates vs. Composition and Temperature

Etch rates increase as temperature increases; however less ideal etch behavior results as etch rates increase. Etch rates decrease as KOH concentration is increased over 18wt%. This is because of the etching reactions that take place. Silicon-silicon bonds are broken when a strong base, such as KOH (which has an abundance of OH⁻ ions), is present. This etching results in the formation of Si(OH)₄, the consumption of four water molecules, and the release of two hydrogen gas molecules. As the amount of water in the solution is lowered, the reaction becomes limited [11]. Below is a table taken from [1] with silicon orientation-dependant etch rates related to percent composition, temperature, and orientation.

Etchant	Temperature (°C)	Direction (plane)	Etch rate (µm min-1)	Remarks
200/ 1/011	20	(100)	0.025	Neer Deels atch rate at the
20% KOH:	20	(100)	0.023	Near Peak etch rate at the
80% H2O	40	(100)	0.100	conc. across temperature
	60	(100)	0.45	
	80	(100)	1.4	
	100	(100)	4.1	
30% KOH:	20	(100)	0.024	Smoother surfaces than at
70% H2O	40	(100)	0.108	lower concentration
	60	(100)	0.41	Faster etch rate for (110)
	80	(100)	1.3	than for (100)
	100	(100)	3.8	
	20	(110)	0.035	
	40	(110)	0.16	
	60	(110)	0.62	
	80	(110)	2.0	
	100	(110)	5.8	
40% KOH:	20	(100)	0.020	
60% H2O	40	(100)	0.088	
	60	(100)	0.33	
	80	(100)	1.1	
	100	(100)	3.1	
20% KOH:	20	(100)	0.015	Lower etch rate
80% 4 H2O:	40	(100)	0.071	Smoother
1 IPA)	60	(100)	0.28	Less undercutting
	80	(100)	0.96	Lower (100) : (111)
	100	(100)	2.9	etch-rate ratio
44% KOH:	120	(100)	5.8	High Temperature
56% H2O		(110)	11.7	0 1
_		(111)	0.02	
23.4% KOH:	80	(100)	1.0	Sensitive to boron
63.3% H2O:		(110)	0.06	concentration
13.3% IPA				
	1			

Table 2: Silicon Orientation-Dependant Etch Rates of KOH to Percent Compositions, Temperature, and Orientation [1]

III. KOH

Masking

Rectangular openings aligned with <110> directions in the plane of a wafer on (100) silicon results in the exposed $\{100\}$ planes etching rapidly while the $\{111\}$ planes etch slowly. As a result, the $\{111\}$ plane becomes revealed, making a 54.7° angle with the plane of the wafer. As the etch continues, the $\{111\}$ planes will eventually meet, creating a 'V' groove. The $\{111\}$ etch is not zero, so some undercutting does occur. A square mask aligned similarly to the rectangular mask will result in a pyramidal pit.

Another characteristic of KOH anisotropic etching is the result of a convex corner on a mask. When a mask has convex corners, the {111} planes that make up the corners are etched away. If this etch is allowed to go to completion, the masking layer is completely undercut and the mask is left hanging over a 'V' groove pit.

According to Senturia, a likely explanation for this is that {111} planes typically have a single dangling surfaceatom bond. At a corner where two {111} planes meet, two dangling bonds must be present. These are etched away, resulting in other fast-etching planes to be exposed.

This undercutting characteristic occurs with other shapes as well. Senturia gives the example of an ellipse. Etching an elliptical mask results in a rectangular v-groove pit whose edges are aligned to <110> directions. Because of this undercutting, many issues regarding mask alignment can occur. A very small rotational error can cause features to be undercut and greatly enlarged [9]. Further information can also be found in [10].

Three masking materials are commonly used with KOH etching – Silicon Nitride, Silicon Dioxide, and Boron doped (P+) silicon. Silicon Nitride hardly etches at all in KOH, Silicon Dioxide etches at a rate of approximately 1.4 nm/min in 20wt% KOH at 85°C, and p+ silicon gives between a 10:1 and 100:1 etch reduction depending on etchant concentrations and temperature.

3. LITERATURE

ARTICLE 1: Techniques for Micromachining Multilayered Structures in Silicon

Vertical walls using KOH+IPA and multilayered structures in (100) Silicon

The formation of consistent vertical sidewalls using a KOH+IPA solution has been shown to be unreliable. In this report, reliable vertical sidewalls using a high enough KOH concentration and temperature were achieved. The authors used KOH concentrations of 50% and a temperature of 80°C with reliable selection. The goal of the experiment was to create a structure in the shape of a house. The sloping roof was made from {110} planes and the vertical walls. The roof had to be made smaller due to only having one available mask pattern. This resulted in the discovery of a novel corner compensation technique using a maskless etching technique and KOH+IPA at high KOH concentration. After 15 minutes in a KOH bath using maskless etching, the rounded corners of the roof caused by the high order planes undercutting the mask disappeared. This step did increase surface texturing, however. This technique is further discussed in Article 2 ([3]) [7].



Structures after each processing step: 1. Photoresist and SiO₂ mask, 2. Etching 45" walls, 3. Maskless etching, 4. Repeated mask (extended beyond roof), 5. Etching vertical walls Figure 1: Construction of Microhouse [3]

ARTICLE 2: Anisotropic Etching of {100} and {110} Planes in (100) Silicon

Etch selection, surface smoothness, and corner compensation

Anisotropic etching of (100) silicon can produce vertical {100} walls or sloping {100} and {110} walls, inclined at 45° or 54° respectively. To achieve these walls, the pattern edges must be in the <100> direction, 45° from the prime wafer flat. For {111} walls, the structure must be aligned to the <110>. These examples are shown in Figure 1 from [3]. The selection of the wall depends on relative etch rates \mathbf{R}_{100} and \mathbf{R}_{110} . The plane forming the sidewall is always the one with the slowest etch rate aligned to the mask edge.

Pure aqueous solutions of KOH form vertical walls ($\mathbf{R}_{100} < \mathbf{R}_{110}$), while solutions with isopropyl alcohol added form {110} walls ($\mathbf{R}_{110} < \mathbf{R}_{100}$). According to [3], the IPA plays no role in the chemical reaction, however at most concentrations it reduces the etch rates of the high-order crystal planes (reversing the order of \mathbf{R}_{100} and \mathbf{R}_{110}). At high concentrations, the \mathbf{R}_{100} plane remained the slowest.

At 50+% KOH concentrations in KOH+IPA solutions, structures with vertical walls formed. At 50% KOH concentration, 700mn/min etch rates were achieved. It was found that stirring influenced what concentration the transition from sloping to vertical walls occurred, with more stirring lowering the transition concentration.

Texturing of the etched surfaces was also looked at. Six samples etched at 75°C for either 20 or 40 minutes (3 in KOH, 3 in KOH+IPA) were used. Low concentration KOH in KOH+IPA resulted in inclined 45° {110} walls that were rough and formed parallel grooves. Tetrahedral pyramids also covered the wafer surface. Smoother surfaces were observed using concentrations close to 35%. {100} walls produced with pure KOH at low concentrations had very rough surfaces. Higher concentrations had better surface smoothness, but were still not as good as those produced with IPA added. Using 50% KOH, the KOH+IPA solution produced vertical walls. Figure 2 compares the results of 50% KOH and KOH+IPA etched features.



Figure 2: Comparison of Etch Samples [3]

A novel maskless etching technique for corner compensation of {110} bound structures was discovered. High-order fast-etching planes form the corners of island-type structures. These corners, when etched in KOH+IPA at low KOH concentration, were undercut by either {221} or {331} crystal groups. Square corners were achieved on these {110} bound structures using a second etch step after removal of the mask. Using maskless etching, the fast etching planes determine the shape of the structure. Using a solution that etches ($\mathbf{R}_{110} < \mathbf{R}_{100}$) resulted in the structure being etched inwards, leaving the height and incline of walls unchanged while causing the rounded corners to disappear. This step did increase surface roughness, however, and a solution to minimize this must be found [3].



Figure 3: Corner Compensation Technique [3]

ARTICLE 3: Simulation of Two-Dimensional Etch Profile of Silicon during Orientationdependent Anisotropic Etching

Orientation dependence and smooth, round etch profiles

Orientation dependence in chemical anisotropic etching of single-crystal silicon was evaluated. Etch rates for various crystallographic orientations were measured under a range of etching conditions (KOH concentrations from 30 to 50% and temperatures from 40 to 90°C). Depending on the etchant temperature and concentration of KOH, orientation dependence varies greatly. The resulting data allows numerical prediction of etch profiles in silicon. Using multistep chemical anisotropic etching, three-dimensional microstructures on single-crystal silicon can be achieved. This work was performed by one of the same authors of [2], which provides a much more thorough analysis of etch rate dependence [4].

ARTICLE 4: Simulation of Three-Dimensional Etch Profile of Silicon during Orientationdependent Anisotropic Etching

Orientation dependence and smooth, round etch profiles

This article expands upon the preceding article written by the same authors. The authors have developed a three-dimensional profile simulator that is able to determine the shape of a wafer given initial orientation and three-dimensional shape. The authors have also developed two processes for the creation of microstructures. The authors called the first a "multi-step anisotropic etching process" and the second a "separated anisotropic etching process."

The three-dimensional etch profile simulator was generated through experimentation using a concave hemispherical sample that was ground and polished. The radius of the hemisphere was 25mm and the sample was 35mm thick and 76mm on a side. Etching was performed using various etch conditions. The samples were etched in KOH with concentrations of 35, 40, and 45wt% or TMAH with a concentration of 25wt% and temperatures of 40°C-80°C. The authors created the database for the simulation by mapping both the activation energy and the coefficient of the Arrhenius equation as a function of the crystallographic orientation. This was applied to what is called the Wulff-Jaccodine graphing method. Details of how this method works are found in the article. The simulation was tested against the results of experimental etches. It was shown that the simulation predicted the actual results very well.



Comparison of experimental and predicted etch profiles Figure 4: Etch Profiles [12]

Once the simulation was proven effective, two new etching processes were developed. The first, a multistep anisotropic etching process, proved capable of creating round etch profiles and sloping etch profiles. This round profile was created on a cantilever using a two-step process. The sloping etch profile was created for the electrode of a diaphragm pump actuated by electrostatic force. This sloping electrode proved a large electrostatic force. The second process to be developed was a separated anisotropic etch. The authors were able to create a three-dimensional separation structure. An impeller structure, suspended from the wafer by eight beams, was created after 280 minutes of etching. The simulation system and the two new etch process allow many new three-dimensional structures to be developed [12].



Figure 5: Examples of New Etch Techniques [12]

ARTICLE 5: Characterization of Orientation-dependent Etching Properties of Single-Crystal Silicon: Effects of KOH Concentration KOH concentration

Etch rates for various crystallographic orientations have been measured for a range of etching conditions. KOH concentrations of 30 to 50% and temperatures of 40 to 90°C were examined. All etchants consisted of KOH and water. This report's goal was to create an etching simulator in order to satisfy the needs of

designing the multiple steps of a KOH etching process. Complex structures require the knowledge of etch rates for all orientations of the structure. In order to optimize the amount of data collected, it was decided that a hemispherical single-crystal silicon substrate would be best. The authors calculated that a hemisphere of radius 22 µm would be adequate for minimal interference from neighboring planes. The top of the hemisphere was oriented in the (110) direction. Maximum etch depth was limited to 100-150 µm in order to prevent this interference. After etching, the sphere was probed at 2° increments from 20-90° all the way around the sphere (8500 points total). As a result, the etch rates of a dense net of orientations were evaluated [2].

ARTICLE 6: Fast Wet Anisotropic Etching of Si{100} and {110} with a Smooth Surface in Ultra-**High Temperature KOH Solutions**

High etching rates and surface roughness

KOH solutions with temperatures near its boiling point were analyzed. Using p-type {110} and {100} wafers, Plasma-CVD Silicon Nitride was deposited and used as an etch mask. Etching was performed using temperatures between 80 and 145°C and KOH concentrations of 10-50wt%. IN order to get the Etchant as close to boiling as possible without it actually boiling during etching, the boiling points for various concentrations were tested. Once determined, tests were performed examining the various KOH concentrations from 80° until the boiling point for the respective solution for both $Si\{100\}$ and $Si\{110\}$ wafers.

Smooth surfaces and ultra-high etch rates were obtained. The etching rates of Si{100} near the boiling point were 5-9 times higher than at 80°C in concentrations of more than 32wt%. Si{110} etch rates were 4-20 times higher. Using an Arrhenius plot, etch rates were nearly linear from 80°C to near the boiling point. This shows that the activation process in the higher temperature is the same as that in the lower temperature.

The smooth surfaces obtained for $Si\{100\}$ samples were etched near the boiling temperature above about 30wt% KOH. The surface smoothness is very similar at 80°C in 32wt% KOH and at 145°C in 50wt% KOH. The {110} samples were covered in long zigzag shapes with a concentration below 40wt%, but decreased as this concentration was approached. As KOH concentration increased beyond 40wt%, the surfaces became dominated by hillocks up until about 120°C, with the hillocks gradually disappearing with an increase in etching temperature. At near the boiling point 45-47wt% KOH, the authors got very smooth surfaces with the ultra high etching rate of $20 \,\mu$ m/min [5].



Figure 6: Si{100} – Etch Rate and Surface Smoothness [5]



ARTICLE 7: Micropyramidal Hillocks on KOH Etched {100} Silicon Surfaces: Formation, Prevention and Removal

Formation of micropyramidal hillocks are an unwanted occurrence of micromachining. Smooth and pyramid free surfaces are wanted. A number of possibilities for the formation of hillocks have been theorized. Some have suggested that the pyramids are a result of H_2 bubbles, SiO₂ precipitates, or reaction products acting as pseudomasks. The goal of this report is to find more detailed answers (and solutions) to the formation of micropyramidal hillocks.

Micropyramids created by KOH etching are typically of an exact shape. The two types have either rectangular or octagonal bases. The edges of rectangular hillocks are always aligned with each other and parallel to the <100> crystal directions. They are also sometimes partly entangled. The octagonal micropyramids always have a larger height-to-base ratio than the rectangular pyramids and are <110> oriented. No pyramids were found transitioning between the rectangular and octagonal shapes, but both were found on the same samples.

In order to examine the belief that SiO_2 precipitates can act as a pseudomask, different wafer types were etched. There was no significant difference in any wafer concerning area density and size of the micropyramids, leading the authors to believe that SiO_2 precipitates cause the formation of the pyramids. The authors believe this is the most probable theory regarding micropyramid formation. The majority of their data as well as that of others agree with this theory, however a problem with this was found. If the formation of the micropyramids were started by SiO_2 precipitates, then the various wafer types should result in significant differences in their formation. This was not so, according to the results.





SEM micrograph of large rectangular (partly enmeshed together) and octagonal micropyramids (0.33 g g^{-1} KOH/H₂O, 65 °C).

SEM micrograph of a peak with a micromask residue.





Micrograph of rectangular micropyramids in curved arrangements with some octagonal micropyramids (arrows) $(0.33 \text{ g g}^{-1} \text{ KOH/H}_2\text{O}, 65 ^{\circ}\text{C}).$ Micrograph of the same chip area as figure 8 after a 1 min re-etching in the same etch bath under the same conditions $(0.33 \text{ g g}^{-1} \text{ KOH/H}_2\text{O}, 65 ^{\circ}\text{C}).$

Figure 8: Micropyramid Formation [6]

The result of H_2 bubble formation was found to be responsible for the rectangular pyramids that followed a curved pattern. This pattern was independent of the wafer material, but depends on the etching conditions. Low KOH concentration and temperature resulted in the curve radii to become enlarged and closed circles to be found. At these conditions, relatively large bubbles occur and detach after a long enough period to act as a pseudomask. As KOH concentration and temperature are increased, the size of the bubbles and the time they remain attached to the substrate are reduced. It is the authors' belief that micropyramids are primarily caused by layer-by-layer etching, or 'peeling', of the {111} planes in connection with plateau generation due to H_2 bubbling.



Normarski micrograph of rectangular micropyramids in curved arrangements (0.25 g g⁻¹ KOH/H₂O, 60 °C). The starting points of complete H₂-bubble pattern and the direction of bubble movement is indicated by arrows. Figure 9: H₂-bubble patterning [6]

Several methods to prevent the formation of micropyramids were examined. High temperature and high KOH concentration baths produced surfaces that were almost micropyramid free. A short re-etching in the same etch bath and the same conditions resulted in a marked reduction of the size of the micropyramids. A short 1 minute re-etch resulted in a substantial removal of the micropyramids. A 9 minute re-etch almost completely removed the micropyramids [6].



 $\label{eq:micrograph} \begin{array}{l} \mbox{Micrograph of rectangular micropyramids in curved} \\ \mbox{arrangements with one octagonal micropyramid (arrow)} \\ \mbox{(0.33 g g^{-1} KOH/H_2O, 65 \ ^{\circ}C)}. \end{array}$

Micrograph of the same chip area as figure 10 after a 9 min re-etching in the same etch bath under the same conditions $(0.33 \text{ g g}^{-1} \text{ KOH/H}_2\text{O}, 65 \,^{\circ}\text{C}).$

Figure 10: Micropyramid Removal - Re-etching [6]

ARTICLE 8: Anisotropic Multistep Etch Processes of Silicon

Anisotropic multistep etching combines a number of etching processes with changes in etchant, etching conditions, and masking. Multistep etches allow for a number of sidewall shapes. The authors experimented with {001} wafers using KOH wet etching and two step processes. Using the software SIMODE, the etching results were simulated and compared with the actual results.

Two types of two-step processing were studied. The first involved back setting the position of the mask edges between steps while using the same etchant. The mask edges were aligned the same for both steps, but tests using either <110> of <100> and KOH or KOH+IPA as etchants. The second process used different etchants for the two steps, but left the mask unchanged. Either KOH was used for the first step and KOH+IPA for the second, or vice versa.

A number of impressive sidewall formations were generated using the multi-step processes. These can all be viewed in [8]. Two figures the authors speak of include a profile in which two sidewalls are left-right combined and another using a different technique, leaving the middle two sidewalls left-right combined and etched to perforation. The authors state that the former "can be used to surround a rectangular membrane reducing the mechanical stress at the edges." It was also stated that the latter "can form springs for bending and torsion."



Measured and simulated profiles of the sidewall shown in figure 1(e). Two sidewalls are left-right combined forming a wall structure.



Measured and simulated profiles of the sidewall shown in figure 4.1(b). In the middle two sidewalls are left–right combined and etched to perforation in the lateral direction producing free-standing bridges. Figure 11: Simulated Multi-step Sidewall Formations [8]



SEM record of a structure of sidewall type 4.1(b) corresponding to the cross section simulation shown in figure 6. The etching was performed at first in 30 wt% KOH at 80 °C for 286 min and then without changing the mask in 35% KOH:IPA at 70 °C for 254 min. The thin vertical wall (thickness between 3.6 and 8.2 μ m) is built from (100) faces, and disappears if etching continues for some minutes more.

Figure 12: Multi-step Sidewall Formation [8]

Upon examination of all specimens, it was found that some faces were very rough or corrugated. This issue could restrict the structure's application of microtechniques. The authors did, however, find that the corrugated {101} faces resulting from etching in KOH+IPA could be smoothed by using tetramethyl ammonium hydroxide and IPA [8].

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