Process Control Challenges of Wet Etching Large MEMS Si Cavities
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Introduction
Anisotropic etching of silicon refers to the directional-dependent etching, usually by alkaline etchants like aqueous KOH, TMAH and other hydroxides like NaOH. With the strong dependence of the etch rate on crystal orientation and on etchant concentration and temperature, a large variety of silicon structures can be fabricated in a highly controllable and reproducible manner. Hence, anisotropic etching of <100> silicon has been a key process in common MEMS based technologies for realizing 3-D structures [1-4]. These structures include V-grooves for transistors, small holes for ink jets and diaphragms for MEMS pressure sensors as shown in Figure 1 [1]. The actual reaction mechanism has not been well understood and comprehensive physical and chemical models for the process have not yet been developed. With increasing numbers of MEMS applications, interest has grown in recent years for process modelling, simulation and software tools useful for the prediction of etched surface profiles [4-6].

In this study, test wafers were processed in dilute TMAH in order to etch deep cavities similar to the ones shown below in Figure 1. Due to the large surface areas being etched from the wafers’ surface, etch byproducts presented a challenge to the process control that produced undesired results. In-line sensors were installed to monitor and control the chemical’s concentration and mass of etch byproducts in real-time. Algorithms were developed to offset the effect of these factors and produced consistent results. The purpose was also to lower the overall cost of ownership (COO) of the process.

![Figure 1: Typical structures formed during alkaline Si etching: a) deep v-groove, b) shallow v-groove and c) silicon etch with undercutting, d) suspended cantilevel and e) mask pattern](image)

Experimental
Wet chemical processes were conducted on a fully-automated GAMA™ wet processing station using 200mm wafers with a typical cavity structure. TMAH was used as the alkaline etchant at a concentration suitable for achieving a maximum etch rate. Silicon etching processes were conducted with the aid of Akrion Systems’ in-situ chemical concentration control system. Occasionally, samples of the baths were taken and titrated for comparison to the NIR results. The goal was to fully etch through the wafers by maintaining a consistent etch rate throughout the entire process.

Results and Discussion
Silicon Etch Rates, Theory vs. Experiment. The chemical reaction for the anisotropic alkaline etching of silicon is well known and a variety of etchants can be used for the process (KOH, NaOH, TMAH). However, CMOS fabs typically cannot use metal hydroxides as the metal ions will
contaminate the fab and damage the devices. As a result, TMAH is often used when the metal hydroxides are not allowed.

The overall reaction mechanism for the etching of silicon with TMAH is shown in equation 1. Based on this reaction, for every 1 mole of silicon etched, 2 moles of TMAH are consumed. Using this theory, a test was conducted with an eleven wafer batch load. The theoretical consumption of TMAH was compared to the actual consumption based on both NIR and titration data. For the purpose of this study, the starting setpoint concentration of TMAH was 5% and allowed to drop to a minimum level. The lower level was selected and then maintained at 3% where the etch rate was kept constant.

\[
\text{Si + 2TMAH + 2H}_2\text{O} \rightarrow \text{SiO}_2(\text{OH})_2^{2-} + 2\text{TMA}^+ + 2\text{H}_2
\]

The results, shown below in Figure 2, reveal that the actual TMAH consumption is much less than predicted. According to theory, the minimum level should have been reached after about 260 minutes. However, it wasn’t reached until about 540 minutes into the process, about 2x the time predicted by the theory.

As a result, a new theory was developed that fit the data much more accurately. The new theory supposes that only 1 mole of TMAH is consumed per 1 mole of Si, as opposed to the original theory of 2:1 (TMAH:Si, based on equation 1). The test was run again and the results are shown in Figure 3. As can be seen, the new theory fits the data very closely.

Based on these results, the alkaline etching mechanism shown in equation 1 clearly does not properly describe the reactions taking place on the surface of the silicon. The etching of silicon is a complex reaction involving multiple reaction steps. So while equation 1 assumes that TMAH is the only source of OH\(^{-}\) for the reaction, looking deeper into the mechanism reveals a possible second source.

The alkaline etching of silicon proceeds through both an oxidation and reduction step. In the oxidation step (equation 2), two hydroxide ions bind to the silicon and the resulting silicate complex along with 4 electrons are released. The free electrons then interact with the water molecules near the silicon surface. The result is the reduction of water shown in equation 3. The silicate complex is then further reacted in equation 4 to form a water soluble complex. The overall chemical reaction is shown in equation 5.

\[
\begin{align*}
\text{oxidation step:} & \quad \text{Si} + 2\text{OH}^{-} \rightarrow \text{Si(OH)}_2^{2+} + 4\text{e}^{-} \\
\text{reduction step:} & \quad 4\text{H}_2\text{O} + 4\text{e}^{-} \rightarrow 4\text{OH}^{-} + 2\text{H}_2 \\
\text{silicate dissolution:} & \quad \text{Si(OH)}_2^{2+} + 4\text{OH}^{-} \rightarrow \text{SiO}_2(\text{OH})_2^{2-} + 2\text{H}_2\text{O} \\
\text{overall reaction:} & \quad \text{Si} + 2\text{OH}^{-} + 2\text{H}_2\text{O} \rightarrow \text{SiO}_2(\text{OH})_2^{2-} + 2\text{H}_2
\end{align*}
\]
etch rate is related to the fourth power of the water concentration and only ¼ power of the TMAH concentration (equation 6) [6]. This may help to explain why the actual results obtained show that only 1 mole of TMAH is consumed per mole of silicon, as the other OH\(^-\) may actually be provided from the reduction of water.

\[
R = k_0[H_2O]^4[TMAH]^{1/4}e^{-E_a/kT}
\] (6)

**Factors Affecting Silicon Etch Rates.** When etching a large amount of silicon as in this study, the effect of H\(_2\) gas evolution on the etch rate and surface uniformity is a concern. As described previously, hydrogen gas is a by-product of the etching mechanism. As the bubbles are generated in solution, they will stick to the wafer surface and prevent chemical from reaching the silicon, essentially stopping the etching process. As a result, surface modifiers such as surfactants or IPA are sometimes added to the solution. For the purposes of this study, no additives were used so the gas generation was of initial concern.

To determine the effect, if any, on the overall etch rate, various wafer loads were tested in order to generate different amounts of H\(_2\) bubbles in solution. The etch rates were determined after 5 hours and the results in Figure 4 show equivalent etch rates with the different load sizes.

![Figure 4: Silicon Etch Rates versus Wafer Load for H\(_2\) Generation](image)

However, even though the etch rates were consistent, it was discovered that the bubbles did generate localized etch patterns, as can be seen in Figures 5a & b. As the bubbles attached to the surface, they masked the etching in that area before eventually dislodging. This resulted in raised feature as that shown in Figure 5b since the area surrounding the bubble still continued to etch. This uneven etching pattern can be problematic for MEMS technology. As a result, Akrion has developed a propriety process that results in a more uniform etch by preventing the H\(_2\) bubbles from sticking to the surface without the use of additives (Figure 5c).

![Figure 5: Si wafer surface: a) w/o propriety process b) w/o proprietary process, raised Si pattern from H\(_2\) bubble on surface and c) with propriety etch process](image)

Another item of concern that can affect the silicon etch rate is the increased amount of silicates in solution over time. In the solar industry, silicate buildup has been known to affect the texturization process, so concentration monitoring and control is crucial [7]. However, even though complete pyramid formation is not a concern in this process, a stable etch rate is still required. As a result, the bath was loaded with different amounts of silicon and etch rates measured after 5 hours. Figure 6 shows no significant change in etch rate with increased silicate level.

![Figure 6: Silicon Loading versus Etch Rate](image)
Computational Fluid Dynamics (CFD), Theory vs. Experiment. Through-silicon etch of large cavities can result in a unique fluid flow pattern within the features as they are etched to greater depths during the process. To better understand the fluid dynamics within the cavities, CFD calculations were performed based on the current test setup. The calculations were performed based on two different etch depths: 300µm and 450µm. The results in Figure 7 show a laminar flow across the surface at both etch depths.

![Figure 7: CFD model for current test setup: a) schematic of possible flows, b) 300µm etch depth and c) 450µm etch depth](image)

Results on actual test wafers however, show a clear difference in appearance as the etch depth increases. Figure 8a shows a smooth, even appearance at an etch depth of 250µm, while 9b shows a horseshoe shape pattern at 500µm. These results differ from the CFD model and emphasize the dynamic nature of the flow inside these types of structures. Clearly, as the etch depth increases local vortices develop within the cavity which affects the uniformity of the etch. In order to minimize this effect, Akrion has developed a proprietary in-process method that allows for a more even flow across the wafer surface and within the cavities. The result is a more uniform etch process.

![Figure 8: Top-down pictures of cavities at various etch depths: 1) 250µm and b) 500µm](image)

Conclusion
Results have shown that Akrion Systems’ propriety wafer process produces a uniform etch by preventing the H₂ bubbles from sticking to the surface regardless of the amount of H₂ gas generated during the process. However, it was discovered that as the etch depth increased, the flow dynamics within the cavities began to play an important role, as was evidenced by a change in appearance of the silicon surface in these cavities. These localized flow patterns can be minimized by the use of a proprietary method developed by Akrion. The use of a closed loop concentration control system also allowed for the minimum use of TMAH while still maintaining the desired etch rate. This reduces the amount of chemical sent to the waste stream, effectively decreasing the overall cost of ownership for the process.

References

