

# Advanced Chemical Concentration Control for Fabrication of Devices Using SiC

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## Abstract

In conventional MEMS fabrication, relatively inert compounds such as  $\text{Si}_3\text{N}_4$  are used as an etch stop or mask for creating patterns on wafers. However, materials such as this require insight as to their etch selectivity corresponding to that of the substrate material and are not suitable for high temperature devices. When developing these high temperature compatible devices, components composed of SiC are desired, and may be used as an etch stop due to it being chemically inert. For these applications, it is common for the substrate or sacrificial layer to be either Si or  $\text{SiO}_2$ . A technique for advanced chemical concentration control during processing is critical to be able to maintain a consistent etch rate, a controlled etch depth, and maintain the desired shape of the pattern. Using NIR technology it is possible to monitor both the concentration of chemicals in the bath as well as that of byproducts created from the etching of Si and  $\text{SiO}_2$ . The system can then increase bath life and the ability to etch consistently within and across batches. In the present paper, we present the mechanism of the advanced concentration control, the results of using either TMAH or KOH to etch Si, as well as its applications for the future of SiC integrated devices.

## Introduction

For the last 20 years, SiC has been investigated as a replacement for traditional etch stops in bulk micromachining due to its properties of being chemically inert in conventional etching solutions.<sup>1</sup> It is also a desired material for high temperature devices due to its thermal properties. Applications for creating such devices as fuel atomizers, pressure sensors, and microfabricated molds can use typical wet etching techniques and take advantage of the properties of the SiC layer which make it chemically resistant.<sup>1</sup> Particularly for SiC-MEMS devices, large area substrates are essential. Due to the difficulty in manufacturing single growth crystal substrates, there has been much interest in epitaxial growth of single and poly-crystalline SiC layers on silicon. After the deposition, bulk etching is able to create microstructures and patterns suitable for the desired devices.

With bulk etching, one major concern is that the byproducts are released from the wafer into the bath. Depending on the open surface area of the patterned wafer, large amounts of silicates can be introduced. This can then create unwanted side effects such as decreased etch rate. By creating stable chemical conditions inside of the bath, it not only allows for consistent etch depth within and between lots, but it also allows for increased bath life which will lessen the cost of ownership for the process.

Naura-Akrion has developed a solution to the problem of consistent chemical control. The novel system tracks the concentration bands allowed within the process using NIR

technology. When the concentration breaches those bands, a small volume will drain from the tank and the same volume of fresh chemical will be added to maintain the purity and concentration in the bath. These parameters are completely customizable to each process and situation, allowing for a wide variety of possible usage.

## 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 an alkaline etchant at a concentration suitable for achieving a maximum etch rate. KOH was used as an alkaline etchant during subsequent testing on Solar wafers following the same methods outlined. Silicon etching processes were conducted with the aid of Naura-Akrion's in-situ chemical concentration control system. Occasionally, samples of the baths were taken and titrated for comparison to the system's readings. The goal for the TMAH testing was to fully etch through the wafers by maintaining a consistent etch rate throughout the entire process. For the KOH testing, a consistent etch rate and reflectance were measured to ensure the versatility of the system.

## Results and Discussion

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). For the purpose of this study, the starting setpoint concentration of TMAH was 5% and allowed to drop to a minimum level of 3% whereafter the etch rate was kept constant. Through the course of the experiment, it was discovered that the generally held overall reaction mechanism for the etching of silicon with TMAH, as is shown in equation 1, did not match the real data.

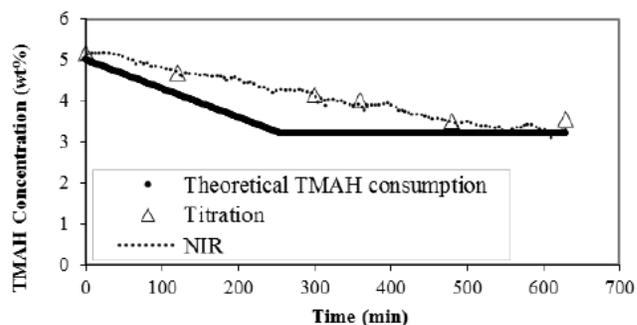
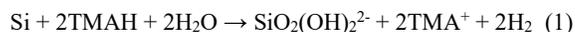
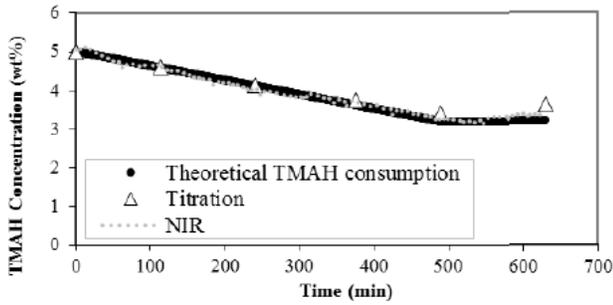


Figure 1. TMAH Concentration Model (2:1) vs Practice

As a result, a new theory was developed which fits 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 2. As can be seen, the new theory fits the data very closely. It can also be seen from Figure 1 that after the TMAH concentration hit the lower limit of 3%, the system initiated the steps of draining and refilling with fresh chemical, which in turn allows the concentration to maintain steady even during processing.

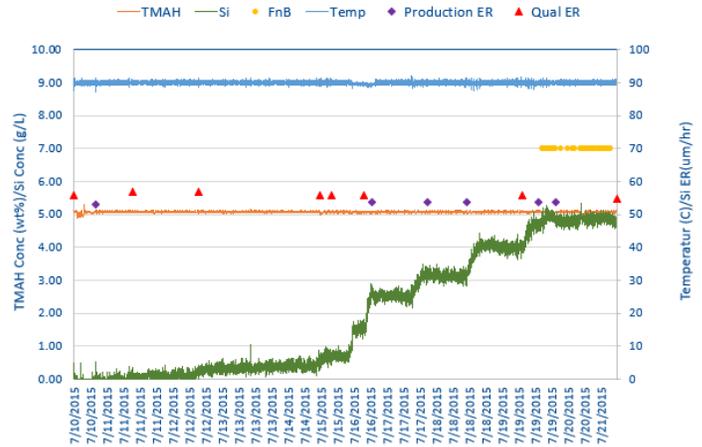


**Figure 2.** New TMAH Concentration Mode (1:1) vs Practice

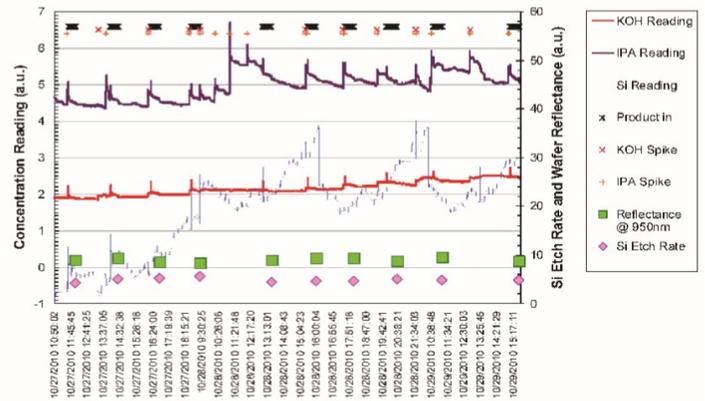
As can be seen in Figure 3, when the system initiates the concentration control process, the bath is then left at a steady state position. Therefore, it is important to set all parameters for upper and lower bounds to allowable values for maintained chemical concentration. In Figure 3, when the Si concentration reaches 5 g/L, the balanced volume of chemical being drained from the tank and added to the bath allows the concentration to stay at 5 g/L. The system does not correct itself to lessen the amount of Si in the bath significantly, as this would provide too much disturbance to the process. The TMAH concentration during the process is also maintained at a steady concentration, thus allowing the bath to sustain itself in an equilibrium between TMAH concentration and Si concentration for an extended period of time. The constant concentration of TMAH also allows the system to keep a constant etch rate, which was monitored before and after the system was activated. The production etch rate was the same across the experiment, and the qual etch rate was comparable to that of normal conditions. This dynamic equilibrium can maintain itself for weeks or even months depending on the process and the needs of the fab, as shown in the chart where the data is collected for 11 days with consistent results. A benefit of the SiC etch processes is that the material itself is inert to the normal chemical concentrations used for bulk etching, therefore the models necessary to track the silicate byproducts in the bath for typical bulk Si etching have already been created.

For the KOH process, testing focused on the bath life capabilities and the ability to simultaneously maintain both the base chemical concentration and Si concentrations within their set allowable bands. From Figure 4, over the course of 77 hours the etch rate is held constant due to the steady state conditions of the process. The silicate maximum level was set at 4.2g/L. The Si reading reached the upper limit, and the system responded accordingly and decreased the amount of Si in the bath without compromising the KOH concentration or the etch rate. Due to the adjustable settings, the Si concentration in this case is adjusted to well below the upper limit each time the system corrects itself. Etch rate testing throughout the 77-hour

process indicated a consistent etch rate and reflectance. Both of these metrics represent the steady state of the bath once the chemical concentration monitor system is activated. Using not only KOH to etch but a mixture involving the surfactant IPA has important implications as well for SiC devices. Pressure sensors are being designed which use these two chemicals in various concentrations at high temperatures.<sup>5</sup> The process for designing these pressure sensors involves deposition of 3C-SiC on a bulk substrate of Si which undergoes backside etch. The NIR chemical concentration system is able to control these types of conditions just as well as if there was only one chemical involved.



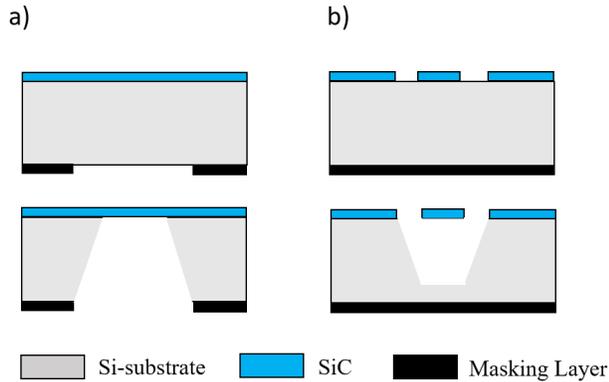
**Figure 3.** Concentration of TMAH and Si in bath with measured etch rates over 11 days



**Figure 4.** Concentration of KOH and Si with measured etch rates across 77 hours

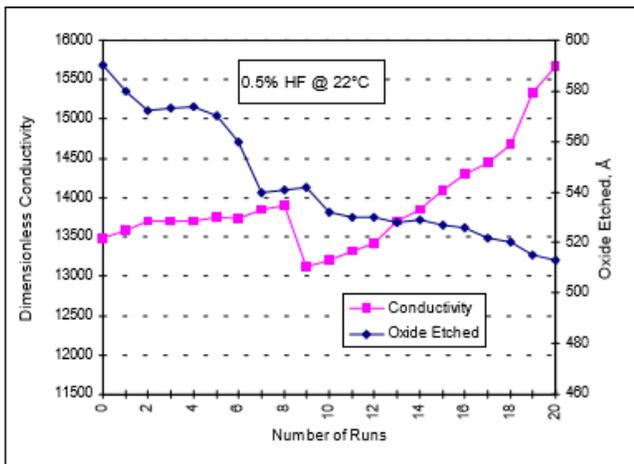
This novel process can be especially useful when a pattern requires a long etch time. Molds to create some SiC devices require an etch depth of up to a few hundred microns, in which case the quantity of byproducts in the bath should be closely monitored so as to not negatively interfere with the etch.<sup>2</sup> This system has been shown to work with through etching 200mm sized wafers of 550um thickness. In this way, this system is capable of handling not only the front-side etching to create and potentially release devices from Si molds, but also the bulk back side etching to release the microstructures as pictured in Figure 5. This process will also aid in situations where oxide is

required as a secondary etch stop for patterning as a constant concentration in the bath will directly relate to a constant selectivity.

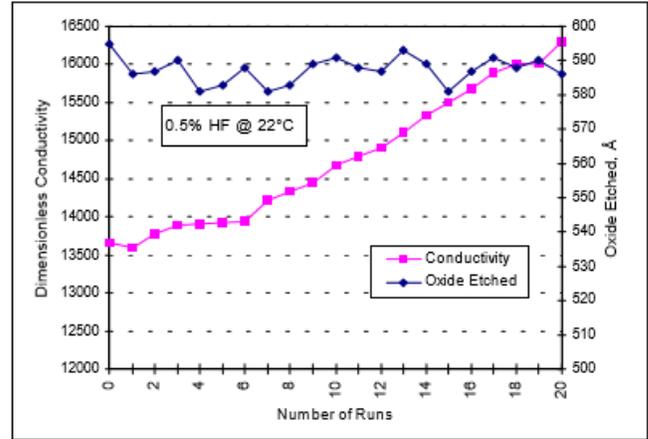


**Figure 5.** View of potential etching of micromachining for: (a) back side and (b) front side etching.<sup>3</sup>

For a similar process to this chemical concentration control system, conductivity may also be used to monitor the amount of chemical that is being used during etching. A prime example of a situation in which this design would be useful is when etching SiO<sub>2</sub> with HF. This is an important step for many SiC devices as the HF etch releases the device from the mold. During this process, etching by-products may affect the linear conductivity-concentration relationship seen for many acids. Therefore, a correction must be developed to correlate the amount of SiO<sub>2</sub> etched to the change in conductivity. Naura-Akrion calls this system of conductivity monitor and control ICE™. Through testing in a production tool, a correlation was found and implemented between the concentration of by-products, specifically H<sub>2</sub>SiF<sub>6</sub>, the concentration of HF, and the conductivity value over 23 runs with 50 oxide wafers. Based on this new formula for the expected conductivity, the setpoint would be renewed after each etching process is completed. Examples of oxide being etched without and then with this concentration control scheme are shown in Figures 6 and 7 respectively. From the figures it is apparent that the adjustment for conductivity from run to run results in a more uniform etch of SiO<sub>2</sub>.



**Figure 6.** Effect of Dissolved SiO<sub>2</sub> on Etch Rate and Bath Conductivity



**Figure 7.** Stability of SiO<sub>2</sub> Etch rate with Modified Conductivity Algorithm

## Conclusions

Results have shown that Naura-Akrion's novel closed loop concentration control system allows for a minimum usage of etchant, such as TMAH or KOH, while still maintaining the desired etch rate. This reduces the amount of chemical sent to the waste stream, decreasing the overall cost of ownership for the process. It also allows for consistent results within lots and between lots for wafer processing, effectively reducing the need to changeout baths after specific number of lots or time, depending on the demands of the process. This not only saves on more chemical cost, but also reduces the total time taken to qualify the baths in the long run after a chemical changeout occurs. The current state of SiC device manufacturing involves deposited films on bulk Si which must be etched. With closed loop concentration control, the process is more robust and results in less costly manufacturing.

## References

- Mehregany, M., Zorman, C., Rajan, N., & Wu, C. H. (1998). Silicon carbide MEMS for harsh environments. *Proceedings of the IEEE*, 86(8), 1594-1609. doi:10.1109/5.704265
- Mehregany, M., & Zorman, C. A. (1999). SiC MEMS: Opportunities and challenges for applications in harsh environments. *Thin Solid Films*, 355-356, 518-524. doi:10.1016/s0257-8972(99)00374-6
- Sarro, P. M. (2000). Silicon carbide as a new MEMS technology. *Sensors and Actuators A: Physical*, 82(1-3), 210-218. doi:10.1016/s0924-4247(99)00335-0
- Kashkoush, I., Rieker, J., Chen, G., & Nemeth, D. (2015). Process Control Challenges of Wet Etching Large MEMS Si Cavities. In *Solid State Phenomena* (Vol. 219, pp. 73-77). Trans Tech Publications.
- Marsi, N., Majlis, B. Y., Mohd-Yasin, F., & Hamzah, A. A. (2014). The fabrication of back etching 3C-SiC-on-Si diaphragm employing KOH + IPA in MEMS capacitive pressure sensor. *Microsystem Technologies*, 21(8), 1651-1661. doi:10.1007/s00542-014-2267-8