

Study of Fast Etching of Silicon for the Fabrication of Bulk Micromachined MEMS Structures

V. Swarnalatha, A. V. Narasimha Rao, P. Pal

Abstract—The present research reports the investigation of fast etching of silicon for the fabrication of microelectromechanical systems (MEMS) structures using silicon wet bulk micromachining. Low concentration tetramethyl-ammonium hydroxide (TMAH) and hydroxylamine (NH₂OH) are used as main etchant and additive, respectively. The concentration of NH₂OH is varied to optimize the composition to achieve best etching characteristics such as high etch rate, significantly high undercutting at convex corner for the fast release of the microstructures from the substrate, and improved etched surface morphology. These etching characteristics are studied on Si{100} and Si{110} wafers as they are most widely used in the fabrication of MEMS structures as wells diode, transistors and integrated circuits.

Keywords—KOH, MEMS, micromachining, silicon, TMAH, wet anisotropic etching.

I. INTRODUCTION

WET anisotropic etching is extensively used in silicon bulk micromachining for the fabrication of micro/nanostructures (e.g. cantilevers, diaphragms, etc.) for the development of MEMS and nanoelectromechanical systems (NEMS) based sensors and actuators [1]-[4]. Although dry etching such as deep reactive ion etching (DRIE), focused ion beam, laser micromachining, etc. is used to fabricate microstructures for applications in MEMS/NEMS, wet etching is the most preferred technique owing to its several benefits such as low cost, ability to fabricate 3D structures with slanted sidewalls, orientation dependent, controllable etch rate with incorporation of additives, high undercutting which is useful to fabricate suspended structures, high selectivity to mask materials, etc. Another major advantage of wet etching is the ability of batch fabrication which makes it the most preferred choice for industrial applications. As a result, wet etching remains an active area of research.

MEMS and NEMS based sensors and actuators usually employ freestanding structures such as cantilever, fixed-fixed beams, diaphragms. Despite having the advantage of batch fabrication, slow etching increases the fabrication time and is a great concern for industries. To minimize the etch time to improve productivity, high etch rate is desirable. In order to fabricate the suspended microstructures with free end such as cantilever, high undercutting is needed to remove the underneath material [1], [5], [6]. Besides, smooth etched

surface morphology is required especially when surface is used for optical application. However, the challenging factor in wet etching has been the low etch rate of commonly used etchants. As a result, attempts have been made to increase the etch rate in wet anisotropic etching. Ultrasonic agitation and microwave irradiation during etching have been used to increase the etch rate [7], [8]. Although these methods increase the etch rate, they have some associated disadvantages. For example, ultrasonic agitation may rupture the fragile structures, while microwave irradiation may damage the microstructures. Another method to improve the etch rate is to increase the etching temperature to the boiling point of the etchant. Tanaka et al. [9] and Tang et al. [10] studied the etching characteristics of KOH and TMAH, respectively, below and above the boiling points of the etchant. Although the etch rate in both studies is increased significantly, the requirement of high temperature is the main drawback. Various kinds of additives such as reducing agent, alcohol, surfactants are also reported to alter the etch characteristics of KOH [11]-[16] and TMAH [17]-[21]. In our previous work, we studied the etching characteristics of Si{100} in various concentrations of NH₂OH-added KOH and TMAH solutions [22], [23]. In this work, we have studied the etching characteristics of various concentration of NH₂OH-added 5% TMAH solution to improve the etching characteristics of Si{100} and Si{110}. TMAH is selected due to its complementary metal-oxide-semiconductor (CMOS) compatibility. The lower concentration is chosen as it exhibits higher etch rate compared to high concentration TMAH.

II. EXPERIMENTAL

4 inch, Cz-grown, Si{100} and Si{110} wafers with a resistivity of 1-10 Ω-cm are used for the experiments. Thermal oxide is used as mask layer. Oxide layer on silicon surface is patterned using photolithography followed by oxide etching in buffered hydrofluoric (BHF) acid. Photoresist used in photolithography process is removed by acetone followed by rinsing in de-ionized (DI) water. Now the wafers are diced into 2x2 cm² size square and rhombus shaped pieces. The silicon samples are then dipped in piranha bath (H₂SO₄: H₂O₂::1:1) for 10 min. In piranha bath, most of the contaminants are dissolved in the solution and a thin oxide layer grows on the silicon surface which dissolves remaining contaminants attached to the silicon surface. This step is followed by DI water rinse. Chemically grown oxide layer is removed by dipping into 1% HF for one minute and finally rinsed with DI water thoroughly. Now the samples are ready for etching experiments. The cleaned samples are placed vertically in a PFA made chip holder containing multiple slots

V. Swarnalatha and A. V. Narasimha Rao are with the Indian Institute of Technology Hyderabad, Kandi Sangareddy, India (e-mail: ph14resch11005@iith.ac.in, ph14resch11003@iith.ac.in).

P. Pal is with the Indian Institute of Technology Hyderabad, Kandi Sangareddy, India (phone: 91-40-2301-6035; e-mail: prem@iith.ac.in).

in order to etch many samples at a time. Thereafter, the samples are dipped in the etchant container which is immersed in the constant temperature bath. 5 wt% TMAH is used as main etchant, while NH_2OH is employed as additive to alter the etching characteristics. The concentration of NH_2OH in TMAH solution is varied from 5-20% in step of 5%. All experiments are performed at 70 ± 1 °C. The etched samples are characterized using 3D measuring laser microscope (OLYMPUS OLS4000), optical microscope (OLYMPUS MM6C-PC), scanning electron microscope (SEM) and spectroscopic ellipsometry (J.A.Woolman Co.Inc).

III. RESULTS

To contemplate the fabrication of MEMS components via wet anisotropic etching using $\text{Si}\{100\}$ and $\text{Si}\{110\}$, different etching characteristics should be known. Out of many etching characteristics, most important characteristics to be measured are etching rate, undercutting at convex corners, and etched surface morphology. In this work, we have investigated the etching characteristics of $\text{Si}\{100\}$ and $\text{Si}\{110\}$ in various concentrations of NH_2OH -added 5% TMAH.

A. Etch Rate

The etch rate of wet anisotropic etchant is a great concern for industrial applications. It measures as etch depth per unit time. In order to improve productivity, it should be high. Fig. 1 shows the etch rate of $\text{Si}\{100\}$ and $\text{Si}\{110\}$ in pure TMAH and with varying concentrations of NH_2OH . It can easily be noticed from Fig. 1 that the etch rate of $\text{Si}\{100\}$ and $\text{Si}\{110\}$ increases more than double when 5% NH_2OH is added to pure TMAH and then increases further by increasing concentration of NH_2OH . It saturates when the NH_2OH concentration reaches to 15%.

B. Undercutting at Convex Corners

The undercutting is the lateral etching which occurs under the masking layer. Convex corner undercutting is the desirable feature in the fabrication of overhanging microstructures, for example, the realization of cantilever beams made of different materials such as p+-Si, SiO_2 , Si_3N_4 etc. via bulk micromachining for applications in MEMS [1], [3]-[6], [24]-[27]. The undercutting at convex corner on $\text{Si}\{100\}$ is measured along $\langle 110 \rangle$ directions on square shape mask patterns, while on $\text{Si}\{110\}$ it is measured along $\langle 112 \rangle$ directions on acute and obtuse corners of rhombus shape mask geometries. Undercutting at convex corners on $\text{Si}\{100\}$ and $\text{Si}\{110\}$ with schematic views is presented in Figs. 2 and 3, respectively. In both cases, the undercutting increases significantly with increasing concentration of NH_2OH and saturates when NH_2OH concentration reaches to 15%.

C. Surface Morphology

In wet anisotropic etching, microscopic surface roughness is the result of many factors. Among various factors, most

important causes are hydrogen bubble formation during etching which hinders the surface reactions and acts as a micromask on the surface and/or the deposition of etching products on the surface during etching process such as SiO_2 precipitates from the crystal and contaminants over the surface.

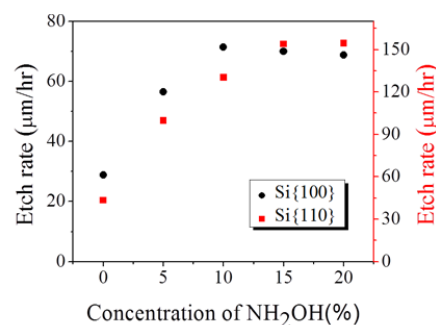


Fig. 1 Etch rate of $\text{Si}\{100\}$ and $\text{Si}\{110\}$ with varying concentrations of NH_2OH (%) at 70 ± 1 °C (etching time: 1 hr)

At the microscopic level, roughness can be the effect of non-uniform removal of atoms from the surface or lattice defects on the surface and extending into the bulk crystal. It is well known that very rough surface appears in low concentration TMAH. Microscopic roughness during etching process appears in the form of micropyramidal hillocks in the case of $\text{Si}\{100\}$ and nose shape structures in the case of $\text{Si}\{110\}$ surface. The surface roughness of $\text{Si}\{100\}$ and $\text{Si}\{110\}$ samples etched for 2 hrs is presented in Fig. 4. As the concentration of additive increases, surface roughness of $\text{Si}\{100\}$ decreases drastically as shown in Fig. 4. It can be seen from Fig. 4 that the surface roughness of $\text{Si}\{100\}$ is high at 5% concentration of NH_2OH , but it is comparatively very less at high concentrations of NH_2OH . At the same time, Fig. 4 shows the variation in etched surface roughness of $\text{Si}\{110\}$ with varying concentrations of NH_2OH . Even though there are fluctuations in the surface roughness of $\text{Si}\{110\}$ while adding NH_2OH , it is less than that in pure TMAH.

IV. CONCLUSION

The effect of different concentrations of NH_2OH on etching rate, surface roughness, and undercutting at convex corners on $\text{Si}\{100\}$ and $\text{Si}\{110\}$ wafers are studied. It is found that the etch rate of $\text{Si}\{100\}$ and $\text{Si}\{110\}$ increases nearly two-fold and three-fold, respectively, on addition of NH_2OH . Undercutting also follows the same trend as that of etch rate. It increases drastically in NH_2OH -added TMAH. The surface roughness decreases with an increase in the concentration of NH_2OH . In a nutshell, the etching characteristics of TMAH improved favorably and very useful for the fabrication of micro/nanostructures using silicon wet bulk micromachining.

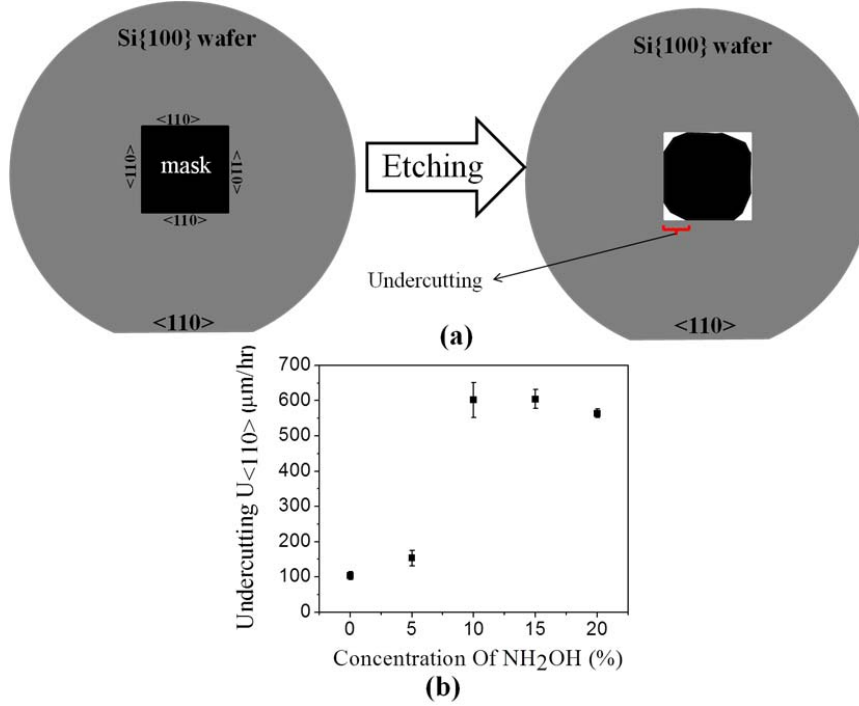


Fig. 2 Convex corner undercutting on Si{100} along <110> directions: (a) schematic view and (b) quantitative change with varying concentrations of NH_2OH (Etching time: 1 hr and etching temperature: $70 \pm 1^\circ\text{C}$)

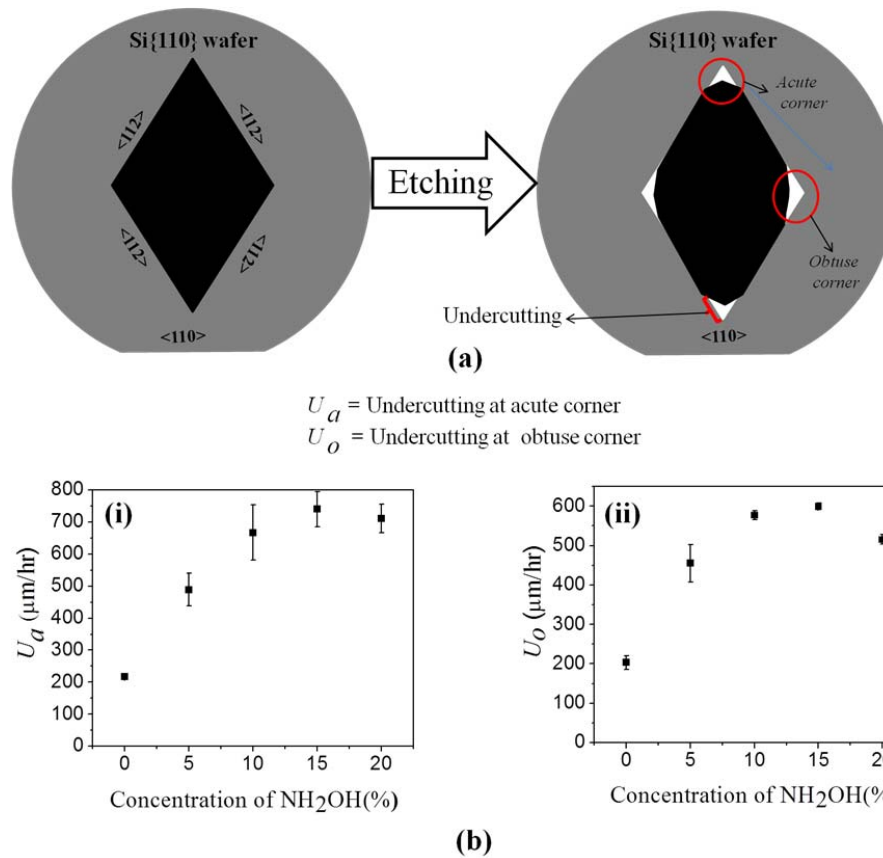


Fig. 3 Convex corner undercutting on Si{110} along <112> directions: (a) schematic view and (b) quantitative variation with varying concentrations of NH_2OH (Etching time: 1 hr and etching temperature: $70 \pm 1^\circ\text{C}$)

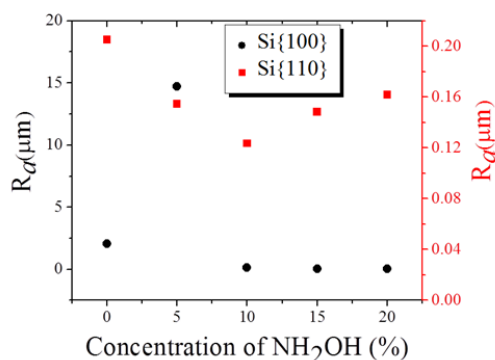


Fig. 4 Average surface roughness of Si{100} and Si{110} with varying concentrations of NH₂OH (Etching time: 2 hr and etching temperature: 70±1°C; R_a = Average surface roughness)

ACKNOWLEDGMENT

This work was supported by research grant from the Department of Science and Technology (Project No. SR/S3/MERC/072/2011) and the Council of Scientific and Industrial Research (CSIR, Ref: 03(1320)/14/EMR-II), New Delhi, India. The authors are thankful to Prof. K. Sato, Aichi Institute of Technology Toyota, Japan for his suggestions.

REFERENCES

- [1] P. Pal, and K. Sato, *Silicon wet bulk micromachining for MEMS*. Pan Stanford Publishing, Singapore, 2017.
- [2] I. Zubel, and M. Kramkowska, "Possibilities of extension of 3D shapes by bulk micromachining of different Si (hkl) substrates," *J. Micromech. Microeng.*, vol. 15, pp.485-93, Dec. 2005.
- [3] P. Pal, K. Sato, M. A. Gosalvez, B. Tang, H. Hida, and M. Shikida, "Fabrication of novel microstructures based on orientation dependent adsorption of surfactant molecules in TMAH solution," *J. Micromech. Microeng.*, vol. 21, pp. 015008 (11pp), Dec. 2011.
- [4] P. Pal, and K Sato, "Fabrication methods based on wet etching process for the realization of silicon MEMS structures with new shapes," *Microsyst. Technol.*, vol. 16, pp. 1165-1174, Jul. 2010.
- [5] P. Pal, K. Sato, and S. Chandra, "Fabrication techniques of convex corners in (100)-silicon wafer using bulk micromachining: A Review," *J. Micromech. Microeng.*, vol. 17, no. 10, pp. R111-R133, Sep. 2007.
- [6] P. Pal, and K. Sato, "A comprehensive review on convex and concave corners in silicon bulk micromachining based on anisotropic wet chemical etching," *Micro Nano Syst. Lett.*, vol. 3, no. 1, pp.1-42, Dec. 2015.
- [7] J. Chen, L. Liu, Z. Li, Z. Tan, Q. Jiang, H. Fang, Y. Liu, "Study of anisotropic etching of (100) Si with ultrasonic agitation," *Sens. Actuators A*, vol. 96, no. 2, pp.152-156, Feb. 2002.
- [8] J. A. Dziuban, "Microwave enhanced fast anisotropic etching of monocrystalline silicon," *Sens. Actuators A*, vol. 85, no.1, pp.133-138, Aug. 2000.
- [9] H. Tanaka, S. Yamashita, Y. Abe, M. Shikida, and K. Sato, "Fast etching of silicon with a smooth surface in high temperature ranges near the boiling point of KOH solution," *Sens. Actuators A*, vol. 114, no. 2-3, pp. 516-520, Sep. 2004.
- [10] B. Tang, K. Sato, D. Zhang, and Y. Cheng, "Fast Si (100) etching with a smooth surface near the boiling temperature in surfactant modified tetramethylammonium hydroxide solutions," *Micro Nano Letts.*, vol. 9, no. 9, pp. 582-584, Sep. 2014.
- [11] R. Sotoaka, "New etchants for high speed anisotropic etching of silicon," *J. Surf. Finish. Soc. Jpn.*, vol. 59, no. 2, pp. 104-106, 2008.
- [12] H. Tanaka, M. Takeda, and K. Sato, "Si (100) and (110) etching properties in 5, 15, 30 and 48 wt% KOH Aqueous solution containing Triton-X-100," *Microsyst. Technol.*, 1-8, 2017.
- [13] P. Pal, A. Ashok, S. Haldar, Y. Xing, and K. Sato, "Anisotropic etching in low-concentration KOH: effects of surfactant concentration," *Micro & Nano Letts.*, vol. 10, no. 4, pp. 224-228, Mar. 2015.

- [14] C. R. Yang, P. Y. Chen, C. H. Yang, Y. C. Chiou, and R. T. Lee, "Effects of various ion-typed surfactants on silicon anisotropic etching properties in KOH and TMAH solutions," *Sens. Actuators A*, vol. 119, pp. 271-281, Mar. 2005.
- [15] I. Zubel, I. Barycka, K. Kotowska, and M. Kramkowska, "Silicon anisotropic etching in alkaline solutions IV: the effect of organic and inorganic agents on silicon anisotropic etching process," *Sens. Actuators A*, vol. 87, pp. 163-171, Jan. 2001.
- [16] I. Zubel, and M. Kramkowska, "The effect of isopropyl alcohol on etching rate and roughness of (100) Si surface etched in KOH and TMAH solution," *Sens. Actuators A*, vol. 93, no. 2, pp. 138-147, Sep. 2001.
- [17] P. Pal, K. Sato, M. A. Gosalvez, and Shikida, "Study of rounded concave and sharp edge convex corners undercutting in CMOS compatible anisotropic etchants," *J. Micromech. Microeng.*, vol. 17, no. 11, pp. 2299-2307, Oct. 2007.
- [18] Y. W. Xu, A. Michael, and C. Y. Kwok, "Formation of ultra-smooth 45° micromirror on (100) silicon with low concentration TMAH and surfactant: Techniques for enlarging the truly 45° portion," *Sens. Actuators A*, vol. 166, pp. 164-71, Mar. 2011.
- [19] P. Pal, K. Sato, M. A. Gosalvez, Y. Kimura, K. Ishibashi, M. Niwano, H. Hida, B. Tang, and S. Itoh, "Surfactant adsorption on single crystal silicon surfaces in TMAH solution: orientation-dependent adsorption detected by in-situ infra-red spectroscopy," *J. Microelectromech. Syst.*, vol. 18, pp. 1345-1356, Dec. 2009.
- [20] P. Pal, M. A. Gosalvez, and K. Sato, "Etched profile control in anisotropic etching of silicon by TMAH+Triton," *J. Micromech. Microeng.*, vol. 22, pp. 065013 (9pp), May. 2012.
- [21] B. Tang, P. Pal, M. A. Gosalvez, M. Shikida, K. Sato, H. Amakawa, and S. Itoh, "Ellipsometry study of the adsorbed surfactant thickness on Si{110} and Si{100} and the effect of pre-adsorbed surfactant layer on etching characteristics in TMAH," *Sens. Actuators A*, vol. 156, pp. 334-341, Dec. 2009.
- [22] A. V. Narasimha Rao, V. Swarnalatha, A. Ashok, S. S. Singh, and P. Pal, "Effect of NH₂OH on etching characteristics of Si {100} in KOH solution," *ECS J. Solid State Sci. Technol.*, vol. 6, no. 9, P609-P614, Jan. 2017.
- [23] V. Swarnalatha, A. V. Narasimha Rao, A. Ashok, S. S. Singh, and P. Pal, "Modified TMAH based etchant for improved etching characteristics on Si {100} wafer," *J. Micromech. Microeng.*, vol. 27, no. 8, p. 085003, Aug. 2017.
- [24] P. Pal, and K. Sato, "Complex three dimensional structures in Si{100} using wet bulk micromachining," *J. Micromech. Microeng.*, vol. 19, p. 105008, (9pp), Sep. 2009.
- [25] P. Pal, and K. Sato, "Various shapes of silicon freestanding microfluidic channels and microstructures in one step lithography," *J. Micromech. Microeng.*, vol. 19, no. 5, p. 055003, (11pp), Apr. 2009.
- [26] P. Pal, and S. Chandra, "Bulk-micromachined structures inside anisotropically etched cavities," *Smart Mater Struct.*, vol. 13, pp.1424-1429, Oct. 2004.
- [27] G. T. Kovacs, N. I. Maluf, and K. E. Petersen, "Bulk micromachining of silicon," *IEEE Pro.* 1998, vol. 86, pp. 1536- 551.