

Silicon Microfluidic Channels and Microstructures in Single Photolithography Step

Prem Pal and Kazuo Sato

Department of Micro-Nano systems Engineering,
Nagoya University, Nagoya 464-8603, Japan
E-mail: prem@kaz.mech.nagoya-u.ac.jp

Abstract- In this paper, a fabrication method of suspended silicon microfluidic channels and various shapes of microstructures of desired thickness in (100)-Si wafers using single photolithography step is presented. The fabrication method uses wafer bonding with silicon nitride (Si_3N_4) as intermediate layer, local oxidation of silicon (LOCOS), and complementary metal oxide semiconductor (CMOS) process compatible wet anisotropic etching. The etching process is performed in two steps in non-ionic surfactant Triton-X-100 [$\text{C}_{14}\text{H}_{22}\text{O}(\text{C}_2\text{H}_4\text{O})_n$] added and pure tetramethyl ammonium hydroxide (TMAH) solutions. The surfactant added TMAH is used to define the shape of the structures, whereas pure TMAH is employed for their release.

I. INTRODUCTION

The suspended silicon MEMS structures of desired thickness on single (100)-Si wafers are fabricated by time controlled backside etching (dry or wet) after defining their shapes with required thickness at the front-side, as shown in Figure 1(a) [1-4]. In this case, the thickness of the structures may vary at different locations of the wafer due total thickness variation (TTV). In order to realize the uniform thickness microstructures throughout the wafer surface, SiO_2 based silicon on insulator (SOI) wafers are used, as shown in Figure 1(b) [5, 6]. In both cases, fabrication process requires front-to-back alignment with high accuracy, and cannot be used for the formation of densely packed microstructures if only wet anisotropic etching is employed. Moreover, the size of the backside mask opening for releasing the structures by wet anisotropic etching varies with the thickness of the wafer. In case of micromachined cavities sealed by wafer bonding for the realization of suspended structures, accurate alignment as well as the monitoring of pressure inside cavities during bonding process are highly desirable [7-11]. In all cases, two masks are needed for the fabrication of freestanding structures. Apart from the free-standing MEMS structures (e.g. cantilever beams, diaphragms etc.), microfluidic channels are also very useful structures for developing a system (or device) to carry and delivery liquids [12]. The overhanging silicon dioxide microfluidic channels uses very complex fabrication process and desires reinforcement structures to increase its strength to carry the heavy liquids [12]. Thus, the fabrication of suspended silicon microfluidic channels and densely arrayed microstructures of desire thickness on (100)-Si wafers using single sided lithography and wet (or

dry) etching processes is very difficult.

In this paper, a fabrication technique is proposed and demonstrated for the realization of suspended silicon MEMS structures such as cantilever, microfluidic channels etc. in single photolithography step. The method provides the following advantages:

- i. Easy control over the thickness of the structural layer throughout the wafer.
- ii. The problem of time control etching during etch release step is eliminated.
- iii. The use of dry etching for defining curved edges and sharp convex corners on (100)-silicon wafer is replaced by surfactant-added TMAH.
- iv. The cost of fabrication is reduced as only wet anisotropic etching is used for defining the shape of structures and their release.
- v. The front-to-back alignment step, which requires expensive equipment, is eliminated.
- vi. The extra mask required for defining the windows on the back-side mask layer for release is not needed. The process utilizes only one mask and a single photolithography step.

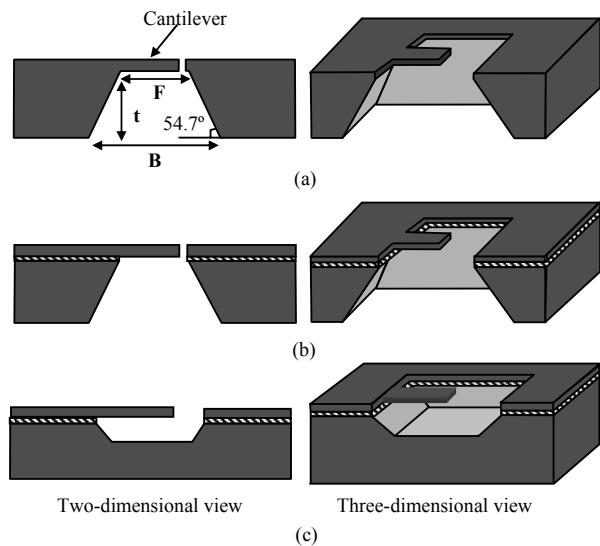


Figure 1: Schematic views of suspended cantilever beams realized in {100}-Si wafers using (a) time controlled etching (b) SOI wafer (c) micromachined sealed cavities by wafer bonding.

vii. Densely placed structures can easily be fabricated.

II. EXPERIMENTAL

The P-type, CZ grown (100)-Si wafers of three-inch diameter were used in order to fabricate the aforementioned type structures. In the proposed method, the realization of suspended silicon microfluidic channels and structures requires two wet etchant. In one case, the etchant must provide negligible undercutting at the curved edges, sharp convex and rounded concave corners, and the edges aligned along $\langle 100 \rangle$ directions in order to define the shape of the structures, while in other case it should exhibit high undercutting at these kinds of corners and edges for fast release of the structures. The surfactants are known to change the etching characteristics of TMAH solution when they are added in a very small amount [13-18]. Thus, the etch rates of (100) and (110)-Si, undercutting at sharp convex and rounded concave corners are measured in different concentration of pure and 0.1% v/v Triton-X-100 added TMAH.

The schematic views of the process steps used for the realization of simple freestanding silicon cantilever beams of required thickness with one photolithography step are shown in Figure 2. The process steps are generic and can be used for other structures. Firstly, the LPCVD (low pressure chemical vapor deposition) silicon nitride deposited wafers are bonded with thermally oxidized silicon wafers. Prior to bonding the surfaces are activated by RCA cleaning method using $\text{NH}_4\text{OH}:\text{H}_2\text{O}_2:\text{H}_2\text{O} :: 1:1:6$ and $\text{HCl}:\text{H}_2\text{O}_2:\text{H}_2\text{O} :: 1:1:5$ solutions at 70 °C followed by thorough rinse in DI water [7, 19-21]. The room temperature bonded wafers are annealed at 1050 °C for 1 hour in an oxygen environment. Thereafter, the oxide of the top wafer is removed and time controlled wet anisotropic etching in 25 wt% TMAH is performed to reduce the wafer thickness upto required level with smooth surface finish (Fig. 2(c)). A chemical mechanical polishing (CMP) process is employed to remove the micro pyramids (or hillocks) formed during wet anisotropic etching step. This is followed by standard cleaning process. The bonded wafers are then oxidized, followed by patterning using photolithography and oxide etching steps. After this step, the anisotropic etching is performed in Triton-X-100 added 25 wt% TMAH solution. As discussed in the next section, this etchant exhibits reasonable {100}-Si etch rate with minimum undercutting at the edges not aligned with the $\langle 110 \rangle$ directions, curved edges, sharp convex and rounded concave corners. Thereafter, the oxide is removed and local oxidation of silicon (LOCOS) process is carried out (Fig. 2(e)). At this step of thermal oxidation only top surface and side walls formed in Triton added TMAH are oxidized. The silicon nitride is then removed in hot phosphoric acid. Since the structures are now to be released, high undercutting at sharp convex corners and non- $\langle 110 \rangle$ directions is desirable for their fast release. Therefore, at this step of anisotropic etching, either 20 wt % or 25 wt% TMAH is used for their quick release. Finally, the oxide and nitride is removed chemically. The released structure is shown in Fig. 2(h) schematically.

III. RESULTS AND DISCUSSION

The etching parameters including the etch rates of {100} and {110}-Si, and undercutting at sharp convex and rounded concave corners, are all essential features for the determination of the dimensions of mask patterns for the realization of desired etched profiles and freestanding structures. The etch rates of (100) and (110)-Si in pure and Triton added TMAH are shown in Figures 3 and 4, respectively. The etch rate of {100}-Si in pure TMAH increases with decrease in concentration, whereas in Triton added TMAH it increases with increase in concentration. In case of {110}-Si wafer, etch rate increases with increase in concentration of pure TMAH, while it is almost independent of the TMAH concentration when 0.1% v/v Triton is added.

The comparisons of undercutting ratios at sharp convex (l_1/d ; l_1 : undercutting along $\langle 110 \rangle$ direction for etch dept d) and rounded concave (l_2/d ; l_2 : undercutting along $\langle 100 \rangle$ direction for etch dept d) corners in pure and Triton added TMAH are presented in Figures 5 and 6, respectively. It is clearly seen that the undercutting reduces to a significantly low level in surfactant added TMAH. As shown in Figures 3, 4, 5 and 6, that the addition of Triton in low concentration (10 wt% or 20 wt%) TMAH affects all etching parameters, whereas in case of 25 wt% TMAH, {100} etch rate is almost unaffected.

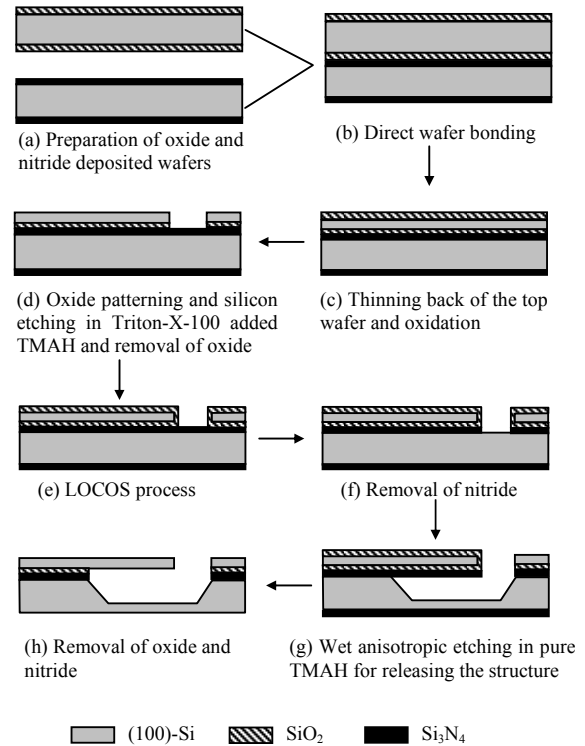


Fig. 2. Schematic view of process steps used for the realization of suspended Si structures of desired thickness.

If the etching parameters for all concentrations presented in Figures 3, 4, 5 and 6 are analyzed collectively, 0.1% v/v Triton in 25 wt% TMAH suppress the etch rates of non-{100} planes significantly. On the other hand, 20 and 25 wt% pure TMAH provides very high undercutting at

non-sharp concave corners, and non- $\langle 110 \rangle$ and curved edges.

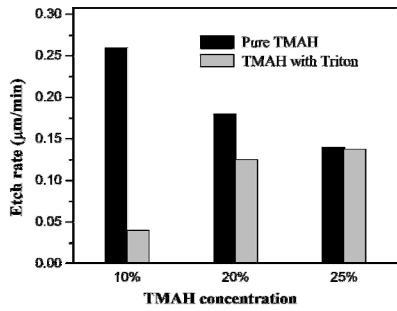


Fig. 3. Etch rates of (100)-Si in different concentration of pure and Triton X-100 added TMAH at 60 °C.

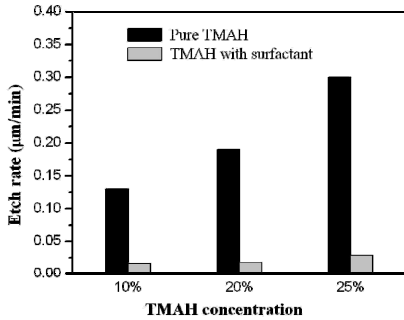


Fig. 4. Etch rates of (110)-Si in different concentration of pure and Triton X-100 added TMAH at 60 °C.

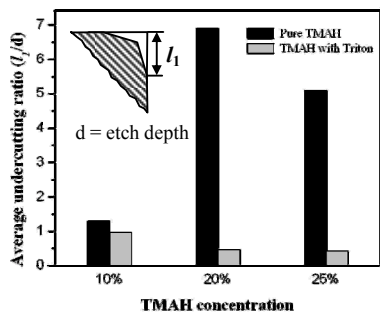


Fig. 5. Undercutting ratio at sharp convex corners (l_1/d) in different concentration of pure and Triton X-100 added TMAH at 60 °C; l_1 : undercutting along $\langle 110 \rangle$ direction at convex corner, d : etch depth at Si(100) wafer surface.

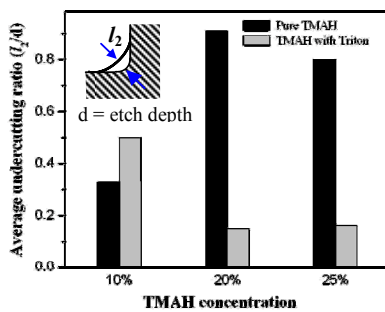


Fig. 6. Undercutting ratio at rounded concave corners (l_2/d) in different concentration of pure and Triton X-100 added 25 wt% TMAH at 60 °C; l_2 : undercutting along $\langle 100 \rangle$ direction at rounded concave corner, d : etch depth at Si(100) wafer surface.

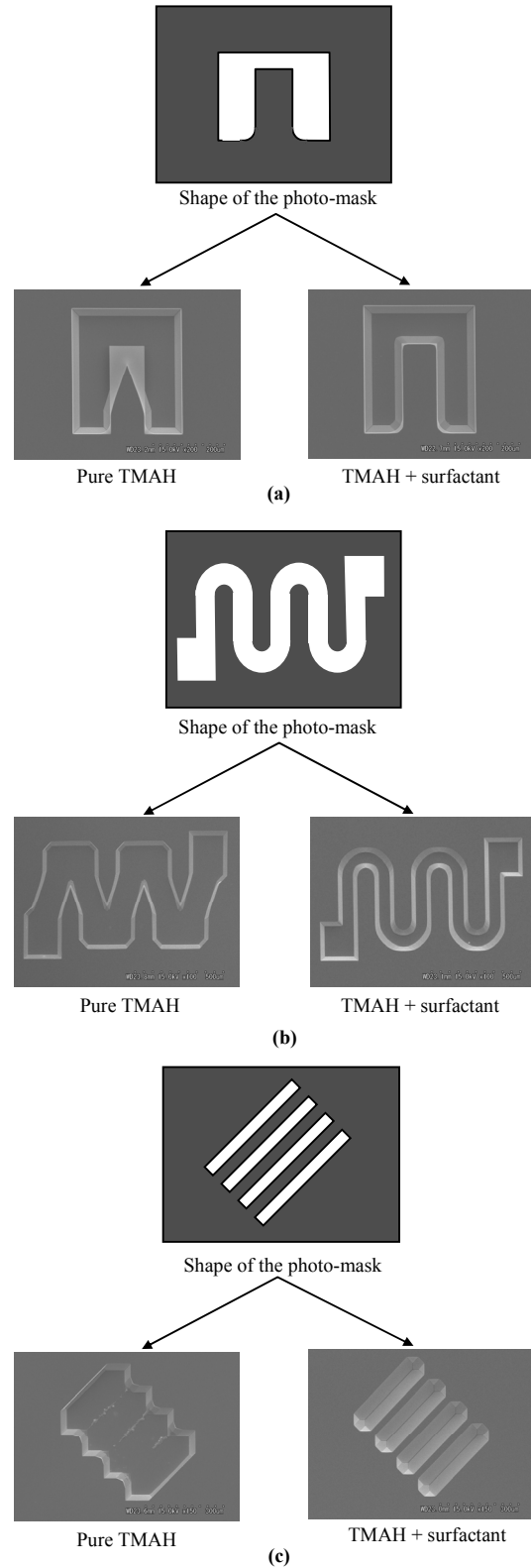


Fig. 7. Different types of MEMS structures fabricated in (100)-Si wafers using 25 wt% TMAH+0.1% v/v Triton solution: (a) cantilever beam with sharp convex and rounded concave corners (b) serpentine shape microfluidic channels (c) 45° mirror. SEM pictures shown in (b) and (c) are taken after removal of masking layer.

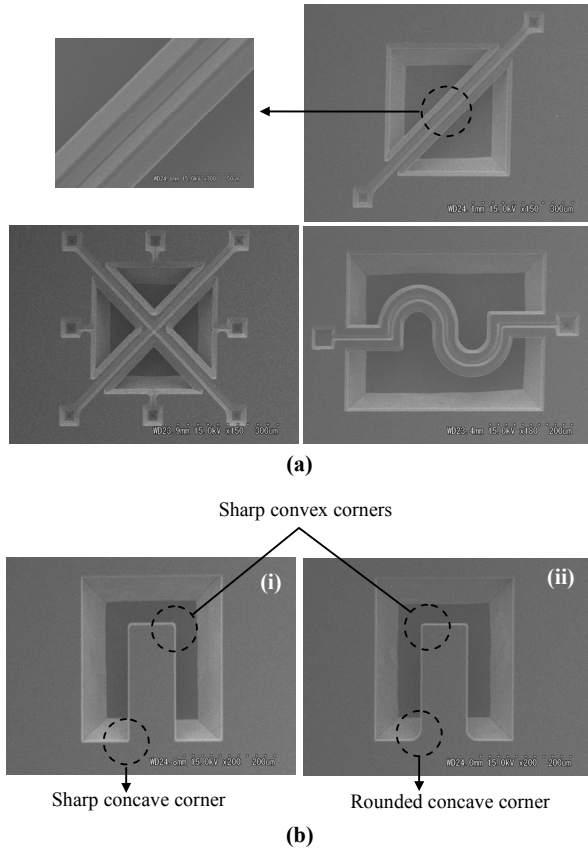


Fig. 8. SEM pictures of fabricated different kinds of suspended silicon (a) Microfluidic channels (b) Cantilever beams with (i) sharp concave and convex (ii) rounded concave and sharp convex corners (beam thickness = 4.5 μm). All structures are realized in single step UV lithography.

In the silicon semiconductor fabrication processes, $\{110\}$ and $\{111\}$ -Si oriented silicon wafers are used only for specific applications. Most MEMS structures as well as the CMOS devices are formed in/on $\{100\}$ oriented silicon wafers. In case of $\{100\}$ -Si wafers, the amount of undercutting at different shapes corners and edges must be negligible in comparison to the etch rate of $\{100\}$ -Si in order to attain the etched profile identical to the shape of mask pattern. As discussed in the previous paragraph, 25 wt% TMAH+0.1% v/v Triton solution provides such etching characteristics i.e. the etch rate of non- $\{100\}$ -Si in this solution is nearly one order of magnitude less than that in pure 25 wt% TMAH. In order to demonstrate the applications of Triton added TMAH for the fabrication of new shapes structures, various kinds of mask patterns are used. Figures 7(a), 7(b) and 7(c) show the cantilever shaped structures with sharp convex and rounded concave corners, serpentine channels and 45° mirror, respectively. All these structures are formed in $\{100\}$ -Si wafers. An important feature can be noticed from the SEM pictures shown in Figure 7 that the pure TMAH provides high undercutting at the non- $\langle 110 \rangle$ and curved edges, while Triton added TMAH exhibits minimum undercutting at those types of edges. In the fabrication of some class of MEMS structures

such as cantilever beams made of dielectric ($\text{SiO}_2/\text{Si}_3\text{N}_4$), metal, and $\text{P}^+\text{-Si}$, as high as possible undercutting is desirable for their fast release [22]. The pure 25 wt% (or 20 wt%) TMAH is the best choice for such type of application as it provides very high undercutting at sharp convex corners and non- $\langle 110 \rangle$ edges.

Fig. 8 shows the SEM pictures of suspended silicon microfluidic channels of different shapes, and various kinds of microstructures with rounded concave and sharp convex corners. All these structures are formed using only one photolithography step and two different etching solutions (with and without surfactant added 25 wt% TMAH) in series. For all kinds of structures, the same process steps are used as illustrated in Fig. 2. The grooves of microfluidic channels are nicely shaped even in case of rounded types. The dimensions of the mask patterns for the desired shapes of the structures (or resultant shapes for given mask patterns) can be estimated using trigonometric relations considering the undercutting and etch rates of different planes as presented in Figures 3, 4, 5 and 6. These etching results can also be used to estimate the time required during etch release step. The area surrounding the microfluidic channels and cantilever beams, as shown in Figure 8, should be selected in such a way that no V-grooves should form in this area during first etching step illustrated in Figure 2(b). Additionally, in case of microfluidic channels, the surrounded area must be sufficiently enough in order to release the structure by undercutting process during second step of etching in pure TMAH, as described in Figure 2(g). These calculations can be done keeping in view the crystallographic restriction of silicon in wet anisotropic etching solutions.

It is essential to emphasize here that the wafer bonding in this work is performed only to develop a fabrication technique with the aforementioned objectives. The detailed study of wafer bonding with silicon nitride as intermediate layer is beyond the scope of the present work. As mentioned in the previous section, the thickness of the one of the bonded wafers for structural layer was tailored by wet etching process. Thus, it may vary at different locations owing to the total thickness variation (TTV) of the wafer. In order to have a precise control over the thickness of the structural layer, the bonded and etched back method is recommended [23, 24]

IV. CONCLUSION

Different types of fixed and freestanding silicon MEMS structures are fabricated using very economic wet anisotropic etching using single step photolithography. A comprehensive analysis of pure and Triton-X-100 added TMAH solutions at different concentration is performed in order to emphasize their applications under different conditions for the fabrication of different shapes MEMS structures. The surfactant added 25 wt% TMAH is characterized as a best choice for etching any shapes of masking patterns with minimum undercutting at the edges and corners. The high concentration of TMAH (20-25 wt%) is an optimal selection for obtaining high undercutting at non-sharp concave corners and non- $\langle 100 \rangle$ edges. The type

of the etchant can be selected according to the requirement.

ACKNOWLEDGMENT

This work was supported by Japan Society of Promotion of Science (JSPS) through a foreign postdoctoral research fellowship scheme (2008-2010, fellowship ID No. 08053), and a grant-in-aid for scientific research (A) 14205016. We are grateful to Prof. Norikazu Suzuki for providing the chemical mechanical polishing (CMP) facility and for suggestions to use it successfully. Special thanks to H. Hida, S. Ukai, R. Imai, T. Yokota, T. Takumi and Y. Suzuki for their technical assistance.

REFERENCES

- [1] M. Elwenspoek and H. Jansen, *Silicon Micromachining*, Cambridge University Press, UK, 1998.
- [2] W. Lang, "Silicon microstructuring technology," *Mater. Sci. Eng. R*, vol. 17, pp. 1-55, 1996.
- [3] N. Abedinov et al., "Micromachined piezoresistive cantilever array with integrated resistive microheater for calorimetry and mass detection," *J. Vac. Sci. Technol.*, vol. A 19, pp. 2884-2888, 2001.
- [4] Q. Wu, K.M. Lee, and C.C. Liu, "Development of chemical sensors using microfabrication and micromachining techniques," *Sensors Actuators B*, vol. 13-14, pp. 1-6, 1993.
- [5] D. Saya et al., "Si-piezoresistive microcantilevers for highly integrated parallel force detection applications," *Sensors Actuators A*, vol. 123-124, pp. 23-29, 2005.
- [6] B.L. Pruitt and T.W. Kenny, "Piezoresistive cantilevers and measurement systems for characterizing low force electrical contacts," *Sensors Actuators A*, vol. 104, pp. 68-77, 2003.
- [7] Q.Y. Tong and U. Gosele *Semiconductor Wafer Bonding: Science and Technology*, John Wiley and Sons Inc., New York, 1999.
- [8] S. Mack, H. Baumann, U. Gosele, H. Werner, and R. Schlogl, "Analysis of bonding-related gas enclosure in micromechanical cavities sealed by silicon wafer bonding," *J. Electrochem. Soc.*, vol. 144, pp. 1106-1111, 1997.
- [9] P. Pal and K. Sato, "Suspended Si Microstructures with Rounded Concave and Sharp Convex Corners Using Wafer Bonding and Wet Anisotropic Etching," *ECS Transactions*, vol. 16, pp. 133-140, 2008.
- [10] Y. Huang, A.S. Ergun, E. Haeggstrom, M.H. Badi, and B.T. Khuri-Yakub, "Fabricating capacitive micromachined ultrasonic transducers with wafer-bonding technology," *J. Microelectromech. Syst.*, vol. 12, pp. 128-137, 2003.
- [11] M.A. Huff, A. D. Nikolich, and M.A. Schmidt, "Design of sealed cavity microstructures formed by silicon wafer bonding," *J. Microelectromech. Syst.*, vol. 2, pp. 74-81, 1993.
- [12] J.W. Kwon and E.S. Kim, "Multi-level microfluidic channel routing with protected convex corners," *Sensors Actuators A*, vol. 97-98, pp. 729-733, 2002.
- [13] D. Cheng, M.A. Gosalvez, T. Hori, K. Sato, and M. Shikida, "Improvement in smoothness of anisotropically etched silicon surfaces: Effects of surfactant and TMAH concentrations," *Sensors and Actuators A*, vol. 125, pp. 415-421, 2006.
- [14] P. Pal, K. Sato, M. A. Gosalvez and M. Shikida, "Study of rounded concave and sharp edge convex corners undercutting in CMOS compatible anisotropic etchants," *J. Micromech. Microeng.*, vol. 17, pp. 2299-2307, 2007.
- [15] 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," *Sensors Actuators A*, vol. 119, pp. 271-281, 2005.
- [16] D. Resnik, D. Vrtacnik, U. Aljancic, M. Mozek, and S. Amon, "The role of Triton surfactant in anisotropic etching of {110} reflective planes on (100) silicon," *J. Micromech. Microeng.*, vol. 15, pp. 1174-1183, 2005.
- [17] J.S. Jeon, S. Raghavan, and J.P. Carrejo, "Effect of Temperature on the Interaction of Silicon with Nonionic Surfactants in Alkaline Solutions," *J. Electrochem. Soc.*, vol. 143, pp. 277-283, 1996.
- [18] P. Pal, K. Sato, M.A. Gosalvez, and M. Shikida, "Study of corner compensating structures and fabrication of various shapes of MEMS structures in pure and surfactant added TMAH," *Sensors Actuators A*, in press.
- [19] S. Sanchez, C. Gui, and M. Elwenspoek, "Spontaneous direct bonding of thick silicon nitride," *J. Micromech. Microeng.*, vol. 7, pp. 111-113, 1997.
- [20] M.S. Ismail, R.W. Bower, J.L. Veteran, and O.J. Marsh, "silicon nitride direct bonding," *Electron Lett.*, vol. 26, pp. 1045-1046, 1990.
- [21] M. Wiegand et al., "Wafer bonding of silicon wafers covered with various surface layers," *Sensors Actuators A*, vol. 86, pp. 91-95, 2000.
- [22] P. Pal and S. Chandra, "Bulk-micromachined structures inside anisotropically etched cavities," *Smart Mater and Struct.*, vol. 13, pp. 1424-1429, 2004.
- [23] J. A. Plaza, J. Esteve, and E. Lora-Tamayo, "Simple technology for bulk accelerometer based on bond and etch back silicon on insulator wafers," *Sensors Actuators A*, vol. 68, pp. 299-302, 1998.
- [24] W.P. Maszara, P.P. Pronko, and A.W. McCormick, "Epi-less bond-and-etch-back silicon-on-insulator by MeV ion implantation," *Appl. Phys. Lett.*, vol. 58, pp. 2779-2781.