Research on optimal silicon etching condition in TMAH solution and application for MEMS structure fabrication

Dinh Van Dung*

Hanoi Pedagogical University No.2, Hanoi, Vietnam

Received 15 May 2009

Abstract. The research of optimal condition for etching silicon in TMAH solution with controlled etch rate and low surface roughness is the purpose of this study. The investigation on the influence of temperature, agitation, size of etch-window, etch time on etch rate and the surface roughness were carried out. With the TMAH concentration of 20% in weight, the optimal etching conditions were as follows: temperature of about 80 - 90 °C, agitation of 150 - 200 rpm. The etch rate is controlled in range of $0.49 - 0.72 \,\mu$ m/min. The etched surface roughness was lower than 70 nm. Development of TMAH application, a useful procedure for fabricating MEMS structures (piezoresistive accelerometer) was suggested.

Keywords: MEMS technology, TMAH, silicon etching, accelerometer, MEMS fabrication.

1. Introduction

Anisotropic etching of silicon is a key technology for fabricating various 3-dimensional structures for Micro Electro Mechanical Systems (MEMS). Although deep reactive ion etching (Deep RIE) has become popular for realizing high aspect ratio silicon microstructures, the advantages of silicon anisotropic etching technology such as low process cost, better surface smoothness and lower environmental pollution makes them as a complementally technology.

The most popular chemical etchants are Potassium Hydroxide KOH and Tetra Methyl Ammonium Hydroxide (CH₃)₄NOH (TMAH). KOH was proposed for application very early. The salient good points of this etchant are its possibility of making structures with high aspect ratio because of the strong anisotropy in etch rate. However, the drawback of KOH is its incompatibility with the IC technology [1]. To overcome the disadvantages of KOH, Tetra Methyl Ammonium Hydroxide (TMAH) was later introduced as a fruitful solution for MEMS fabrication [2].

TMAH is a weak organic hydroxide without any metal ions, so it is quite compatible with the IC technology. The chemical product is nontoxic and easy in handle [3,4]. Compared to etching in KOH solution, SiO_2 and $Si_3 N_4$ are better mask materials for etching processes in TMAH. Especially, the studies of Tabata [4-6] show that, the Al etching in TMAH solution can be decreased to extremely low etch rate when some appropriate amount of silicon is diluted into the TMAH solution. The presence of integrated circuits as well as Al interconnections is obvious in MEMS fabrications. If the adequate etching condition of which the solution is assumed not to etch Al is found, then the problem of Al

^{*} E-mail: <u>dinhvan_dung@yahoo.com</u>

connections protection and integrated circuits protection in silicon etching process can seem to be solved. Thus, the use of TMAH as the etchant seems to be optimal in the case when only a one-side aligner is available and the thermal SiO_2 is used as the mask material. The point will be especially useful for MEMS devices fabricated by etching through whole silicon wafer such as beam structures in accelerometers, gyroscopes... Some papers reported that, the TMAH solution with the concentrations higher than 20 wt. % allows fabricating smooth surfaces [6,7]. The silicon etch rate and the smoothness of etched surface can be improved considerably by controlling the conditions of etching process.

In this study, the influences of temperature, agitation, etch time, size of etch-windows on the etch rate and etched surface roughness were carried out. Based on the obtained results, the optimal conditions for etching silicon in TMAH were suggested. Finally, a useful procedure for fabricating MEMS structure using beams as mechanical element (for example: MEMS piezoresistive accelerometer) with the TMAH etchant was presented.

2. Experiments

In order to maintain the concentration and temperature of TMAH solution as well as the agitation at controlled speed during etching process, a special etching system IKA-WERKE MODEL 5000 was used (Fig. 1). The system consists of a thermostat which allows heating and maintaining temperature of the solution in the range of room temperature to 250° C and a magnetic stirrer with controlled speed of 0 -650 round per minute (rpm). The TMAH solution is contained in a vessel of 5-liter volume covered by a double-wall lid. A cool water flow running through the lid can make chemical product vapor condensed during the etching process in order to maintain the constant concentration of solution.

Silicon wafers of 2-inch, (100) orientation, n-type, and polished double side, phosphorous doped with sensitivities of 1 - 20 Ω .cm, 260 ± 25 μ m thickness were used for experiments. The protection material was only SiO₂ layer formed on two sides of silicon wafer by wet thermal oxidation. In the oxidation condition of 1100^oC, wet oxygen 1.5 l/min flow, oxidation



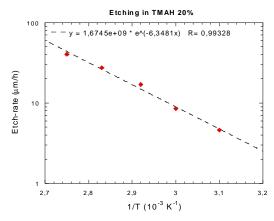
Fig. 1. The etching system of Model 5000.

time 3 hours, the thickness of the oxide layer was about $1.1 \,\mu\text{m}$. The oxide layer is sufficient to protect the samples during etching time including the case of etching through whole wafer thickness. Etchwidows were formed by photolithography technique. In the investigations on influence of temperature, stirring and etch time, the size of etch-window were chosen at $1 \times 1 \,\text{mm}^2$. For the studies on influence of etch-window size, the etch-window was chosen with the edge size of 1, 2, 4, 6, and 8 mm.

The average etch rate was calculated by dividing the etched groove height by etch time. Here, the etch height was measured by the instrument of Interferometric Optical Profilometer LEICA DMRM with very high accuracy. The profile of etched surface can also be measured by using this instrument. The process program of M3D connected to computer allows calculating average arithmetic roughness Ra and peak roughness Rt (distance from the lowest point to the highest point on the etched surface). They are important quantities determining the etched surface quality in MEMS fabrications.

3. Results and discussion

The Figs 2 and 3 show the results on the influence of temperature on etch rate and etched surface roughness. The experimental conditions were chosen as follows: TMAH 20% in weight, agitation of 150 rpm, etch time of 30 minutes.



Etching in TMAH 20% 1400 1200 1000 Roughness (nm) 800 -Ra Rt 600 400 200 0 90 50 60 70 80 Temperature (°C)

Fig. 2. The influence of temperature on etch rate.

Fig. 3. The influence of temperature on surface roughness.

The etch-rate increases strongly with increasing temperature. The fit curve has a shape of an exponential curve. The small deviation of experimental points compared to the fit curve shows that the obtained result corresponds to the theoretical rule. The activation energy is calculated $E_a = 0.548 \text{ eV}$ which is in good agreement with the result of Tabata [5].

At around 60° C, the etched-surface (100) is the roughest. At about 50° C, the average surface roughness is much lower than at 60° C. At these low temperatures, the intersection between wall side (111) and bottom side (100) is unclear. There are many pyramids on the bottom surface (100). At 70°C, the etch-rate is significantly higher and the intersection between (111 and (100) planes is clear, the etched surface (100) is smoother. However, there are still some peaks on the surface and the average roughness is still high.

At high temperatures of 80° C and 90° C, the etch process occurs very fast. On the etched surface (100), no pyramids were observed. The intersection between (111) plane and (100) plane was very clear; the surface smoothness was very good. Although the etch-rate at 80° C was not as high as at 90° C, but the surface quality in the condition of 80° C was better.

30

25

20 15 10

5

0

0

Roughness Ra (nm)

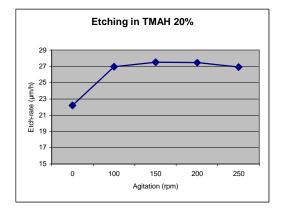


Fig. 4. The influence of agitation on etch rate.

Fig. 5. The influence of agitation on surface roughness.

150

Agitation (rpm)

200

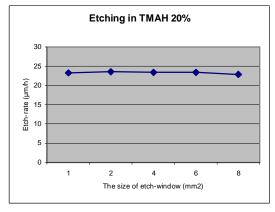
250

100

Etching in TMAH 20%

In the Figs of 4 and 5, the results on the influence of agitation are shown. The experiments were done in the TMAH solution of 20% in weight at temperature of 80°C. The stirring has a good action on the etched product transportation from etched-surface into the solution and fresh solution to the etched surface. So it makes etch process faster. But the agitation at very low speed or very high speed can make opposite effects. The stirring speed of 150 rpm is optimal for all Ra and Rt roughness. The etch-rate has largest value when the solution was stirred at about 150 rpm.

The influences of the size of etch-window on etch rate and surface roughness are presented in Figs 6 and 7. The etch conditions were the following: the agitation of 150 rpm, 30 minutes, temperature 80°C, TMAH 20% in weight. There is a light difference in etch-rate at different sizes of etch-window. The difference was caused by many reasons such as the position of the wafer and the holder in the solution, the declination of surface (100) compared to horizon plane...The surface roughness has also difference but not much when the size of etch-window changes. With the etch-window larger than 1 x 1 mm², no rule of the influence was found in the cases.



Etching in TMAH 20% 300 250 Roughness (nm) 200 Ra 150 Rt 100 50 0 2 6 8 4 The size of etch-window (mm2)

Fig. 6. The influence of size of etch-window on etchrate.

Fig. 7. The influence of size of etch-window on surface roughness.

Finally, the results on the influence of etch time on etch rate and roughness are reported. The experimental conditions were chosen as temperature 80°C, 150 rpm, TMAH 20% in weight. With increasing etch time, the etched groove depth increases. The transportation process of etched products from the etch-surface into solution and fresh solution to silicon surface becomes more difficult. This makes the average etch-rate to decrease lightly with time. Whereas, the amount and the height of pyramids will increase when increasing etch-time. That is the explanations for the results in Fig.8 and Fig.9.

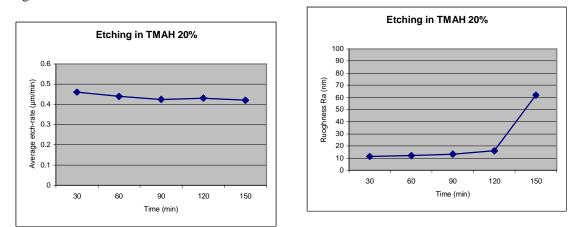


Fig. 8. The dependency of average etch-rate on time.

Fig. 9. The influence of etch time on surface roughness.

A good etched surface was obtained in the etching condition of TMAH 20 wt.%, temperature 80° C, and 150-rpm agitation as shown by SEM picture in Fig. 10. The etched surface (100) was very smooth with Ra = 12 nm, Rt = 63 nm.

4. TMAH application for MEMS structure fabrication

Al is etched in TMAH solution, but the etch rate decreases very sharply when the suitable amount of silicon was dissolved into the solution (Fig 11) [6]. The etch rate can be lower than 0.001 μ m/min if the amount of 3.5 silicon mol is dissolved into the TMAH 22 wt. % solution.

In MEMS fabrications, especially for the structures using piezoresistive effect, the electrical part of sensor including Al interconnections and integrated circuits is usually integrated on one chip, so the protection of the electrical structures is very important in etching process for forming structure.

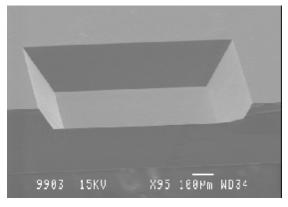


Fig. 10. SEM picture of etched surface in TMAH 20 wt. %, 80 °C, 150 rpm.

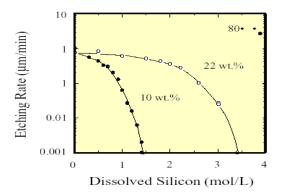


Fig. 11. The dependence of Al etch rate on diluted silicon amount in the TMAH 22wt.% solution [4].

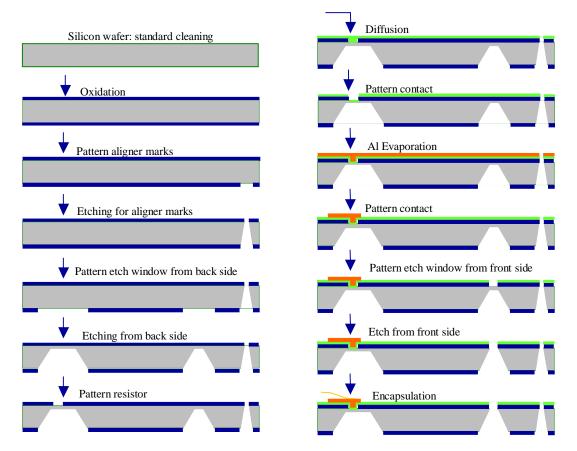


Fig. 12. Fabrication procedure of MEMS piezoresistive accelerometer using TMAH.

Based on the low Al etching in TMAH, a useful suggestion for fabricating MEMS using beam structure as mechanical sensitive element such as in piezoresistive accelerometer was given in Fig 12. In the procedure, we use only the photolithography instrument of one side aligner. The aligner marks for locating position precisely from two sides of the wafer are formed by etching through whole the wafer thickness. The protection material during etching process is only SiO₂ making by simply thermal oxidation. The first steps are carried out to make aligner marks; next the steps of making membrane and mesa mass are done. The electrical part is fabricated before releasing the beam structure in order to work easily lithography process. Then, the step of releasing beam structure by etching through whole the wafer is done without making Al protection layer in solution.

5. Conclusions

The obtained results show that, it is quite possible to control etch rate and lower etched surface roughness by controlling temperature and agitation suitably. In the TMAH solution of 20 % in weight, the optimized silicon etching condition in which the silicon etch rate is high and the etched surface roughness is low are as follows: temperature of $80 - 90^{\circ}$ C, agitation of about 150 - 200 rpm. When the solution temperature increases from 80 to 90 °C, the etch rate increases considerably from 0.49 µm/min to 0.72 µm/min. At about 150-rpm stirring, the average arithmetic roughness is lower than 70 nm. For the MEMS structures with thickness larger than 5 µm, the surface roughness lower than 70 nm is quite acceptable.

The Al etching at low etch-rate in TMAH makes the protection of electrical part in etching process to become much easier. For the technology condition in which a one-side aligner is available and the thermal SiO_2 is used as the mask material, aligner marks for locating precisely position from two sides of wafer were form by etching through whole the wafer thickness, the use of TMAH as the etchant seems to be technology solution allows being successful in fabrication of complex MEMS structures, especially beam structure such as in accelerometer, gyroscope... The suggested solution for accelerometer fabrication is quite possible to carry out in the technology condition in ITIMS laboratory.

Acknowledgement. This research was carried out in Institute of Electronics Fundamental (IEF), University of South Paris, France and International Training Institute for Materials Science (ITIMS), Hanoi University of Technology, Vietnam.

References

- [1] H.R.Huff, R.R.Burgess, Semiconductor Silicon (1973).
- [2] M. Asano, T. Cho, H. Muraoka, Electrochem. Soc. Ext. Abstr. (1976) 354.
- [3] O. Tabata, R. Asahi, H. Funabashi, S. Sugiyama, *Tec. Dig. of Int. Conf. On Solid-State Sensors and Actuators* (1991) 811.
- [4] O. Tabata, R.Asahi, H. Funabashi, K. Shimakao, S. Sugiyama, Sensors and Actuators A 34 (1992) 51.
- [5] O. Tabata, M. Yashima, T. Yoshioka, K. Sato, The 10th Int. Conf. On Solid-State Sensors and Actuators (1999) 542.
- [6] O. Tabata, Sensors and Materials Vol. 13, No. 5 (2001) 271.
- [7] J. van Suchtelen, K. Sato, E. van Veenendaal, A.J. Nijdam, J.G.E. Gardeniers, W.J.P. van Enckevort, M. Elwenspoek, Proceedings of the IEEE Micro Electro Mechanical Systems (1999) 332.