

# DEEP REACTIVE ION ETCHING OF PYREX GLASS

Xinghua Li, Takashi Abe <sup>a</sup> and Masayoshi Esashi <sup>b</sup>

Graduate School of Engineering, Tohoku University  
E-mail : [lixh@mems.mech.tohoku.ac.jp](mailto:lixh@mems.mech.tohoku.ac.jp)

<sup>a</sup>Venture Business Laboratory, Tohoku University

<sup>b</sup>New Industry Creation Hatchery Center, Tohoku University  
Aza-Aoba, Aramaki, Aoba-ku, Sendai 980-8579, JAPAN

## ABSTRACT

We have developed a deep reactive ion etching of Pyrex glass in SF<sub>6</sub> plasma. High etch rate ( $\sim 0.6 \mu\text{m/min}$ ) and smooth surface ( $R_a \sim 4 \text{ nm}$ ) were achieved at low pressure (0.2 Pa) and high self-bias ( $\sim 390 \text{ V}$ ). This result indicates energetic ions for physical sputtering and for enhancing chemical reactions are required to etch materials which produce nonvolatile reaction products. Vertical etch profile (base angle  $\sim 88^\circ$ ), high aspect ratio ( $>10$ ) and through-out etching of Pyrex glass ( $200 \mu\text{m}$  in thickness) were achieved when the mask opening is narrower than  $20 \mu\text{m}$ . Relatively low selectivity to the mask material due to the energetic ion is overcome using thick and vertical electroplated Ni film as a mask. We also find out the base angle of the etch profile depends on the mask profile and the opening width.

## INTRODUCTION

Microfabrication of glasses is important for microelectromechanical systems (MEMS) because it is widely used for anodic bonding with silicon. For example, the fabrication of holes having high aspect ratio is demanded to obtain electrical feedthrough from packaged sensors[1]. The glasses are also used for microoptics. Typical approaches to etch a glass such as ultrasonic machining, sand blast erosion and wet chemical etching are difficult to achieve fine ( $<10 \mu\text{m}$  in size) and high aspect ratio ( $>10$ ) structure.

Reactive ion etching (RIE) has a potential for batch fabrication of microstructure by using photolithography. However, the etch rate of glass by using a conventional RIE is extremely low ( $\sim 0.1 \mu\text{m/min}$ )[2]. There are many problems in the etching of materials (Glass, PZT etc.) which contain atoms as lead or sodium. These atoms make nonvolatile halogen compounds (PbF<sub>2</sub>, NaF etc.) as the reactant. In the results, the etching has problems as low etch rate, low selectivity to mask and low aspect ratio. Recently, high speed directional etching of silicon was performed by the RIE which employed inductively coupled plasma (ICP)[3]. High density plasma is sustained at low pressure ( $\sim 1 \text{ Pa}$ ) by using ICP sources [4]. We adapted the ICP plasma to the deep RIE of quartz and PZT [5-7]. The highest etch rate was  $1 \mu\text{m/min}$  for quartz and  $0.3 \mu\text{m/min}$  for PZT.

In this article, the ICP deep RIE of Pyrex glass has been studied systematically as a function of pressure, self-bias voltage and stage temperature. We also show an application of the etching technique in the MEMS field.

## EXPERIMENTAL SETUP

### Deep RIE system

Etching was performed in an ICP RIE system developed by Kong *et al* [3]. The schematic illustration of the etcher is shown in figure 1. An inductively coupled plasma source with one turn coil (ICP antenna) located on the top quartz window was used in this study.

The internal diameter of the reactor is 145 mm and the

gap width between the window and the stage is 13 mm. The chamber is evacuated by using large capacity turbo molecular pump (200L/min) during etching and it makes possible to decrease the deposition of reaction products on the wafer surface. A strong permanent magnet (Sm-Co) is located on the quartz plate to densify the plasma. These advantages of the etcher make it possible to etch materials which produce nonvolatile reaction products. The diameter of the wafer stage is 80 mm. Radio frequency (RF) power sources of 13.56 MHz were connected to both the ICP antenna and the stage. The RF power of the ICP antenna in this experiment was constant at 150 W. The other RF power supplied to the stage creates a self-bias voltage ranging from -190V (50W) to -390 V (140W) in this work. The stage temperature was controlled ranging from 233 K to 293 K by circulation of a chilled coolant. A silicone grease was used to fix the wafer onto the stage.

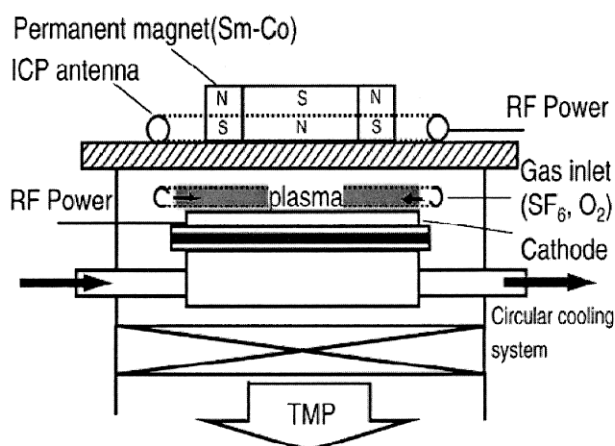


Figure 1 Schematic illustration of inductively coupled reactive ion etching apparatus.

### Sample preparation

The process step is shown in figure 2. A thin film of gold/chromium was sputtered on a Pyrex glass. A positive photoresist (AZ P4400, Clairant (Japan), K.K.) was coated on the gold/chromium film by a spinner and patterned by photolithography. The thickness of photoresist was 4  $\mu$ m and the profile was vertical to the glass substrate. The thick nickel pattern

was selectively electroplated on the exposed parts of the gold/chromium and then the photoresist was removed by acetone. Pulse plating method was applied to obtain the nickel film having both low internal stress and high purity. Finally, RIE was carried out with SF<sub>6</sub> as the etching gas.

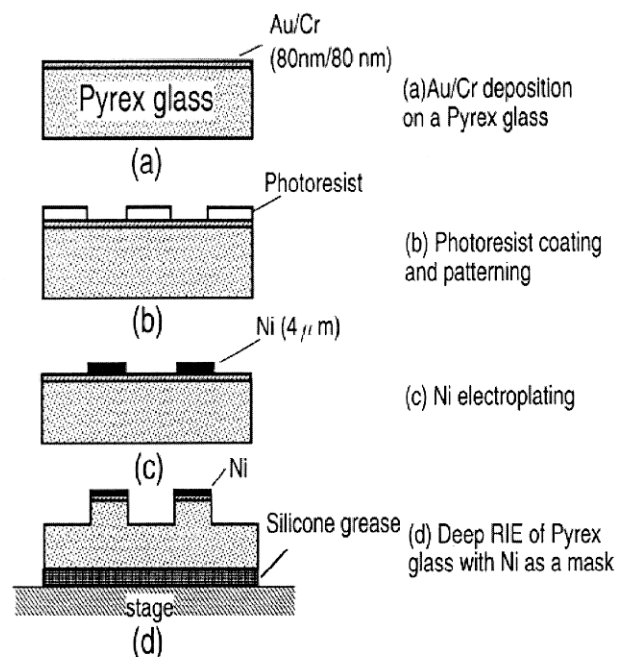


Figure2 Deep RIE process of Pyrex glass.

### Characterization of etched profile

The etch profiles and etched surface roughness have been investigated using both scanning electron microscope (S-2250N, HITACHI) and stylus profilometer (P-10, TENCOR). The electroplated nickel film used as the mask was removed using HNO<sub>3</sub> after the RIE.

## RESULTS AND DISCUSSION

### Etch rate vs. pressure

The etch rate of Pyrex glass with SF<sub>6</sub> gas is shown in figure 3 at high self-bias voltage(-390 V) as a function of pressure. The etch rate decreases as the pressure increases. On the other hand, the surface roughness become rough as the pressure increases. A dense needle

like microstructure was formed at high pressure (1.6 Pa) as shown in Fig. 3C. High etch rate ( $0.6 \mu\text{m}/\text{min}$ ) and smooth surface ( $R_a \sim 4 \text{ nm}$ ) were obtained at low pressure (0.2 Pa).

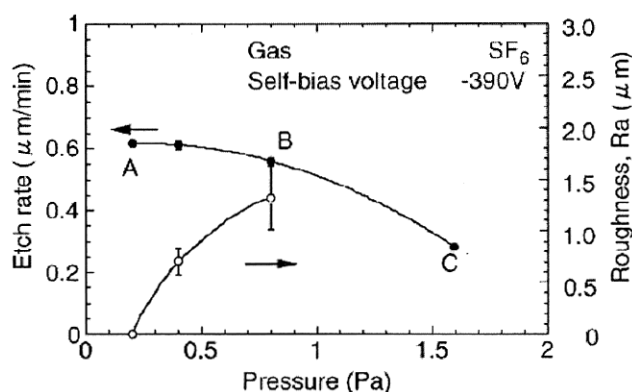
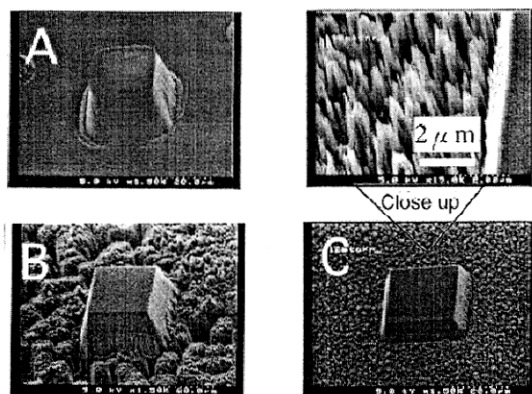


Figure 3 Etch rate of Pyrex glass as a function of gas pressure.

#### Etch rate vs. self-bias voltage

The etch rate at 0.2 Pa as a function of self-bias voltage is shown in figure 4. The etch rate increases as the voltage increases. The etched surface become flat like a mirror at high self-bias voltage ( $>390 \text{ V}$ ) as shown in figure 4B. This is due to the increased ion energy for physical sputtering and for enhancing chemical reactions.

#### Etch rate vs. stage temperature

The etch rate at 0.2 Pa and 0.8 Pa are shown as a function of the stage temperature in figure 5. The self-bias voltage is constant at  $-390 \text{ V}$ . The temperature dependence of the etch rate at 0.2 Pa is negligible and the rate is  $0.6 \mu\text{m}/\text{min}$ . On the other hand, the etch rate at 0.8 Pa

increases as the temperature increases. These results indicate chemical reactions resulting in the formation of volatile  $\text{SiF}_x$  products and nonvolatile products ( $\text{AlF}_3$ ,  $\text{NaF}$ ) predominate at high pressure. To remove the reaction products immediately, low chamber pressure is required. Thus, the optimum conditions to achieve the high speed etching are high self-bias voltage ( $-390 \text{ V}$ ), low pressure (0.2 Pa).

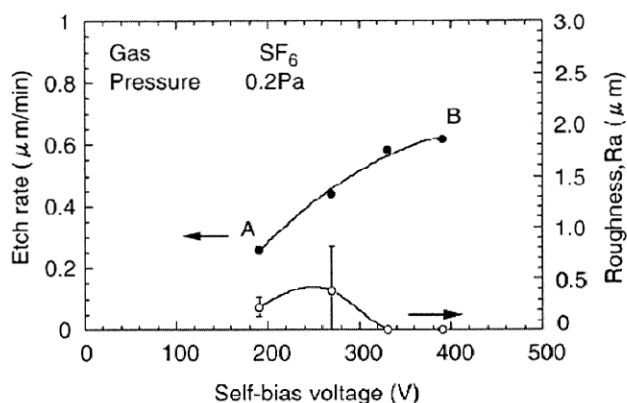
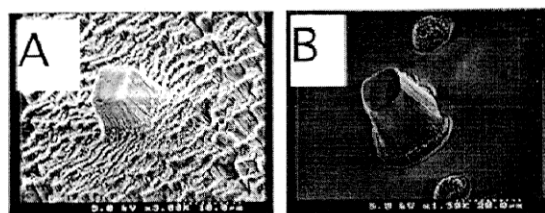


Figure 4 Etch rate of Pyrex glass as a function of self-bias voltage to substrate.

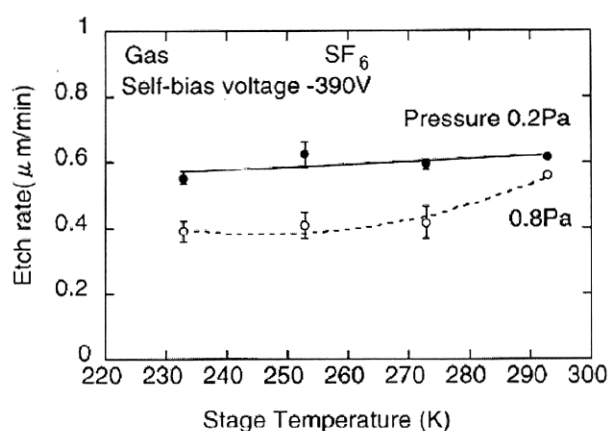


Figure 5 Etch rate of Pyrex glass as a function of stage temperature.

## Micro-loading effect

The etched depths were measured as a function of etching time and different opening widths of the mask at the optimum condition. As shown in figure 6, the micro-loading effect is observed at smaller opening width. However, the effect is less than that of the etching results of silicon carried out by Kong *et al* [3].

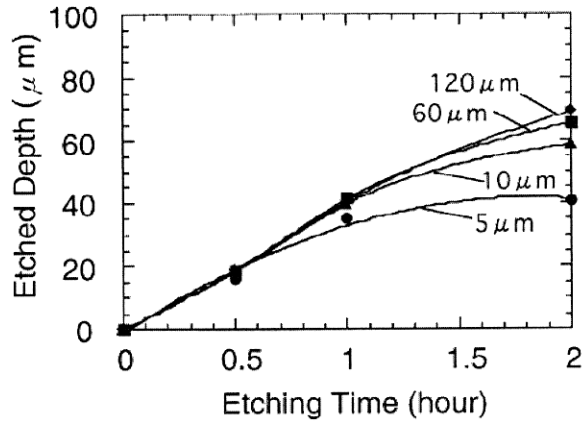


Figure 6 Etched depth as a function of the etching time and different opening widths of the mask.

## Base angle vs. mask opening width

Figure 7 showed the base angle of the etch profile as a function of mask opening width. The sample was etched for 60 min at the optimum condition. The base angle depends on the chemical compositions of the sample. In the case of fused silica, the base angle is vertical at the mask opening width from  $5\ \mu\text{m}$  to  $100\ \mu\text{m}$ . On the other hands, the base angle is close to vertical as the mask opening width decreases in the etching of Pyrex glass (See Figure 7A and B). It means the quantity of nonvolatile products deposit onto the side wall depends on area exposed to the ion flux.

We note that the base angle depends on the mask profiles as shown in figure 8. Their mechanisms are summarized in the figure. When a tapered mask is utilized, V shape is formed. The reason is considered as the reflection of ion and/or the mask erosion caused by the mask etching. Vertical mask profile mask profile is needed to obtain vertical etch profile.

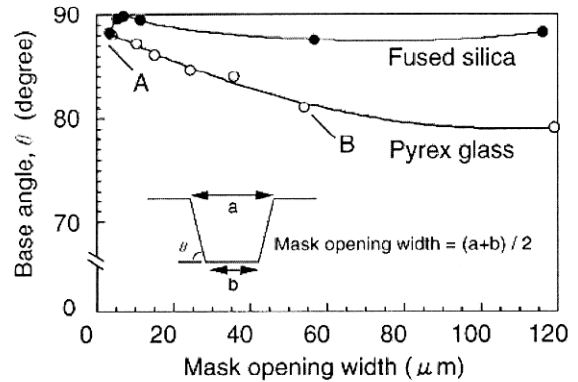
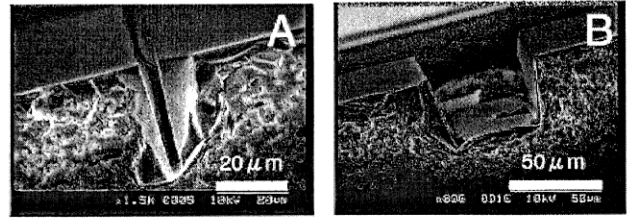


Figure 7 The base angle of etch profile as function of mask opening width.

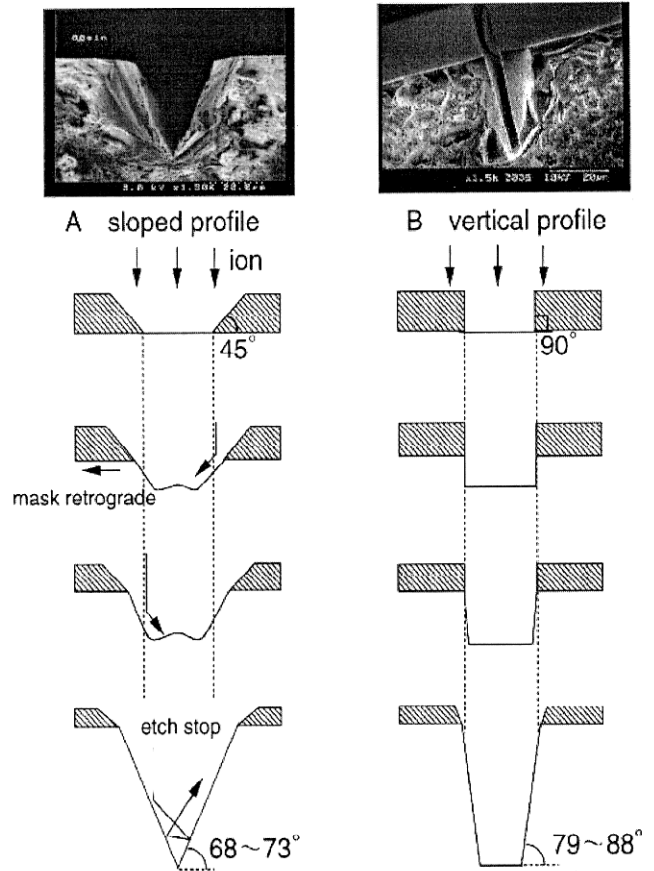


Figure 8 The model of etched profile in regard to the difference of cross section of mask.

### Through-out etching and its application

Figure 9 shows through-out etching of Pyrex glass ( $200\ \mu\text{m}$  in thickness) with the mask opening width of  $10\ \mu\text{m}$ . High aspect ratio ( $>10$ ) and directional etching (base angle  $\sim 88^\circ$ ) were achieved with mask opening width below  $20\ \mu\text{m}$  owing to the minimized deposition of nonvolatile products. As shown in figure 10, the through-out etching of large pattern can be made by scooping out unwanted parts. If standard method without the scoop out is applied to the fabrication, the structure has base angle of  $\sim 80^\circ$ .

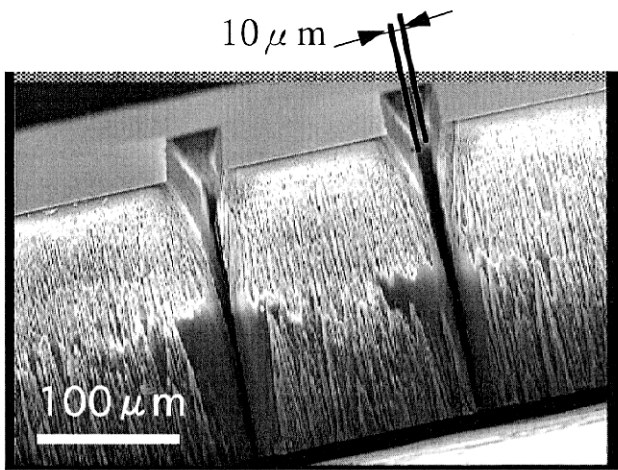


Figure 9 The through-out etch of Pyrex glass ( $200\ \mu\text{m}$  in thickness) by deep RIE (mask opening is  $10\ \mu\text{m}$ ).

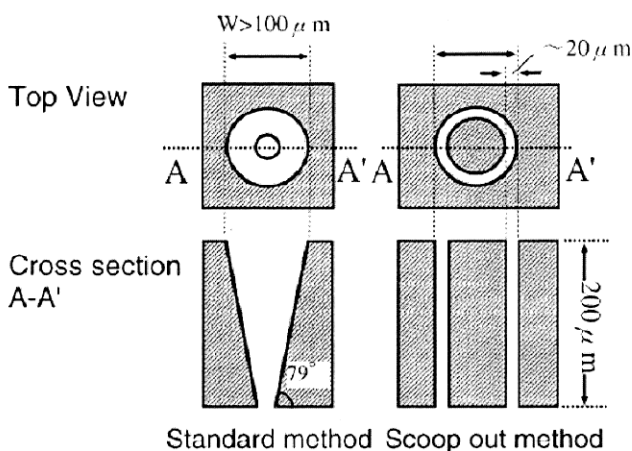


Figure 10 The scoop out etching for through-out etching of Pyrex glass.

Our etching techniques can be applied to fabricate an electrical feedthrough in Pyrex glass plate used for packaged microsensor [1]. The electrical feedthrough from a cavity was made through glass holes as shown in figure 11. Our technology makes it possible to fabricate fine pitch holes in Pyrex glasses compared with conventional methods as sand blast erosion.

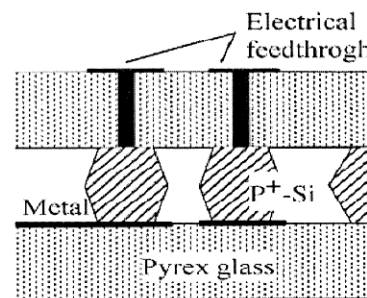


Figure 11 An example of the electrical feedthrough from the packaged cavity.

### CONCLUSIONS

We have developed a deep reactive ion etching of Pyrex glass in  $\text{SF}_6$  plasma. High etch rate ( $\sim 0.6\ \mu\text{m}/\text{min}$ ) and smooth surface ( $R_a \sim 4\ \text{nm}$ ) were demonstrated. The high etch rate is due to the use of both large capacity turbo molecular pump to decrease the deposition of reaction products on the wafer and a strong permanent magnet (Sm-Co) to densify plasma. These advantages allow to etch materials which produce nonvolatile reaction products. Vertical etch profile (base angle  $\sim 88^\circ$ ), high aspect ratio ( $>10$ ) and through-out etching of Pyrex glass ( $200\ \mu\text{m}$  in thickness) were achieved when the mask opening is narrower than  $20\ \mu\text{m}$ . We also find out the base angle of etch profile depends on the mask opening width of exposed parts.

### ACKNOWLEDGEMENTS

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## REFERENCES

- [1] M. Esashi "Encapsulated micro mechanical sensors" *Microsystem Technologies* 1, 2-9, (1994).
- [2] S. Ronggui, G. C, Righini "Characterization of reactive ion etching of glass and its applications in integrated optics" *J. Vac. Sci. Technol. A* 9 (5), 2709-2712, (1991).
- [3] S.Kong, M.Minami and M. Esashi "Fabrication of reactive ion etching systems for deep silicon machining" *T. IEE Japan* 117-E, 10-13 (1997).
- [4] J. Hopwood "Review of inductively coupled plasmas for plasma processing" *Plasma Sources Science and Technology*, 1, 109-116 (1992).
- [5] S.N.Wang, X.H.Li, K.Wakabayashi, M.Esashi"Deep reactive ion etching of lead zirconate titanate using sulfur hexafluoride gas"*J.Am.Ceram.Soc*,82[5] 1339-41(1999).
- [6] T.Oosato, M. Esashi "Quartz resonating gyroscope (Development of quartz micromachining by ICP-RIE)" *The 16 th Sensor Symposium, kawasaki*, pp78(1998).( in Japanese).
- [7] T. Abe, M. Esashi "One-chip multi-channel quartz crystal microbalance(QCM) fabricated by Deep RIE" *Proc. of Transducers '99*, pp 1246-49 (1999).