Sensors and Materials, Vol. 15, No. 3 (2003) 137–145
MYU Tokyo

S & M 0515

Glass Etching Assisted by Femtosecond Pulse Modification

Chienliu Chang*, Takashi Abe[1,2] and Masayoshi Esashi[3]

Venture Business Laboratory, Tohoku University
[1]Graduate School of Engineering, Tohoku University; [2]PRESTO, JST
[3]New Industry Creation Hatchery Center, Tohoku University
01 Aza-Aoba, Aramaki, Aoba-ku, Sendai 980-8579, Japan

(Recieved July 18, 2002; accepted January 28, 2003)

Key words: femtosecond laser, micromachining, etching, Pyrex glass, modification

In this paper the etching of Pyrex glass assisted by femtosecond pulses is reported. The process consists of 2 steps: (1) irradiating the glass by focused pulses from a femtosecond laser, (2) etching the glass in diluted HF (hydrofluoric acid) a solution. The glass can be modified without cracking by applying a femtosecond laser with a very low intensity. The etched depth of Pyrex glass increases from 12 to 131 μm with femtosecond laser irradiation from 0 to 1170 kJ/cm^2, and etching in a 5% HF solution for 230 min. This technique provides an alternative way of achieving a high etching rate of Pyrex glass for applications to micro-electro-mechanical systems (MEMS), and of forming suspended structures on the surface of a Pyrex glass substrate by using modified glass as a sacrificial layer.

1. Introduction

Recently, there has been a great demand for the miniaturization of electronics, machines, and optics. It is well known that glass is one of the best candidates for packaging in microelectronics, optoelectronics, and micromachining. In MEMS, Pyrex glass is a very attractive material for packaging, because of its low thermal and electrical conductivities, low thermal expansion coefficient, and good mechanical strength. Particularly, silicon can be anodically bonded to Pyrex glass, but not silica glass. In general, sensors or actuators are fabricated on a silicon substrate, and then the silicon substrate is anodically bonded to

*Corresponding author, e-mail address: clchang@mems.mech.tohoku.ac.jp

Pyrex glass to create devices with electrical interconnections.[1] This makes micromachining of Pyrex glass a very important issue for applications to MEMS packaging. On the other hand, prototype production is time-consuming for mask fabrication, but laser machining provides a maskless approach to the micromachining of transparent dielectrics, such as glass. Several researchers studied laser ablation using CO_2 and UV laser.[2–5] However, because the band gap of transparent dielectrics, such as quartz, sapphire, and optical fibers, is large for the photon energy of UV and visible lasers, it is difficult to machine them.[6]

Currently, femtosecond (fs) laser pulses can deposit energy in solids rapidly and precisely. The light is absorbed by electrons, and the optical excitation ends before the lattice is perturbed.[7] Because of the instantaneous direct evaporation of irradiated material, ultrashort laser pulses which minimize thermal damage are often used to ablate materials.[7–11]

At an ultrahigh intensity of $I > 10^{12}$ W/cm^2, ablation of transparent dielectrics occurs via multiphoton absorption causing morphological changes and crack generation.[12,13] The energy deposition initiated by multiphoton absorption can induce a selective reaction in the vicinity of the focused point of the laser beam where the photon density is sufficiently high.[14] Furthermore, the tightly focused 100 fs laser pulses initiate micro-explosions inside the transparent dielectrics at intensities approaching 10^{21} W/m^2.[15] In contrast with laser ablation, the material modification induced by the laser without cracking provides an attractive micromachining alternative using glass as the material for the sacrificial layer. For example, Fig. 1 schematically shows the process flow of a microcantilever using modified glass as sacrificial material, so laser modification can simplify the surface micromachining in this way. Besides, bulk modification of the optical fibers is possible using the self-channeled plasma induced by the femtosecond laser.[16] Similarly, the use of femtosecond pulses induces a refractive index change for forming the optical waveguide or gratings in bulk glasses.[17–19] Because $Li_2O \cdot SiO_2$ soluble in diluted HF acid precipitates in bulk glass by the irradiation of femtosecond pulses,[14] three-dimensional microchannels inside the transparent material can be formed by subsequent wet etching.[20,21]

In this paper we propose a method of modifying a glass using a femtosecond laser without generating cracks and drilling the glass by wet etching at a higher rate. Because the laser intensity applied to irradiate Pyrex glass is much less than the optical damage threshold,[15,22,23] Pyrex glass is modified without generating any cracks. Since modified Pyrex glass can be drilled at a high etching rate, the metallic interconnections between an IC chip and silicon transducers can possibly be formed inside Pyrex glass by subsequent electroplating.[1] Another application of modified Pyrex glass is it can be used as a sacrificial material in surface micromachining as shown in Fig. 1. This is not only a novel concept, but has the potential to be a unique technique for MEMS packaging.

2. Experimental Procedures

Figure 2 schematically shows the process flow for the experiment. A 320-μm-thick wafer of Pyrex glass was prepared and cleaned (Fig. 2(a)). First, a metal film (Cr: 50 nm, Au: 100 nm) was sputtered onto the glass surface (Fig. 2(b)). Next, a femtosecond laser irradiation was focused on the back surface of the metal film (Fig. 2(c)). The irradiated

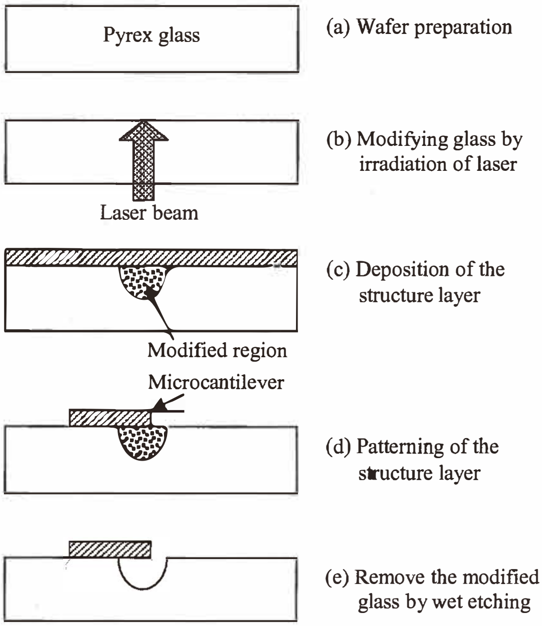


Fig. 1. Process flow of a microcantilever by using modified glass as sacrificial material.

area on the metal film was ablated and peeled off after the first few shots. The etching window (the hole on the Cr/Au layer) was opened simultaneously as shown in Fig. 2(c'). The remaining area on the metal film was used as an etching mask during the subsequent wet etching of HF solution. Masking of the Pyrex surface using conventional photoresists is not possible since they are not resistant to HF. The flatness of the Pyrex glass is maintained because of the Cr/Au mask which benefits from the subsequent bonding or lithographic processes. Some ablated spots on the metal film can be used as alignment marks in subsequent lithographic processes.

At this stage, cracks had not been generated on the irradiated surface of the Pyrex glass, and the modified region of the irradiated glass was near the etching window. Wax was coated throughout the back surface of the glass substrate and also used as an etching mask to protect the substrate from the subsequent HF etching. Finally, the glass substrate was immersed in a 5% (weight) aqueous solution of HF acid, and then an etched hole formed as shown in Fig. 2(d). The etchant solution of 5% HF acid was kept at room temperature and stirred by a magnet stirrer at the rate of 10 rpm.

The experimental setup of the laser system is schematically shown in Fig. 3. The laser system of femtosecond pulses is a commercially available regenerative amplified Ti: Sapphire laser (Clark-MXR, CPA1000). It can be modified to electronically adjust the pulse length and energy. It generated and amplified 775 nm-wavelength pulses at a

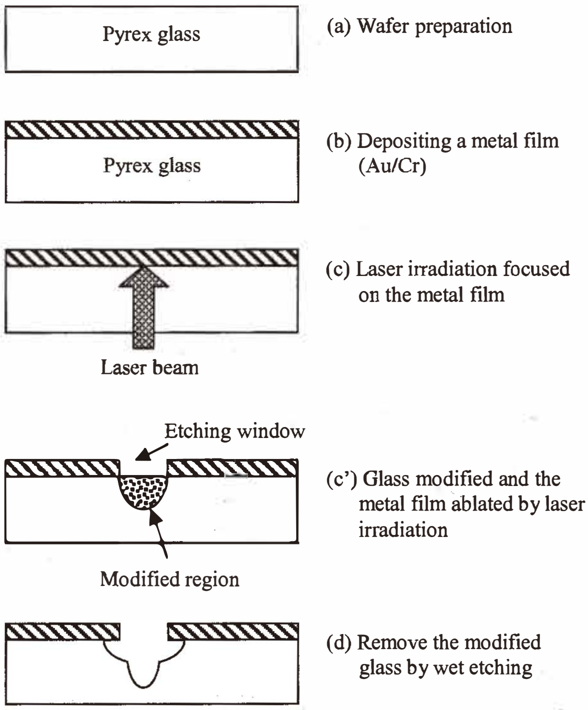


Fig. 2. Process flow of glass etching assisted by femtosecond pulse laser.

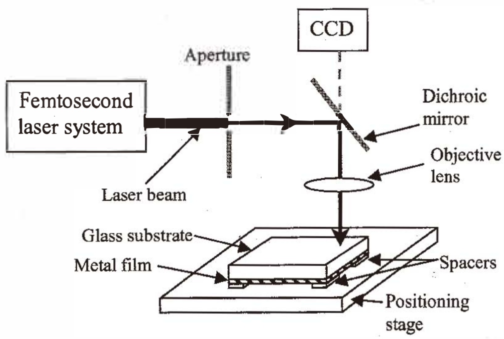


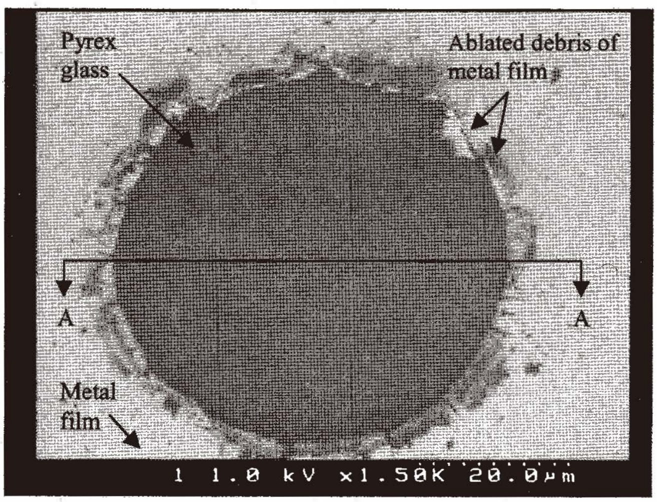
Fig. 3. Experimental setup of the laser system.

repetition rate of 1 kHz with a pulse width of approximately 150 fs (FWHM). In the experiment, the diameter of the aperture is set at 1 mm. The Pyrex glass was irradiated by focused fs-laser pulses through an objective lens with a focal length of 30 mm. This objective lens gave a spot diameter of approximately 56 μm on the metal film. The optical power through the objective lens was tuned to 0.018 W, so that the maximum laser power intensity was about 0.7 kW/cm^2 which was much less than the optical damage threshold, and micro-explosions were not generated inside the Pyrex glass in this experiment.[15,23] The workpiece, a Pyrex glass substrate coated with a metal film on the back surface, was mounted on an x-y-z positioning stage. Spacers with a height of 2 mm were inserted between the workpiece and the positioning stage to prevent severe redeposition of the ablated debris of the metal film.

3. Results and Discussion

Figure 4(a) shows a scanning electron microscope (SEM) photograph of an etching window ablated under the conditions of 0.018 W laser power, 1 mm aperture diameter, and 100 s irradiation time. The ablated debris of the metal film was redeposited on the periphery of the etching window. No cracks were observed on the glass surface in the etching window.

The surface profile on the centerline of the etching window was measured in Fig. 4(b) with a Stylus instrument (KLA/Tencor). As shown in Fig. 4(b), the height of ablated debris was about 300 nm and the metal thickness was about 150 nm. Since the depth of the

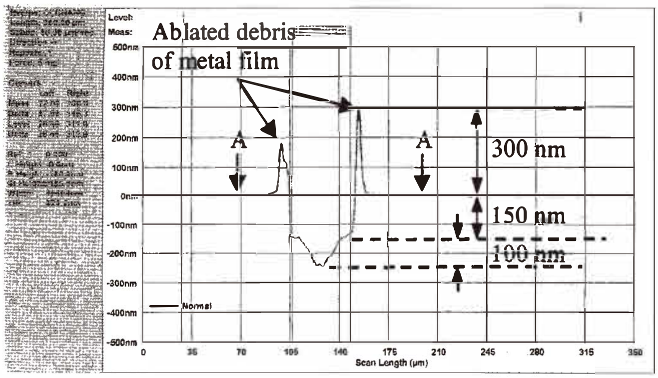


(a)

Fig. 4(a). SEM photograph of an etching window ablated under the conditions of 0.018 W laser power, 1 mm aperture diameter, and 100 s irradiation time.

etching window is within 100 nm, and the glass surface in the etching window is sufficiently flat and smooth for conventional lithography, this surface of the modified Pyrex glass is applicable for subsequent lithographic processes in surface micromachining.

Figure 5 shows the measured etched depths of modified Pyrex glass versus etching time for different irradiation times of the femtosecond laser. As shown in Fig. 5, the slope of a



(b)

Fig. 4(b). The measured surface profile of the etching window. The height of ablated debris was about 300 nm, metal thickness was about 150 nm, and depth of the etching window was about 100 nm.

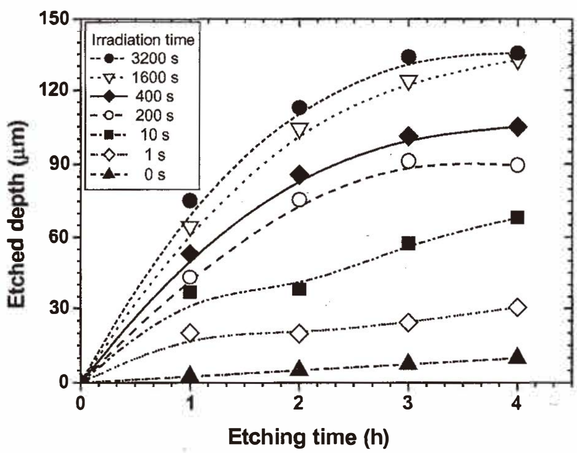
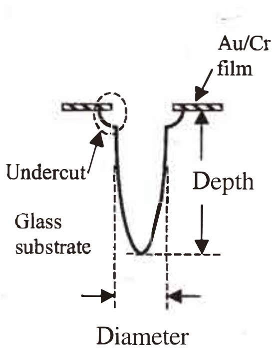


Fig. 5. Etched depth vs etching time for different irradiation times of the femtosecond laser.

curve means the etching rate in depth. The etching rate decreases gradually with longer etching time, and almost approaches a minimum after an etching time of about 3 h. That is to say, after an etching time of about 3 h, the modified glass is almost etched away completely and the unmodified glass is etched subsequently. The morphological profile will not change abruptly due to the low etching rate of unmodified glass. Although a longer irradiation time gives a deeper etched depth, an irradiation time longer than 1600 s gives insignificant change in etched depth.

Figure 6(a) shows the schematic profile of an etched hole. If glass is etched without any laser irradiation, a hole with a bowl-like shape will be etched on the glass surface due to the isotropic etching of HF solution.(24,25) For the same reason, the unirradiated glass near the periphery of the etching window was etched to have an undercut as shown in Fig. 6(a). Figure 6(b) shows the measured depth and diameter of the etched hole versus the irradiation time and the fluence after an etching time of 230 min which is longer than 3 h. As the discussion above, an etching time longer than 3 h gives insignificant change of morphological profile due to the low etching rate of unmodified glass. A longer irradiation time (or fluence) gives a deeper depth and a larger diameter of etched hole. When the irradiation time is increased to 1600 s (fluence=1170 kJ/cm^2), the maximum irradiation time for the efficient etching of modified Pyrex glass, the etched depth increased to about 131 μm. Under the same conditions and time of etching, the etched depth for unirradiated glass was only 12 μm.



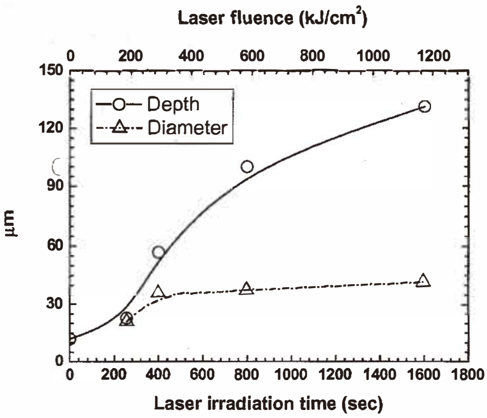
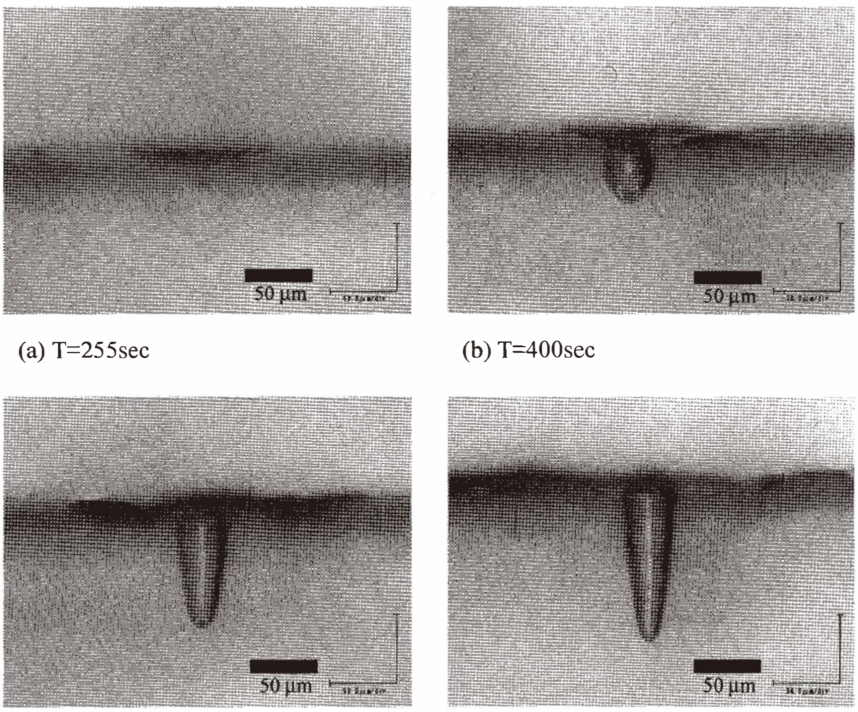


Fig. 6. (a) Schematic profile of an etched hole. (b) Depths and widths of the etched holes vs laser irradiation time and laser fluence. The etching time was 230 min. The etchant was 5% HF acid.

Figure 7 shows the micrographs of the profiles of etched holes after etching in 5% HF solution for 230 min. As seen in Fig. 7, the metal mask on the Pyrex surface was damaged. It was considered that the metal film was not sufficiently durable for long-time etching and some pinholes existed on the mask surface. The adhesion and quality of the mask film can be improved by optimizing the sputtering conditions.(25) By optimizing the etching solution and conditions, the etching rate and the smoothness of etched Pyrex glass can be increased.(24,25)

4. Conclusions

The modification of Pyrex glass without cracking using a femtosecond pulse laser has been achieved. Because the etching rate of modified Pyrex glass is much higher than that of unmodified Pyrex glass, etching deep holes in bulk Pyrex glass is possible. Furthermore, applying this etching technique to surface micromachining, it is possible to construct suspended microstructures using modified glass as a sacrificial layer on the surface of a Pyrex glass substrate.



(a) T=255sec (b) T=400sec

(c) T=800sec (d) T=1600sec

Fig. 7. Micrographs of the profile of etched holes after immersing in 5% HF solution for 230 min. (T= laser irradiation time).

Acknowledgements

This work is supported by a Grant-in-Aid (No. 13305010) from the Japanese Ministry of Education, Culture, Sports, Science and Technology. A part of this work has been performed in the Venture Business Laboratory, Tohoku University. The authors would like to thank Associate Professor M. Sasaki and Dr. Y. Haga of Tohoku University for their help with the setup of the laser system.

References

1 D.-W. Lee, T. Ono, T. Abe and M. Esashi: J. MEMS **11(6)** (2002) 215.
2 B. Braren and R. Srinvasen: J. Vac. Sci. & Technol. B **6** (1988) 537.
3 R. Nowak, S. Metev, G. Sepold and K. Grosskof: Glastech. Ber. **66** (1993) 227.
4 S. R. Jackson, W. J. Metheringham and P. E. Dyer: Appl. Surf. Sci. **86** (1995) 223.
5 P. E. Dyer, R. J. Farley, R. Giedl and D. M. Karnakis: Appl. Surf. Sci. **96–98** (1996) 537.
6 K. Midorikawa: O plus E, Sep. (1999) p.1130.
7 M. Lenzner, J. Krüger, W. Kautek and F. Krausz: Appl. Phys. A **68** (1999) 369.
8 R. Baehnisch, W. Gross, J. Staud and A. Menschig: Sensors & Actuators **74** (1999) 31.
9 K. Furusawa, K. Takahashi, S. H. Cho, H. Kumagai, K. Midorikawa and M. Obara: J. Appl. Phys. **87(4)** (2000) 1604.
10 M. Ye and C. P. Grigoropoulos: J. Appl. Phys. **89(9)** (2001) 5183.
11 S. Küper and M. Stuke: Appl. Phys. Lett. **54(1)** (1989) 4.
12 J. Krüger and W. Kautek: Appl. Surf. Sci. **96–98** (1996) 430.
13 S. Ameer-Beg, W. Perrie, S. Rathbone, J. Wright, W. Weaver and H. Champoux: Appl. Surf. Sci. **127–129** (1998) 875.
14 Y. Kondo, J. Qiu, T. Mitsuyu, K. Hirao and T. Yoko: Jpn. J. Appl. Phys. **38(10A)** (1999) L1146.
15 E. N. Glezer and E. Mazur: Appl. Phys. Lett. **71(7)** (1997) 882.
16 S. H. Cho, H. Kumagai, I. Yokota, K. Midorikawa and M. Obara: Jpn. J. Appl. Phys. **37(6B)** (1998) L737.
17 K. Miura, J. Qiu, H. Inouye, T. Mitsuyu and K. Hirao: Appl. Phys. Lett. **71(23)**, (1997) 3329.
18 R. Waki, M. Watanabe, K. Yamasaki, A. Marcinkevincius, S. Juodkazis, S. Matsuo and H. Misawa: Ext. Abstr. 48th Spring Meet. Jpn. Soc. Appl. Phys. 30-a-ZF-4 (JSAP, Tokyo, 2001) p. 1122.
19 J. Qiu, T. Nakaya, J. Si and K. Hirao: Ext. Abstr. 48th Spring Meet. Jpn. Soc. Appl. Phys. 30-a-ZF-3 (JSAP, Tokyo, 2001) p. 1122.
20 A. Marcinkevicius, S. Juodkazis, M. Watanabe, M. Miwa, S. Matsuo, H. Misawa and J. Nishii: Optics Letters **26(5)** (2001) 277.
21 S. Matsuo, A. Marcinkevicius, R. Waki, K. Yamasaki, M. Watanabe, V. Mizeikis, S. Juodkazis and H. Misawa: 8th Symposium on "Microjoining & Assembly Technology in Electronics" (JWS, Yokohama, 2002) p. 489.
22 J. Wang, H. Niino and A. Yabe: Appl. Surf. Sci. **154–155** (2000) 571.
23 R. A. B. Devine, R. Dupree, I. Farnan and J. J. Capponi: Phys. Rev. B **35(5)** (1987) 2560.
24 T. Diepold and E. Obermeier: J. Micromech. Microeng. **6** (1996) 29.
25 S. Shoji and M. Esashi: Tech. Digest of the 9th Sensor Symp. (IEE of Japan, Tokyo, 1990) 27.