

## New Deep Glass Etching Technology

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**Summary:** A new masking technology useful for wet etching of glass, to a depth of more than 300  $\mu\text{m}$ , is reported; multilayers of metal in combination with thick SPRT220 photoresist, are used. This new method was successfully developed for fabricating a 200  $\mu\text{m}$  thick diaphragm for a micro peristaltic pump. Various mask materials, which can be patterned by standard photolithography and metal etching processes, were investigated. The main advantage of this newly developed method was the application of hydrofluoric acid etching to create deep cavities with a uniform membrane without pinholes, and to minimize lateral undercutting of the glass.

**Keywords:** deep glass etching, masking technology, micro peristaltic pump

**Category:** 7 (Fluidic devices)

### 1 Introduction

Technology for the microfabrication of glass substrates is gaining in importance for the fabrication of MEMS devices. Glass has many advantages as a material for MEMS applications, such as good optical properties, high electrical insulation, and it is easily bonded to silicon substrates at a low temperature. Microfluidic devices such as micro pumps [1], micro pressure/flow sensors [2], monolithic membrane valve/diaphragm pumps [3], and micro polymerase chain reaction (PCR) devices [4-7], have been developed with integrated glass elements. Being transparent under a wide wavelength range ( $\sim 300$ -2000 nm for Pyrex), glass is a prime candidate for incorporation into micro bioanalytical devices where optical detection of the bioanalytes is used, for instance, micro capillary electrophoresis ( $\mu\text{CE}$ ) devices [8-11].

Deep glass etching methods have been investigated extensively, thus yielding an increased utilization of glass in MEMS. These technologies include wet chemical etching [12-18], deep reactive ion etching (DRIE) [19], and powder blasting [11,20-21]. DRIE can provide high aspect ratios of the order of 10 [19]; however, the etching rate is relatively low. Powder blasting can provide a fast etching rate of 25  $\mu\text{m}/\text{min}$ , but the process requires special facilities and the surface roughness is usually about 1  $\mu\text{m}$  [20].

Wet glass etching with hydrofluoric acid (HF) is the most widely used method because the etching rate is fast and a large quantity of glass wafers can be processed simultaneously. The most widely used masks for glass etching in HF are photoresist [9,12-13,18] and Cr/Au photoresist combinations [6,8]. However, the formation of pinholes through defects within the metal mask is a notorious problem. This becomes especially severe when deep etching is

required. Until now the etching depth has been limited to below 50  $\mu\text{m}$ . A second problem found for HF etching is the fast undercutting of the Cr mask, which leads to a more rapid lateral etching of the glass than vertical etching. This leads to a poor aspect ratio generally smaller than 1 for etched cavities.

Many other materials, such as SU-8 [13-14], anodically bonded silicon [15-16], polysilicon [3,13], silicon carbide [10,17], amorphous silicon [5,8,10,17], electroplated gold [13], and their combinations have been employed to solve these etching problems. The removal of SU-8 after the hardbake required by deep etching was found to be difficult and time consuming. Etching through a silicon mask is required before it is anodically bonded onto the glass. This silicon etching process normally takes more than 10 hours for a 500  $\mu\text{m}$  thick 4 inch wafer. Depositions of silicon-based thin film masks on the glass have a risk of contaminating the facilities by sodium ions from the glass. Therefore these processes are not generally feasible in most microfabrication laboratories.

Although the application of metal masks, for deep etching with HF, has the two problems mentioned above, the approach is simple and compatible with standard microfabrication facilities. In this work, a novel glass etching mask, consisting of a multilayer of metal and the thick photoresist, was investigated to eliminate pinholes formed in the Pyrex glass (Pyrex 7740, Schott) and to minimize the undercutting during deep etching in HF. This etching mask can be patterned by standard photolithography and a metal etching process.

### 2 Investigation of Masking Methods for Deep Glass Etching

To eliminate the pinholes when an etch depth of 300  $\mu\text{m}$  on a 500  $\mu\text{m}$  thick Pyrex wafer is required, 4

types of mask with different thicknesses were investigated. These are Cr/Au (60/500nm) covered with Shipley S1818 photoresist (2.2 $\mu$ m), Cr/Au/Cr/Au (60/400/60/400nm) covered with S1818 (2.2 $\mu$ m), and Cr/Au/Cr/Au (60/400/60/400nm) covered with Shipley SPRT220 photoresist (10 $\mu$ m and 20 $\mu$ m). The etchant was concentrated HF (48%). The etching results in Fig. 1 (a, b and c) show that pinholes of various densities and diameters appeared in the Pyrex, when the first three kinds of mask were used. Only the mask with Cr/Au/Cr/Au (60/400/60/400nm) and 20  $\mu$ m thick SPRT220 were used successfully to etch the Pyrex up to 300  $\mu$ m deep without pinholes appearing. The recipe for patterning this successful mask will be described in detail in section 3 with regard to the process used on the Pyrex for fabrication of a micro pump.

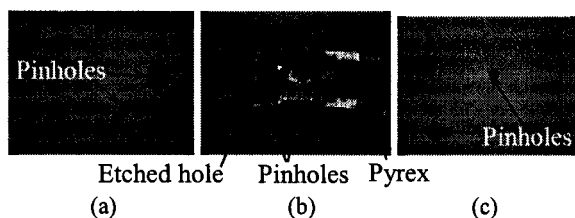


Fig. 1: Pinholes with various densities and diameters were formed in the Pyrex after 38 minutes of etching in 48% HF when different masks were used. (a) With a mask of Cr/Au (60nm/500nm) covered with Shipley S1818 (2.2 $\mu$ m). (b) With a mask of Cr/Au/Cr/Au (60/400/60/400nm) covered with S1818 (2.2 $\mu$ m). (c) With a mask of Cr/Au/Cr/Au (60/400/60/400nm) covered with SPRT220 (10 $\mu$ m).

Fig. 2 shows an etched Pyrex chip in a 500  $\mu$ m thick substrate using a mask of Cr/Au/Cr/Au (60/400/60/400nm) with 20  $\mu$ m thick SPRT220 for a micro pump PCR chip [22]. The through holes such as the inlet, the outlet and wire bonding holes were etched from both sides of the wafer simultaneously. The pump membrane was only etched from the top. The pump membrane is larger than 6 cm<sup>2</sup> and 200  $\mu$ m thick and there are no pinholes visible under the microscope. The results indicate that this new masking technology for deep etching of glass is able to prevent pinholes forming. This mask was patterned with standard photolithography process and metal wet etching processes.

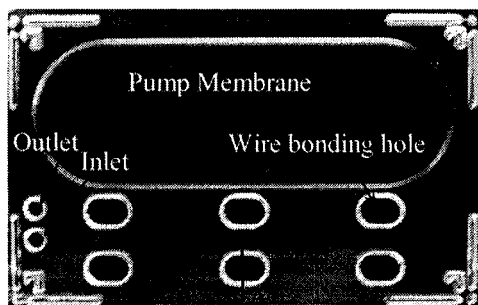


Fig. 2. A Pyrex chip etched in 48% HF for a micro peristaltic pump using the novel mask. The etch depth is 300  $\mu$ m. The total chip size is 44 mm $\times$ 27 mm.

In order to investigate the undercutting using this novel mask, a 750  $\mu$ m thick Pyrex wafer was patterned on one side, using the same photolithographic mask used to pattern the top surface of the Pyrex shown in Fig. 2. The bottom of the Pyrex was protected by this novel mask. Fig. 3 shows an SEM photograph of an etched Pyrex wafer cut along the cross-sectional line A-A shown in Fig. 2. Fig. 3 indicates that the lateral undercutting is 420  $\mu$ m while the etching depth is 329  $\mu$ m. So the extra undercutting of the Pyrex, in addition to that expected by an isotropic etching, is less than 100  $\mu$ m. The aspect ratio of isotropically etched Pyrex, defined as the ratio of etching depth to the lateral undercutting, is 0.78. This is better than the result of 0.66 reported in [15] using the anodically bonded silicon as the mask.

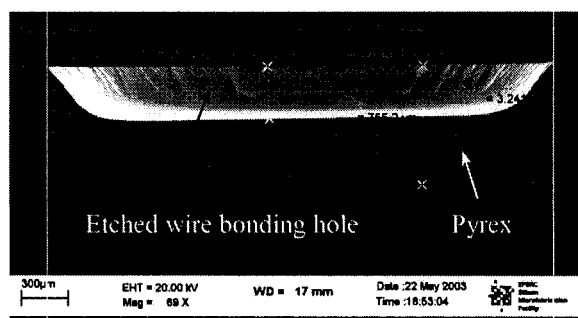


Fig. 3. Cross-sectional view of a 329  $\mu$ m deep wire bonding hole etched in 750  $\mu$ m thick Pyrex glass using the novel multilayered mask. The width of window on the mask is 2.4 mm, the lateral undercutting is 420  $\mu$ m.

### 3 Fabrication Process for A Micro Pump

The novel masking technology for deep glass etching was utilized to fabricate a micro peristaltic pump to be integrated into a PCR chip [22]. The fabrication process for the micro pump includes two parts: the process on the Pyrex wafer and the process on the silicon wafer. After this, the two wafers were bonded together by anodic bonding technology.

The fabrication process on a 500  $\mu$ m thick Pyrex wafer is illustrated in Fig. 4. The process starts with the etching of alignment marks (1  $\mu$ m deep) in 7:1 buffered hydrofluoric acid (BHF), on both sides at the same time, using 2.2  $\mu$ m thick Shipley S1818 photoresist as the mask (a). After stripping off the S1818 photoresist, a multilayer of Cr/Au/Cr/Au (60/400/60/400nm) was evaporated on both sides (b). Two layers of Shipley SPRT220 photoresist were then spun on the upper side of the Pyrex wafer. After spinning the first layer of 10  $\mu$ m thick, the photoresist was softbaked for 5 minutes at 95  $^{\circ}$ C on a hotplate. Another layer of 10  $\mu$ m thick of SPRT220 was spun and softbaked using the same process parameters as the first layer. The SPRT220 photoresist was patterned to define the pump membrane and inlet/outlet holes (c). The SPRT220 was then hardbaked at 115  $^{\circ}$ C for 10 minutes in an oven. On

the bottom of the Pyrex, the same processes of spinning, softbaking and patterning of SPRT220 were carried out (d). However, the softbaking of the resist was carried out in an oven at 85 ° C for 15 minutes for the first layer and 30 minutes for the second layer. After the patterning of the photoresist, another hardbake at 115 ° C was done for 50 minutes in the oven. After that, the Cr/Au/Cr/Au multilayer was etched sequentially in Au etchant and Cr etchant (e). Finally, the wafer was immersed in 48% HF for 38 minutes to achieve an etch depth of 300 μm and a 200 μm thick pump membrane (f). During the etching step, gentle stirring was applied to help the removal of the generated salt and to provide a uniform HF concentration at the glass surface thus aiding in the reduction of the surface roughness of the glass membrane. When the etching was complete, the SPRT220 was stripped off. The Cr/Au/Cr/Au multilayer was stripped off sequentially by Au etchant and Cr etchant (g). Finally, another layer of Cr (20 nm) was evaporated on the bottom of the Pyrex wafer and then patterned by photolithography and wet etching in Cr etchant (h).

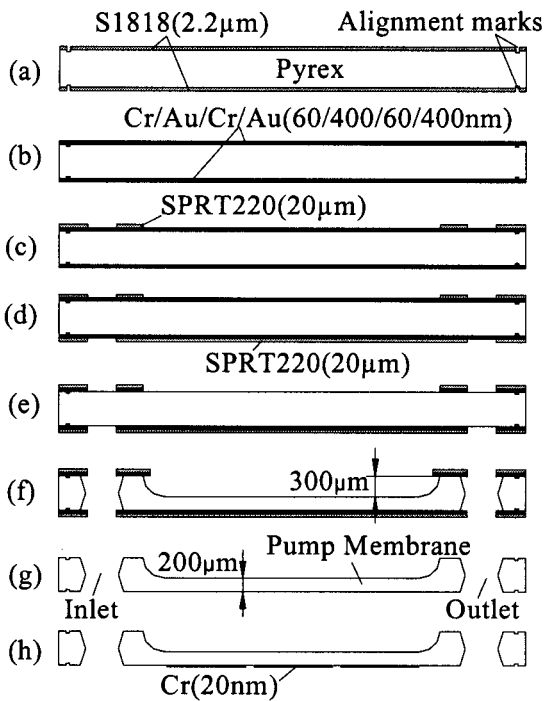


Fig. 4. Fabrication process on the Pyrex wafer

The fabrication process on a 500 μm thick silicon wafer begins by growing a layer of silicon dioxide (100 nm) and depositing a layer of LPCVD silicon nitride (160 nm) on the silicon substrate. SPR510 photoresist was patterned on the wafer, and then the Si<sub>3</sub>N<sub>4</sub>/SiO<sub>2</sub> layers were etched by reactive ion etching (RIE). The photoresist was stripped off before anisotropic etching of silicon was carried out in 30% KOH solution at 70 ° C to fabricate 250 μm deep connection channels. The process on the silicon ended with the stripping of Si<sub>3</sub>N<sub>4</sub> and SiO<sub>2</sub>.

Anodic bonding was carried out when the silicon wafer and the Pyrex wafer were aligned and put together on top of a hotplate at a temperature of 400 ° C. A voltage of 400 Volts was applied to the Pyrex and silicon via anodic and cathodic electrodes connected to the silicon and Pyrex, respectively. The bonding was prevented in the areas where there was Cr patterned on the bottom of the Pyrex [1]. The Newton rings shown in Fig. 5 indicate that three pump chambers were formed. As the final step, three PZT discs were glued on the top of these three pump membranes by conductive glue (Ablebond 84-1LMISR4, Ablestik, Rancho Dominguez, CA). Fig. 6 shows a complete micro peristaltic pump with the glued PZT discs (PZT-5H, Morgan Electro Ceramics) and wire leads.

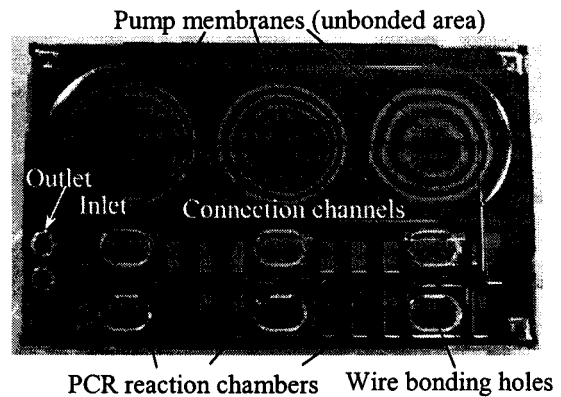


Fig. 5. A micro machined pump chip (before gluing the PZT discs onto the pump membranes).

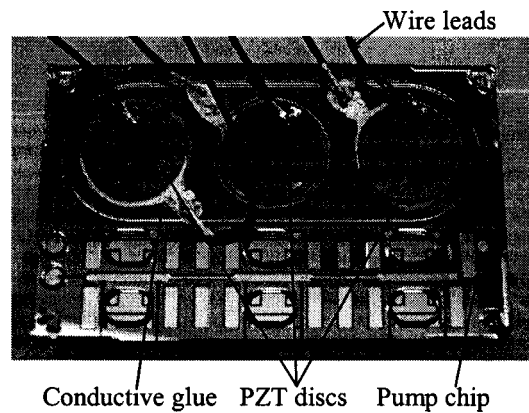


Fig. 6. A complete micro pump with three PZT discs glued onto the pump membranes using conductive glue.

#### 4 Conclusion

A novel masking technology for deep etching of glass employing standard photolithography and metal etching processes was investigated in this work. The results indicate that a mask of multilayered metals (Cr/Au/Cr/Au) combined with thick SPRT220 photoresist makes etching of glass to a depth of 300 μm possible. Pinholes are eliminated in the glass and undercutting of the glass is also greatly minimized by this masking technology. This technology was

successfully applied to fabricate the components of a micro peristaltic pump on a Pyrex substrate.

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