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Fabrication of Glass-based Microfluidic Devices with Photoresist as Mask

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Introduction

Microfluidic systems have become increasingly well-known in different fields of studies. In recent years many new commercialized microfluidic products have emerged in the market. Microfluidic devices are becoming one of the most dynamic parts of BioMEMS technology. The main applications of microfluidics are medical diagnostics, genetic sequencing, chemistry, drug delivery, and proteomics.

Depending on applications and suitability, different types of materials can be used as the substrate for the microfluidic devices such as silicon [1], glass [2, 3], SU-8 [4], polydimethyl-siloxan (PDMS) [5] and Poly methyl methacrylate (PMMA) [6]. However, biocompatibility of the substrate material is very important in vivo and in vitro analyses. Glass as a well-known material with minimum chemical reaction to the body is used in this paper. Due to the low cost and simplicity, commercially available microscopic slides have been used.

Numerous fabrication techniques for microfluidic devices have been reported. Fabrication of microchannels as the main parts of the microfluidic systems plays an important role in operation of the entire system. Different techniques can be utilized for fabrication of microchannels. SU-8 as a low cost negative resist is a famous material to make vertical and high aspect ratio structures. Using SU-8 as master mold and pouring PDMS on the master is another well-known method for microchannel fabrication [4]. Microchannels with vertical and precise walls can be fabricated using deep reactive ion etching (DRIE) on different substrates [7, 8]. Electrochemical etching also is used for making channels in silicon [9]. Despite all the techniques, wet etching is still used as low cost and simple method for fabrication of microfluidic devices. However, various masking techniques have been implemented to make microchannels using glass substrate. Different mask layers for chemically glass etching have been reported using different materials such as Cr, Cr/Au, polysilicon to deposit a layer as a mask on glass in order to make an open region for wet chemical etchant by different deposition methods such as CVD, LPCVD, sputtering or other methods which needs special clean room instruments [10-12].

In this paper we used methods that include coating the glass surface with photoresist, AZ 5214, by spinning the glass substrate on a spin-coater, and post baking followed by immersing the glass in an etchant with special concentrations of Hydrofluoric acid (HF), Ammonium Fluoride (NH4F), Hydrochloric acid (HCL) and DI water in a magnetic stirring bath. HF-based methods [13] usually result in a rough surface, but the special recipe consisting of HCI, Buffered Oxide Etchant (BOE) and DI water provides a smooth surface [14].

Cleaning procedure

Microscopic glass slides with easy accessibility were used as main material to fabricate the entire microfluidic chip. The standard glass slides with 25mm×75mm and 1mm thickness were utilized. The fabrication process starts with cleaning glass substrate using ultrasonic in acetone and methanol for 10 min respectively. Subsequently, the glass substrates were boiled in piranha solution (H_2SO_4:H_2O_2= 3:1) for 15 min. The slides were then immersed in deionized water for 5 min and dried using nitrogen gas. Finally, the cleaned glass substrates were put in a conventional oven for 20 min at 85 ºC.

Photolithography and post-baking

Photolithography is a very important process to achieve the desired photoresist thickness after spin coating. In this step UV exposure time, and hard baking temperature are crucial to achieve the maximum adhesion between photoresist and substrate. In order to accomplish these conditions, AZ5214E, a positive photoresist, was utilized. The resist was spun for 5 seconds at 500 rpm, followed by 20 seconds spin at different speeds (600, 700, 800 and 900 rpm) for result comparison. The photoresist was soft baked on a hotplate at 100ºC for 10 min, resulting moisture-free surface. The dried photoresist was UV-exposed in using a mask aligner at 365nm. Immediately after exposure the resist was subjected to a post-exposure bake on a hotplate at 100ºC for 3 min for adhesion promotion. Development was done in diluted AZ400k developer (DI water: AZ400k = 3:1) at room temperature with a development time of 3-4 min and rinsing in DI water subsequently. A post-bake at 160ºC for 90 min were applied on a hotplate in order to harden the photoresist against attacks of etchants.

Etching process

For producing microchannels, a wet etching process was performed. Proper mixture of etchants can greatly enhance photoresist resistant and etch rate. Initially, 10:1 BOE was prepared. HF-based etchants usually result in a rough surface. However, by adding HCL to BOE solution, a smooth surface is attained. The ratio of the BOE solution to HCL was 5:1. Putting the coated glass in this solution in magnetic stirring bath leads to early attack on some parts of photoresist just in less than 10 min. To overcome this problem, DI water was used to dilute the solution. By adding desired DI water to the solution, the resistivity time of the photoresist was increased up to more than 2 hours (DI water: BOE = 1:1).

For removing photoresist from the glass surface, ultrasonic agitation in acetone was used for 10 min. Subsequently, the same procedure as stated in cleaning procedure section for cleaning etched glass for bonding purposes was performed.

Bonding with UV curable glue

This method involves UV curable glue that is used to bond glass microfluidic chips at room temperature. The use of UV-curable glue is quick, easy, and inexpensive for glass substrates bonding. The glue with low viscosity which ensures formation of a thin layer after spinning was selected (NOA 71, Norland). A thin layer of UV glue was applied on the slide by spinning it at 4000 rpm for 20s. The etched glass slide containing the microchannels was then brought in contact with the glue surface of the plain slide to permanently bond two glass slides together.

Tubings and Connections

In order to complete fabrication of the device, the tubing connection using PDMS was produced. The next step was mixing 25 g of PDMS with 2.5 g (10%) hardening agent and pouring it into the dish. Next the petri dish was cured for 1h. For making holes through the PDMS, we used the typical needle and then cut the squares around each hole using a blade. The silicone glass sealant was used to adhere each square piece PDMS on the glass substrate.

Results and discussions

Glass slides are ideally suited for fabricating glass microfluidic devices due to their accessibility and high etch rate. The glass slides are naturally hydrophilic and the microchannels were made by etching process showed the same behavior (contact angle with water 18–20°).

Based on the previous reports, the photoresist etch mask can resist only for a short time, which is less than 5 min, in highly concentrated HF solutions (40%–49%). However, our results show that the etch mask fabricated by AZ 5214 can withstand attack of diluted buffered HF solution for more than 2h.

The optimum photoresist thickness was determined to be about 7 μm in 700 rpm in order to withstand the attacks of etchant solutions for up to 120 min. Different baking processes were used in this work. The primary purpose of baking is to removing moisture from the photoresist in order to avoid adherence of photoresist to the mask in mask aligner (prebake) and increase the surface adhesion (post-bake). In addition, heating affects on photoresist compounds to become a non-photosensitive product by changing chemical characteristics of photoresist. This can affect on exposure time too. Therefore, determining temperature and heating time have significant impacts on accuracy of the design. Different heating temperatures were utilized in order to optimize the photoresist patterning process for subsequent etching process. In particular, after photoresist spin coating on glass, the substrate was put on a hotplate at 100 ºC for 10 min. Subsequently after UV exposure the coated substrates were put on a hotplate for 3 min at 100 ºC for prebake procedure. Post-bake process was performed after developing the exposed regions at 160ºC for 90 min on a hotplate.

The wet etching process was performed in a plastic container using magnetic stirring plate. Different mixtures of NH_4F/HF/HCL/DI water were studied in order to achieve to a acceptable etch rate, smooth microchannel surface, no underlying glass etching effect, and clear glass slide in all regions without any signs of damages. Etching the glass can cause undercutting effect. Due to this effect width of the channels increase compare to the mask design. In order to compensate for undercutting effect of isotropic etching, determining glass etch rate is an important factor. We used scanning electron microscope (SEM) for etch rate measurements. Fig. 1 illustrates the depth of etch vs. etching time in 90min period. In this figure the depth of the microchannel was measured using SEM every 15 min by Au sputtering. The results shows the etch rate of 1.75 μm/min. Although the etch rate can be achieved to more than this amount with less dilution, however, the smoothness of the substrate surface, undercutting effect and also sedimentation limit this factor. Fig. 2 presents a microscopic view of the effect of etchant concentration with a diluted etchant and without dilution. The less-diluted etchant can cause sedimentation on the etching area which is a reason to avoid more etching because of blocking of the open region for further etching. This can affect the smoothness of the etched surface as well because of high etching rate in some parts and low in other regions. It shows that the edges of the microchannel walls are not so smooth when a non-diluted etchant was applied in comparison with the diluted etchant. This is because of the undercutting effect when the dilution is not enough. This figure also illustrates that the attacks against glass is more destructive especially on edges when no or less DI water is used. The dilution ratio was 2 part of DI water to 1 part of BOE: HCL=5:1. Fig. 3 shows the SEM view of a microchannel after 40 min wet etching. As can be seen the sharp edges and approximately smooth surface was achieved.

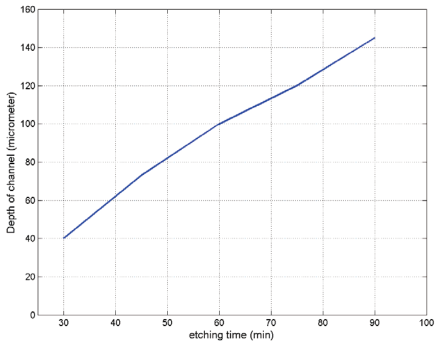
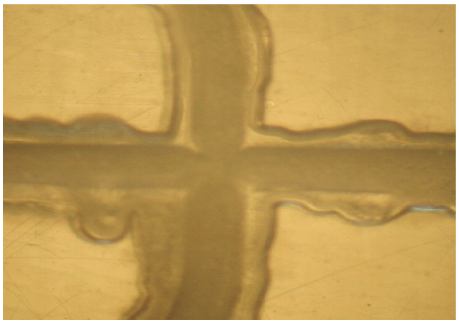
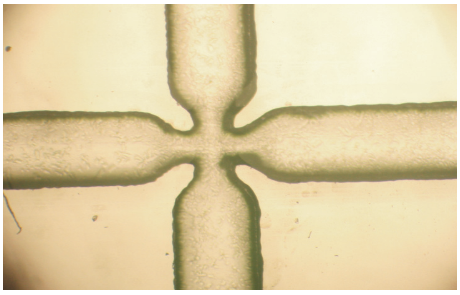


Fig. 1. Depth of channel vs. etching time



a)



b)

Fig. 2. Effect of the (a) non-diluted and (b) diluted etchant on side walls

The bonding was applied using UV glue method. The results for UV glue are shown in Fig. 4(a) by filling the channels with dye water. This figure illustrates no penetration of dye water to other areas after sequential experiments. We used high speed drilling machine with diamond drill bits for making holes through the glass before bonding.

Leaking from connections and tubings in this microfluidic device was eliminated using special PDMS cubic parts. We used 1cm/sec flow rate in microchannels to examine the connections. Fig. 4(b) shows the overall view of a microfluidic device. This figure shows the input and output channels have filled with dye water and the tubings are connected to the inlets and outlets without any leakage inside the channels and also via the connections. Also, it is clear that the UV glue has not clogged the micro-channels.

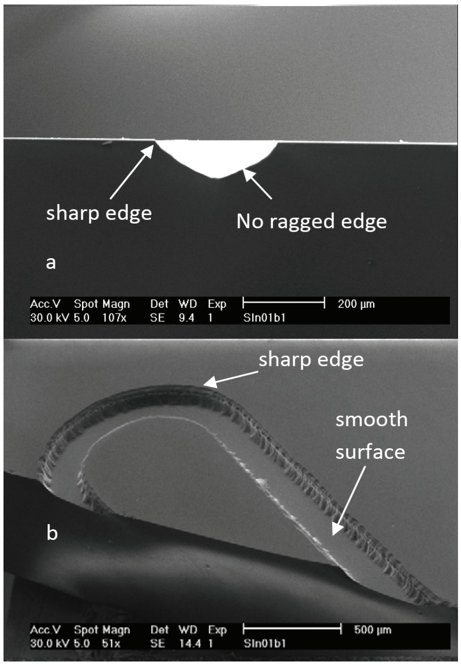
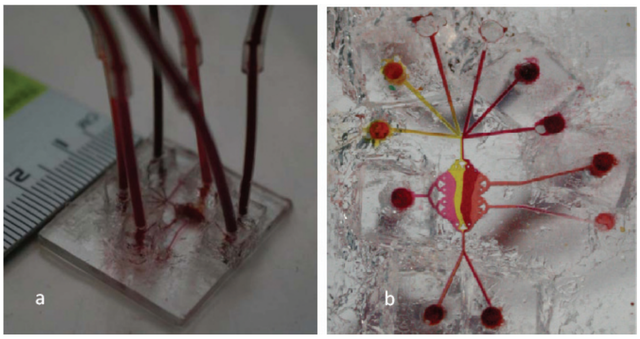


Fig. 3. SEM cross section view (a) and an angle view (b) of the microchannel



a) b)

Fig. 4. (a) tubings and connections and (b) dye water inside the microchamber

Conclusions

In this paper we present a simple and cost effective fabrication method for fabrication of microfluidic devices on glass substrate. This method uses typical microscopic glass slides as a substrate for fabrication of micro-channels with up to 150µm depth. The channel width range about 30µm to 350µm was fabricated. Using photo-resist as a mask led to gain precise results identical to other deposition methods which need sophisticated procedures and instruments. In particular, a photoresist based mask method was introduced for glass etching which can strongly resist against etchant attacks up to 2 hours, showing high adhesion properties on glass substrate for fabrication of microfluidic microchannels. A smooth channel surface with acceptable sharp wall edges was achieved using specific etchant solution by adding HCL to diluted BOE. The UV glue was used to achieve promising bonding results. The tubings and connections also were performed using PDMS. The results show no leakage from connections and no penetration from the microchannels to other non-etched regions with 1cm/sec flow rate.

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This paper presents a low cost method for etching of glass based microfluidic devices. Microchannels with the depth up to 150 µm were achieved by implementing a photoresist and wet etching process. In particular, a photoresist based mask method is introduced for glass etching which can strongly resist against etchant attacks up to 2 hours, showing high adhesion properties on glass substrate for fabrication of microfluidic microchannels. The width of the channels is determined by the width of the lines in photo-mask design and the rate of glass isotropic etching. The channel width range about 30µm to 350µm is fabricated. Commercially available inexpensive microscopic glass slides have been used as substrate. Achieving smooth and clear surface after wet etching process is an important factor for easily flowing fluid through channels and monitoring purposes. It is achieved by implementing special etchant with adding HCL in diluted BOE solution to get smooth and clear surface. The etch rate of the glass strongly depends on the concentration of the etchant. A mixture of different solutions with special ratios has been applied. Finally, typical UV curable glue is utilized for glass-glass bonding. Ill. 4, bibl. 14 (in English; abstracts in English and Lithuanian).

A. Bahadorimehr, B. Y. Majlis. Mikrohidrodinaminių įtaisų stiklo pagrindu su fotorezistu, kuris naudojamas kaip kaukė, gamyba // Elektronika ir elektrotechnika. – Kaunas: Technologija, 2011. – Nr. 10(116). – P. 45–48.

Pateiktas nebrangus mikrohidrodinaminių įtaisų stiklo pagrindu ėsdinimo metodas. 150 µm gylio mikrokanalai buvo gauti implementuojant fotorezistą ir drėgną ėsdinimo procesą. Fotorezistas naudojamas kaip kaukė stiklui ėsdinti. Jis gali būti atsparus ėsdiklui daugiau nei 2 val. ir išlaiko geras adhezijos savybes gaminant mikrohidrodinaminius kanalus. Kanalų plotis priklauso nuo fotokaukės linijų pločio ir stiklo izotropinio ėsdinimo santykio. Gautas kanalo plotis – nuo 30 µm iki 350 µm. Lygus ir švarus paviršius po drėgno ėsdinimo yra svarbus veiksnys užtikrinant sklandų skysčių tekėjimą mikrokanalais. Tai pasiekta naudojant specialų ėsdiklį ir pridedant HCL į atskiestą BOE tirpalą. Buvo naudojami įvairių sudėčių tirpalai. Il. 4, bibl. 14 (anglų kalba; santraukos anglų ir lietuvių k.).