FABRICATION OF GLASS-BASED MICROFLUIDIC DEVICES WITH PHOTORESIST AS MASK

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Abstract: This paper presents a low cost method for etching of glass based microfluidic devices. Microchannels with the depth up to 150µm 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.

Izdelava mikrofluidnih vezij na osnovi stekla s pomočjo fotorezista kot maske

Kjučne besede: mikrofluiden, fotorezist, steklo, mokro jedkanje

Izvleček: V članku je predstavljena poceni metoda za jedkanje mikrofluidnih naprav na stekleni podlagi. Izdelali smo mikrokanale z globino do 150µm z uvedbo fotorezista in mokrega jedkanja. Uporabljeni fotorezist zdrži jedkanje v mokrem jedkalu dolgo do 2 uri in hkrati kaže odlično oprijemljivost na stekleno podlago. Širino kanalov v steklu določimo s širino linij na maski in jedkalno hitrostjo izotropnega jedkanja, oz. spodjedkavanjem. Na ta način nam uspe izdelati širine kanalov od 30µm do 350 µm. Za podlago smo uporabili komercialno dosegljive steklene ploščice namenjene mikroskopiranju. Pomembno je, da po jedkanju dosežemo čisto in gladko površino, ki omogoči normalen pretok tekočine po kanalih. Le-to dosežemo z uporabo posebnih jedkal z dodatkom HCl v razredčeno raztopino osnovnega jedkala BOE. Jedkalna hitrost stekla je močno odvisna od koncentracije jedkala. Poskusili smo različne koncentracijske mešanice raztopin. Za končno zlepljenje stekelc smo na koncu uporabili UV lepilo.

1. Introduction

Microfluidic systems have become increasingly well-known in different fields of studies. In recent years so many new commercialized microfluidic products has been emerged in the market. Microfluidic devices are going to become one of the most dynamic part of the BioMEMS technology trend. The main applications of microfluidics are medical diagnostics, genetic sequencing, chemistry production, drug discovery, and proteomics.

Depending on applications and suitability, different types of materials can be used as the substrate for the microchannels 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 vitro analysis. Glass as a well-known material with the minimum chemical reaction issues is used in this paper. In terms of 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 a master mold and pouring PDMS on master is another well-known method for microchannel fabrication /4/. Microchannels with vertical and precise walls can be achieved using Deep Reactive Ion Etching (DRIE) on different substrates /7, 8/. Despite all the techniques, wet etching process is still used as a low cost and simple method for fabrication of microfluidic devices. However, various masking techniques are implemented to make a microchannel via glass substrate. Different mask layers for chemical wet etching of glass have been reported which use different materials such as Cr /9/, Cr/Au /10, 11/, polysilicon /12/ 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 /12/, sputtering or other methods which needs special clean room instruments. The method in this paper includes coating the glass surface with a photoresists, AZ 5214, by spinning the sample on a spin-coater, and post baking procedure of photoresist followed by immersing the glass in an etchant with special concentrations of Hydrofluoric acid (HF), Ammonium Fluoride (NH-

4F), 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 D.I. water provides a smooth surface /14/.

2. Experimental details

2.1. Cleaning procedure

The fabrication process starts with the glass substrate cleaning by ultra-sonication in acetone and methanol for 10 min respectively. Subsequently the glass substrates were boiled in piranha solution (H2SO4:H2O2= 3:1) for 15 min. The slides were then immersed in deionised water for 5 min and blow drying with nitrogen gas was applied later. Finally, the cleaned glass substrates were put in a conventional oven for 20 min at 85°C to demoisturize.

2.2. Photolithography and post-baking

Photolithography is a very important process due to the desire thickness of photoresist after spin coating, UV exposure time, and hard baking temperature to achieve the maximum adhesion between photoresist and substrate. In order to accomplish these conditions AZ5214, a positive photoresist, was utilized. Coating was obtained by spinning for 5 seconds at 500 rpm, followed by spinning for 20 seconds at different speeds to compare the results. The photoresist layer was soft baked on a hotplate at 100°C for 10 min, resulting moisture-free surface, allowing contact mode exposure. The dried photoresist was UV-exposed in a mask aligner at the wavelength of 365nm in hard contact mode for highest precision purposes. Immediately, after exposure the resist was subjected to a post-exposure bake on a hotplate at 100°C 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 160°C for 90 min were applied on a hotplate in order to harden the photoresist against attacks of etchants.

2.3. Etching process

For producing microchannels a wet etching process was performed. The proper mixture of etchant concentrations can be greatly enhanced the resistant time of photoresist and etch rate as well. First, 10 parts of saturated NH4F solution was mixed with one part of 49% HF to form 10:1 BOE solution. HF-based etches usually result in a rough surface. However, by adding HCL to BOE solution, a smooth surface is attainable. The ratio of the BOE solution and HCL was 5:1. Putting the coating glass in this solution with magnetic stirring was lead 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 100% DI water to the solution, the resistivity time of the photoresist was increased up to more than 2 hours.

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 were performed.

2.4. Bonding with UV curable glue

The method involves UV curable glue that can be used for glass microfluidic chips bonding at room temperatures. The use of UV-curable glue was found to be a quick, easy, and inexpensive method for attachment of glass substrates together. The glue with low viscosity which ensures formation of a thin layer after spinning was selected (NOA 71). A thin layer of UV glue was applied on this 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.

2.5. Tubings and Connections

In order to complete the entire device the tubing connection using PDMS was applied. Mixing 25 g of PDMS with 2.5 g (10%) hardening agent and pour it into the dish was the first step. Next the Petri dish was placed on a hot plate at 100°C and cure 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 in order to adhere each square piece PDMS on glass substrate.

3. Results and discussions

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 glass slides are naturally hydrophilic and the microchannels were made by etching process showed the same behavior (contact angle with water 18–20°).

AZ5214 is a thick positive photoresist which was coated with different speeds on glass substrates (600, 700, 800 and 900 rpm). The optimum 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 process was 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°C for 10 min. Subsequently after UV exposure the coated substrates were put on a hotplate for 3 min at 100°C for prebake procedure. Post-bake process was performed after developing the exposed regions at 160°C for 90 min on a hotplate.

The wet etching process was performed in a plastic container using magnetic stirring plate. Different mixtures of NH4F/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 about 150μm after 90 min of etching using diluted etchant. 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 of avoidance of 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. It 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.

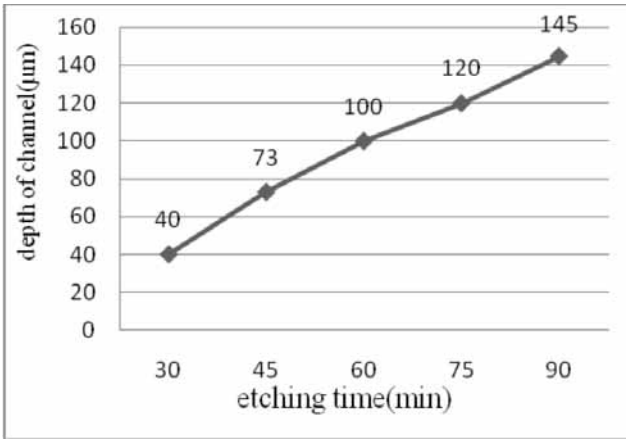

Fig. 1. Depth of channel vs. etching time

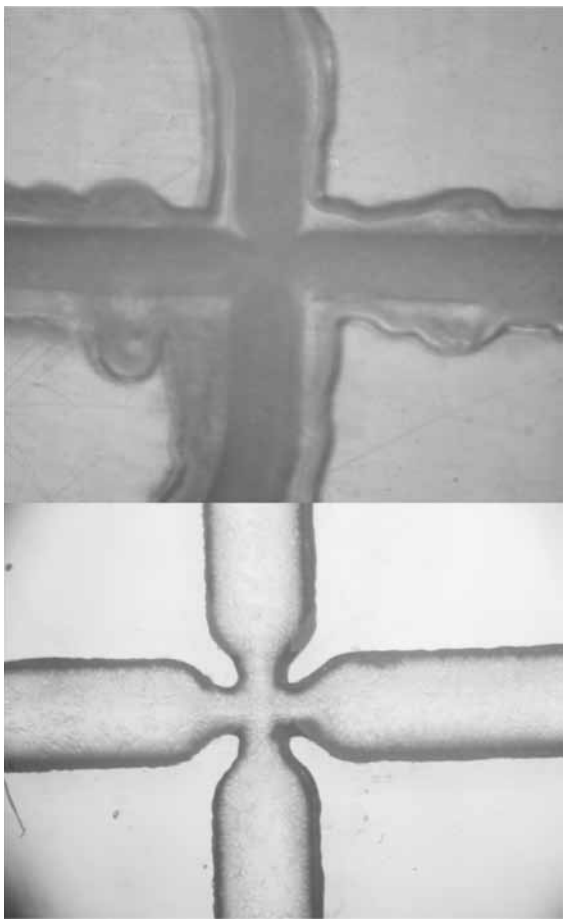

Fig. 2. Effect of the (a) non-diluted and (b) diluted etchant on side walls

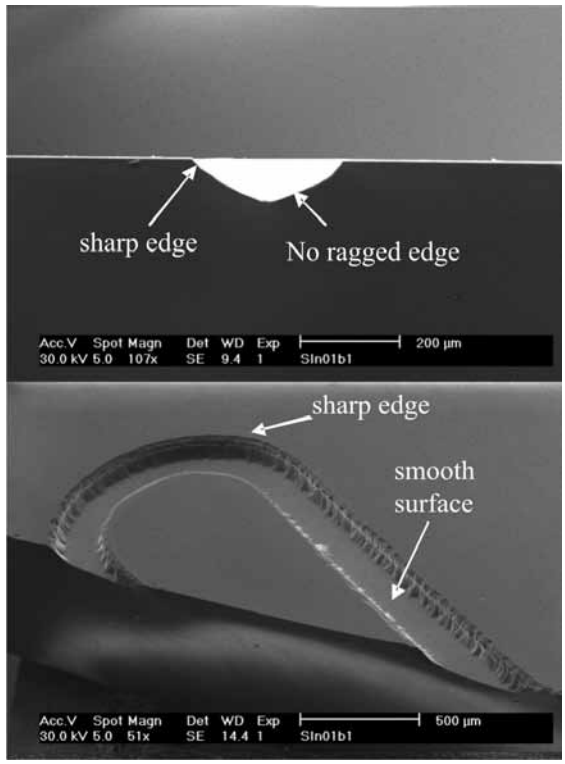

Fig. 3. SEM cross section view (a) and an angle view (b) of the microchannel

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. For making holes through the glass we used high speed drilling machine with diamond drill bits before bonding.

Leaking from connections and tubings in this microfluidic device was eliminated using special PDMS cubic parts. Fig. 4(b) shows the overall view of a microfluidic device. It 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.

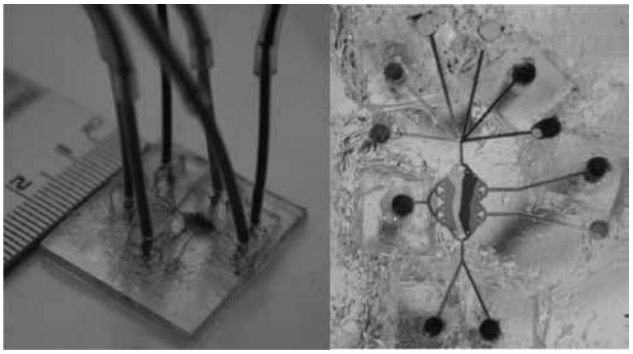

Fig. 4. (a)Tubings and connections and (b) Dye water inside the microchamber

4. Conclusions

In this paper we presented 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. Using photo-resist as a mask led to gain precise results identical to other deposition methods which need sophisticated procedures and instruments. 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.

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