MICROFLUIDICS IN GLASS: TECHNOLOGIES AND APPLICATIONS

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Key words: microfluidics, glass etching, bonding, dielectrophoretic filter

Abstract: The paper presents technological aspects of patterning and assembling of glass wafers for microfluidic applications as well as three applications of the developed technologies. Special masking layer (Cr/Au/Photoresist and a:Si/SiC/Photoresist) were design, fabricated and tested for deep wet etching of glass. As a result an 1mm-thick Pyrex glass wafer was etch-through using an a:Si/SiC/Photoresist mask. Also, a HF(49%)/HCl(37%) solution in ratio 10/1 was found to be optimal for achieving a good surface roughness of the generated surface. Experimental results regarding dry etching of Pyrex glass on ICP reactors show that vertical sidewall can be generated using C_4F_8/He as process gasses. The best result was achieved for silicon (single crystal) mask.

Microfluidic applications require bonding over metallization deposited on silicon wafer. Adhesive bonding can be a solution for direct assembling of glass-to-glass (especially at low temperature- below 200^{O}C). A special adhesive bonding technique, with SU8- negative photoresist applied by contact imprinting, was developed for glass microfluidic applications.

Design considerations, fabrication process and experimental results of microfluidic devices such as: dielectrophoretic (DEP) chip with bulk silicon electrodes, 3D DEP filter as well as a microfluidic chip for cell impedance spectroscopy are also presented.

Mikrofluidika v steklu: tehnologija in uporaba

Kjučne besede: mikrofluidika, jedkanje stekla, vezava, dielektroforetični filter

Izvleček: V prispevku predstavljamo tehnologijo jedkanja in montaže steklenih rezin za uporabo v mikrofluidiki, kakor tri primere uporabe tako razvite tehnologije. Preizkusili smo posebne maskirne plasti (Cr/Au/fotorezist in a:Si/SiC/fotorezist) za globoko mokro jedkanje stekla. Uspeli smo pojedkati skozi 1mm debelo rezino iz pyrex stekla z masko a:Si/SiC/fotorezist. Ugotovili smo, da je raztopina HF(49%)/HCl(37%) v razmerju 10/1 optimalna za doseganje dobre površinske hrapavosti. Eksperimentalni rezultati dobljeni s suhim jedkanjem pyrex stekla v ICP reaktorju kažejo, da lahko dosežemo navpične stene z uporabo mešanice plinov C_4F_8/He. Najboljše rezultate smo dosegli z masko iz silicija.

Mikrofluidika zahteva vezavo silicijevih rezin prekritih z metalizacijo. Vezava z lepljenjem je lahko rešitev za direktno montažo steklo na steklo (sploh pri nizkih tempareturah pod 200 ^{O}C). Razvili smo posebno tehniko vezave z lepljenjem s SU-8 negativnim fotorezistom, ki smo ga natiskali na podlago.

V prispevku predstavimo načrtovanje, izdelavo in eksperimentalne rezultate mikrofluidnih komponent: DEP čip z elektrodami iz silicija, 3D DEP filter, kakor tudi mikrofluidni čip za celično impedančno spektroskopijo.

1. Introduction

Microfluidic devices and systems have become essential elements for biomedical instrumentation and it is considered one of the most promising MEMS application area. Microfluidic dispensing and controlling devices, such as micro pressure/flow sensors /1/, monolithic membrane valve/diaphragm pumps /2/, have been developed with integrated glass components. DNA related microfluidic devices, such as micro flow cells for single molecule handling of DNA /3/, micro injectors for DNA mass spectrometry /4/, and µPCR devices for DNA amplification /5/, have also been presented. Being transparent under a wide wavelength range, glass is a prime candidate for microbioanalytical devices such as microcapillary electrophoresis /6/ or dielectrophoretic devices for cell trapping /7/ or sorting /8/.

The paper presents an extensive investigation of glass microfabrication with focus on wet and dry etching of glass, proposing solution for improving the performances of wet and dry etching process. A second part is dedicated to new techniques for adhesive bonding of processed glass wafer: adhesive bonding using contact imprinting. Finally, three applications are presented: a dielectrophoretic device for cell trapping and sorting, a dielectrophoretic filter and a microfluidic chip for impedance spectroscopy.

2. Micropaterning of glass

Glass is a very suitable material for microfluidics due to its characteristics: good mechanical and optical properties, high electrical insulation and high chemical resistance to many chemicals. The main requirements for the glass used in microsystem technology are: microstructurable using standard lithography process, suitable for metal deposi-

tion, transparent for wide range of wavelength, apt for bonding to silicon.

2.1 Paterning of glass

There are three major groups of technique used for glass etching: mechanical, dry and wet. Mechanical methods include traditional drilling, ultrasonic drilling, electrochemical discharge or powder blasting. However, smooth surfaces cannot be generated using such methods. Dry etching of glass had been reported in /9/ using SF_6. However the etching rate is relatively low. Wet etching remains the most common and low cost method. The etching solution is based on HF. The masking layer depends on the application and on the "thermal budget" of the fabrication process of the device. Photoresist is very often used as mask layer /10-12/, but its area of application is limited. A very commonly used mask is Cr/Au /12, 13/, where Cr layer is used to improve the adhesion of gold to glass. Bu et al /14/ reported etching through 500 µm-thick glass wafer using a multilayer of metal, Cr/Au/Cr/Au, in combination with a thick SPR220-7 photoresist, by etching from both sides of the wafer. Another very commonly used mask material for glass etching is silicon, deposited by different methods: PECVD (amorphous silicon) /12, 15/, LPCVD (polysilicon) /12, 16/ or even bulk silicon /17/. The maximum reported depth was 320µm by Bien et al /12/ with a mask of polished polysilicon (1.5µm) and SU-8 (50µm) as etching mask.

2.2 Wet etching of pyrex glass

2.2.1 Etch rate

In the wet etching of glass, the main material used as masking layer (Si and Au) are inert in the HF-based etchant. As a result the etch rate become an important parameter: a fast etch rate will lead to a deeper etching, while the defect generation will be maintained at the same rate each time. The main solution used for glass etching is based on HF. The etch rate is characteristic for each type of glass, especially due to the different oxides and compositions used during fabrication. Meanwhile, the etch rate is determined by the concentration of HF enchants. To achieve a high etch rate, a maxim concentration of 49% should be used. It should be noted that, for Corning7740, by increasing the HF concentration from 40% to 49%, a rapid increase of 50-60% of the etch rate can be achieved (4.4µm/min to 7.6µm/min). The annealing process has a strong influence on the etch rate of glass. For annealed Pyrex glass the etch rate was from 9.1µm/min (for HF40%) and increase to 14.3µm/min when the HF concentration increases to 49%. Warming the solution to 40-50^{O}C can also increase the etching rate but the method is not recommended for safety reason (an increase quantity of HF vapours). Using ultrasonic for agitation the masking layer can be damage.

2.2.2 Masking layers

The main problems of wet etching are the pinholes and notching defects on edges. These could be observed after certain etch time, and were the result of the interaction between the etchant and mask. These defects presented in Figure 1 limit the etch depth of glass. The main reasons of defect generation are: the residual stress in the masking layer /15/, the stress type (tensile or compressive), the gradient of stress and the hydrophilicity of the surface. We will present in this chapter an analysis of the main masking layer that can be used for the wet etching of glass. The glass etchant was HF 49%.

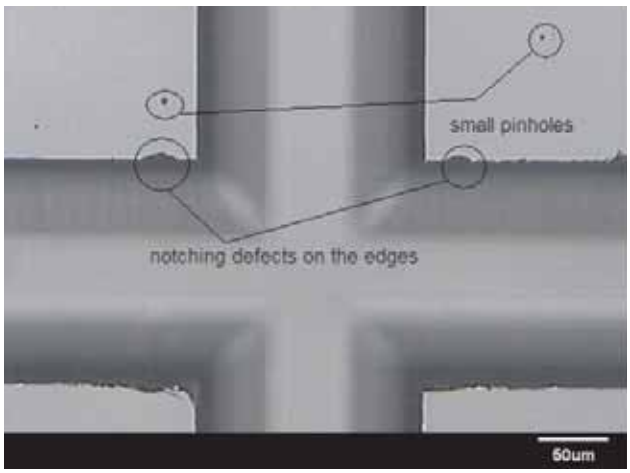

Fig. 1: Optical image of 100µm-deep etched in glass with Cr/Au mask

Photoresist masking layer. For our experiment, we used positive photoresist AZ7220 (from Clariant). In highly concentrated HF solutions, the quality of the photoresist mask was very poor. The maximum etching time – appreciated to be around 3 minutes (equivalent with a depth etch of 22 µm) – was achieved after the photoresist was hard-backed at 120^{O}C for 30 minutes on a hot plate. A huge isotropy was noted (5:1). After etching for a long time, the photoresist mask would peel off. The technique could be used in cases where up to 20 µm deep etching is required such as capillary electrophoresis.

Amorphous silicon (a:Si). Silicon is an inert material in HF-based solutions. It also has the advantage of being a hydrophobic material. Hence, the penetration of etchant through the small impurities of the mask is relatively difficult. The a:Si masking layer presents the advantage of deposition at low temperatures (almost room temperature for sputtering and 300^{O}C for PECVD deposition), but as we analyzed in /15/, the high value of compressive stress induced in this layer (600MPa) limited its application to 20min. The annealing of the masking layer could reduce the value of the stress and improve the performance to up to 30 minutes – equivalent of 200µm /15/. The isotropy of the etching was 1:1.2. The influence of stress is presented in Figure 2, where wet etching of glass was performed using the same a:Si mask, but this was annealed

at 400^{O}C at different times, resulting in a different residual stress in the masking layer.

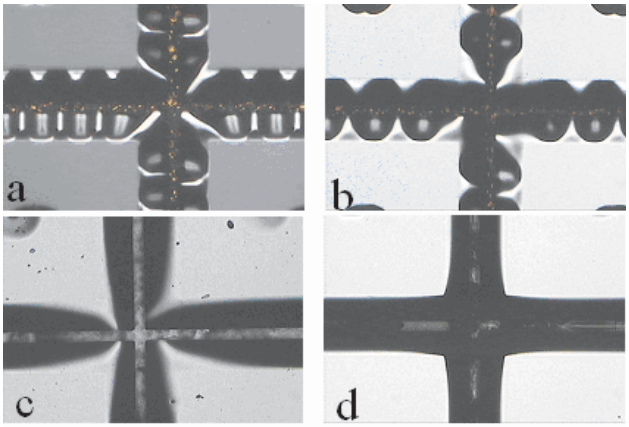

Fig. 2: Optical image glass etching using a:Si mask with different stress values: a) 600MPa b) 300MPa c)100MPa d) 100MPa (tensile)

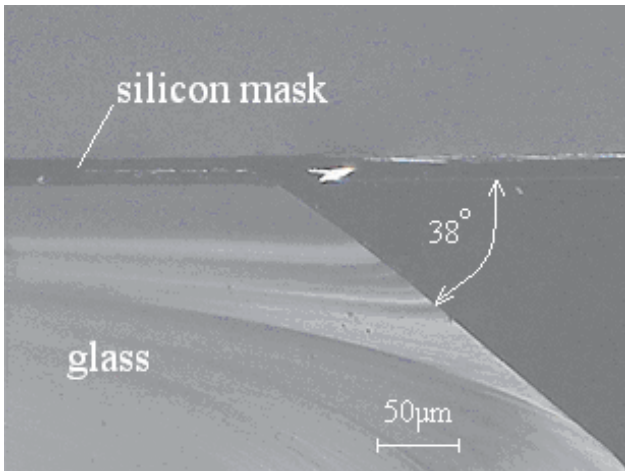

Fig. 3: Etching results with bulk silicon mask.

Polysilicon. The test layer was deposited at 530^{O}C in a furnace. The resulting stress in the layer was 50MPa compressive. The resistance of the mask in HF solution was 30min. (similar to results using a:Si). The isotropy of the etching was very good – 1:1.

Bulk silicon layer. Single crystal silicon can also be used as a mask for glass etching. Wet etching with bulk silicon mask was first reported by Corman *et al* in /17/. In tests, a silicon wafer was anodicaly bonded on a glass wafer on an EVG 601 bonding system. The silicon wafer was thinned up to 30µm in an Adixen ICP-Deep RIE system. After patterning with photoresist, the mask was defined in the silicon layer using a classical Bosch process. The etching result indicated that the mask was perfectly inert in HF solution, but a huge isotropy of the etching process was observed. Figure 3 presents the results of wet etching in HF 49% solution of a Pyrex glass using a bulk silicon mask. The resulting angle between the mask and glass surface (38^{O}) indicated that the interface layer was removed quickly. The main reason for this could be the poor quality of the silicon oxide interface layer (between the silicon mask and glass wafer), which presented an increased etching rate. Similar results had been reported in /17/.

Cr/Cu. The first reported result of etching with the Cr/Cu (50 nm/1µm) masking layer is presented in /15/. The maximal time for a good quality etching process was around 15 minutes (about 100µm-deep etch). The low value of residual stress of the Cr/Cu layer (50-80MPa tensile) and the good selectivity of Cu in the HF etchant can make this layer very suitable for microfluidics applications, where the required depth is below 100µm. If the photoresist mask (AZ7220 from Clariant) used for Cr/Cu layer patterning is hard baked and kept for the glass wet etching process, the etching results can be improved sensitively.

Cr/Au. One of the most commonly used metal masks is Cr/Au. The best results initially obtained with the Cr/Au masking layer were in the range of 50-100µm depth /15/ (7-15 minutes), as a function of the layer thickness. The mechanism of defect generation is very simple: due to the tensile stress in the deposited layer (250-300MPa) the masking layer creped and a large number of defects were generated in the mask. The Au mask surface is hydrophilic. Therefore, once the etchant solution was in contact with the mask, it would penetrate easily, through the mask defects and generate pinholes. To minimize the effect of these cracks in the thick Au layer, a series of deposition/ cooling actions can be applied. After depositing 200-250nm of Au, the deposition was stopped for 10min. The temperature of the wafer would change and possible cracks were generated. The deposition process would then continue. The possibility of generating defects in the same position was reduced when the next layer of Au was deposited. This method of deposition generates a 1.2µm-thick Au layer, and the etching time can be increased to 50 minutes. If the photoresist mask used for Cr/Au mask patterning is hard baked, the performance of the masking layer can also be improved. The photoresist will penetrate and fill the cracks generated by the tensile stress in the Cr/Au layer. Furthermore, the hard baked photoresist surface will make the mask surface hydrophobic. Figure 4 presents a hole with a diameter of 700 µm that was etched through a 500µm Pyrex glass wafer Corning 7740 (annealed). The etching was performed in a Teflon beaker in the same HF solution, with magnetic stirring for 85 minutes. No defect was observed after the removal of the Cr/Au mask.

Low stress a:Si/SiC/photoresist. The target was to generate a mask with low residual stress, no stress gradient along the thickness and a hydrophobic surface. Finally, a multilayer mask consist of low stress a:Si/ SiC/ photoresist was found very suitable for glass etching. Both a:Si and SiC layer were optimized for a low stress value /18/. The hydrophobic surface was generated by keeping and hard baking the photoresist mask. The results of the etching process using a:Si/SiC/photoresist masking layer in

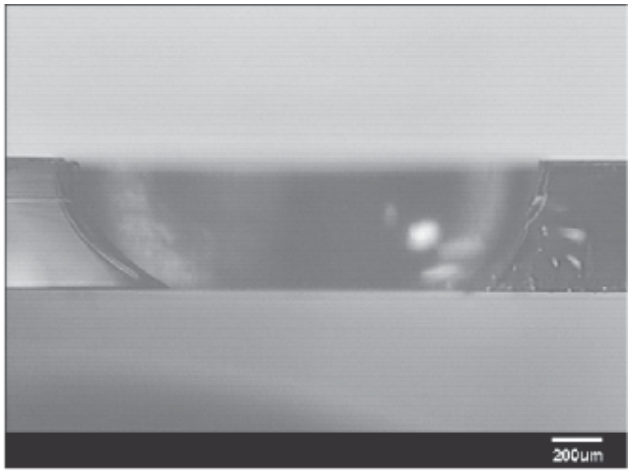

Fig. 4: Cross-section view of the through-etched hole with a Cr/Au/ photoresist mask for an annealed glass.

HF49% are presented in Figures 5, 6. The protection of the backside of the wafers was assured by a wax bonding on a dummy silicon wafer.

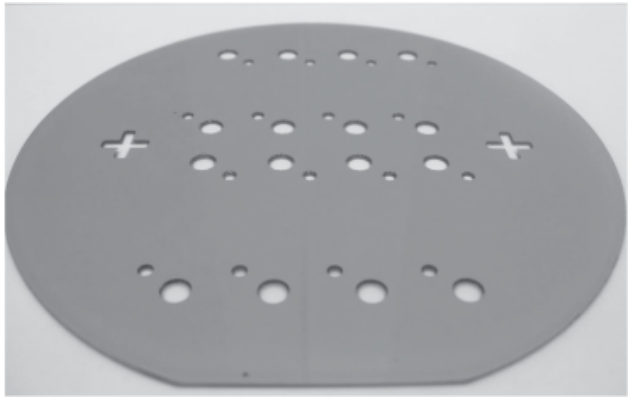

Fig. 5: 500µm-thick Pyrex glass wafer etch-through using a:Si/SiC/ PR mask (the mask was kept on the wafer)

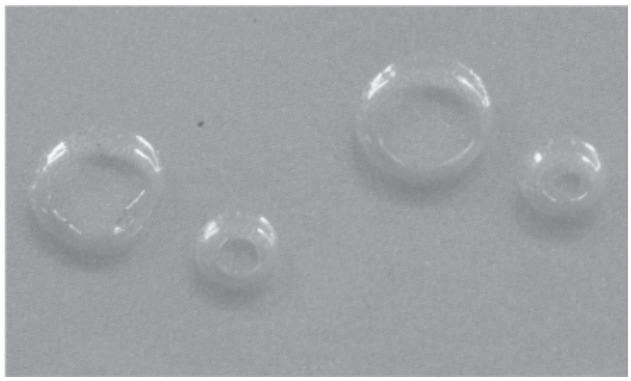

Fig. 6: 1 mm-thick Pyrex glass wafer etch-through in HF49% after removal of using a:Si/ SiC/PR mask

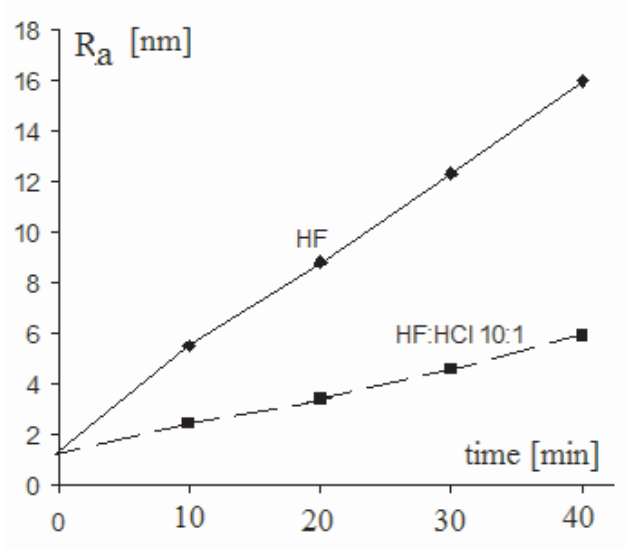

Fig. 7: Improving of surface roughness using HF/HCl solution

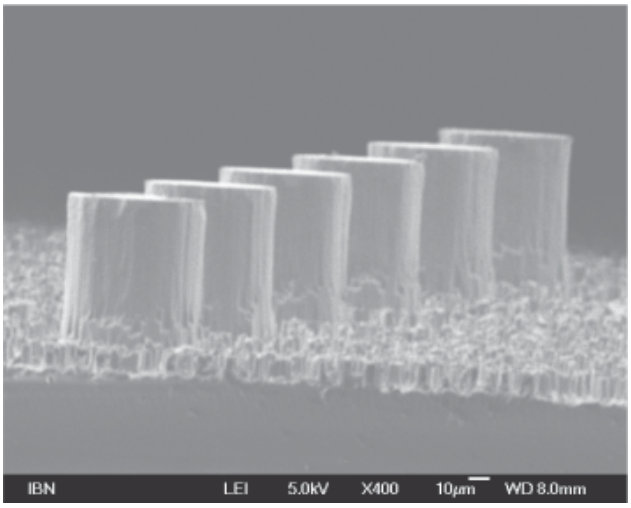

Fig. 8: Glass pillars etch in Deep RIE system using C_4F_8.

2.2.3 The roughness of generated surface

The roughness of generated surface can be an important issue for the wet polishing and deep wet etching of channels. Figure 7 shows the variation of roughness for Corning 7740 versus time for HF solution and when HCl was added (HF/HCl 10/1) solutions. The graph is almost linear for both solutions but with small values for HF/HCl solution. The purpose of HCl was to remove the insoluble products.

2.3 Deep dry etching of pyrex glass

Previous work reported the etching of Pyrex glass using SF_6 and electroplated Ni as the mask /9/. In our experiment we used a Fluorine gas C_4F_8 as the gas etchant. The main advantage of using this gas is the presence of the generated plasma carbon-based radicals that can passivated the trench's walls and result in a profile with vertical

walls. The experiments were performed on an Adixen Deep RIE ICP (oxide etcher). Other critical parameters were the pressure (we performed our experiments at 0.5Pa) and coil power (we use the maximal RF power 2800W). We tested our experiment Al (6µm) and bulk Si (40µm) masks. For the Al mask, a selectivity of 1/10 was achieved, but after long processing (1 hour) we observed that the protected surface (after the mask removal) became rough and mate due to the ion bombardment. For this reason, we looked for a material that could be easily deposited and patterned in the thick layer: bulk silicon. Even though the selectivity was relatively similar (1:15), this masking layer presented some important advantages: its ability to work with a thick layer, good patterning (vertical walls) using classical Bosch process in Deep RIE systems and easy removal in KOH solutions. The results of the etching of 80µm-tall glass pillars using a 40µm bulk Si mask is presented in Figure 8.

3. Assambling at wafer level using adhesive

Assembling technology at wafer level is another key feature in glass microfluidics fabrication. One solution can be wafer-to-wafer anodic bonding of glass to silicon, a well establishes technology that can assure a hermetic sealing. For special applications, glass/silicon/glass a double wafer-to-wafer bonding can be performed /19/.

Adhesive bonding can be another solution for microfluidic devices. It enables joining of silicon or glass wafers at lower temperatures. The technique is less dependent of the substrate material, particles, surface roughness and planarity of the bonding surfaces. Several lithographic patternable materials such as BCB or positive and negative photoresists have already been studied as intermediate layers for adhesive wafer bonding. Beside BCB the epoxy based negative photoresist SU-8 provided very promising results in bonding experiments /20, 21/. The advantages of SU-8 are its flexibility in choosing the layer thickness, its high chemical and thermal stability as well as its good mechanical properties. However, for devices with nonplanar or micromachined surfaces the adhesive layer cannot be applied directly using classical spin-coating methods as these would result in undesirable major non-uniformities of the deposited layer which can affect the functionality of the device. Therefore, in such cases the only solution available for an adhesive bonding is stamping the layer on one of the surfaces, followed by the alignment and bonding process.

A new imprinting technique was developed specially for devices where the bonding area is quite large. In such cases, if the imprinting is performed directly from a dummy wafer, the strong adhesion between the dummy wafer and the bondable surface would make very difficult the detachment of the dummy from the device wafer. The proposed solution to this major problem is to use a Teflon cylinder for first transferring indirectly the SU8 adhesive from the dummy wafer and then imprinting the adhesive layer further on the bonding surface. The process is illustrated in Figure 9. First a thin layer of SU8-5 is applied on a dummy silicon wafer. The SU-8 photoresist was spun on it at 3000 rpm/60 seconds, resulting in a SU-8 layer 12µm-thick (Figure 9a). The next step is the transfer of the adhesive layer onto the surface of a Teflon cylinder with a diameter of 38mm and a length of 120mm (Figure 9b). The process is also illustrated in Figure 10. Further, the cylinder is rolled on the bonding surface and the adhesive layer is partially transferred on this surface (Figure 9c). In the next step, both structured wafers were aligned and brought in contact (Figure 9d). The last step is the wafer-to-wafer bonding, which was performed at different temperatures between 20 and 100°C for 30min at an applied force of 1000N in vacuum (Figure 9e).

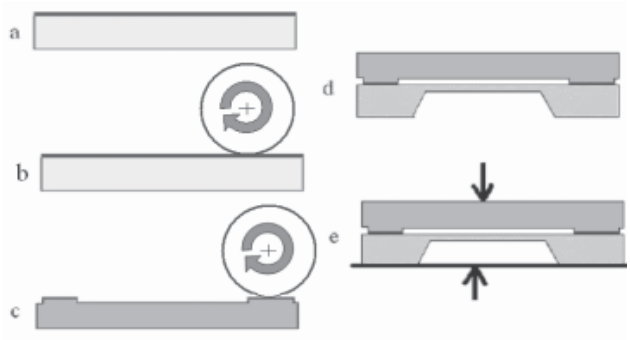

Fig. 9: a) Spinning of SU8-5 photoresist on a dummy wafer, b) Contact imprinting of SU8 on a Teflon cylinder, c) Imprinting of SU8 from the Teflon cylinder on the wafer surface, d) Alignment and contact, e) Wafer bonding.

The measured average residual stress indicated an overall value in the range of 20 to 40MPa (tensile). These low values as well as the good elastic properties and high chemical and thermal stability of the SU-8 show that it is a most suitable material for wafer-to-wafer adhesive bonding. Figure 11 presents the optical image of cross-section through the bonding region, clearly showing both fully bonded and partially bonded areas. The yield of bonding process was high (95- 100%).

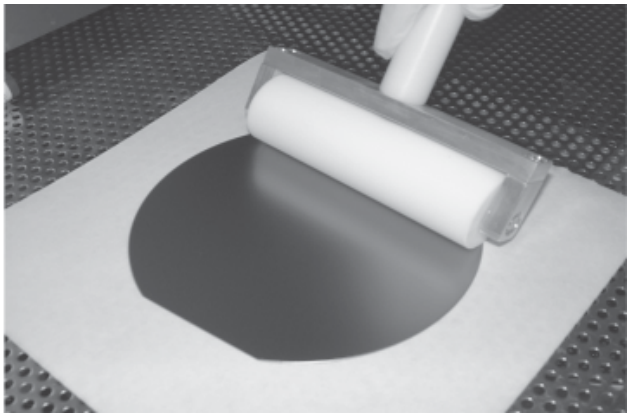

Fig. 10: Imprinting of the SU8 from a dummy wafer to the Teflon cylinder.

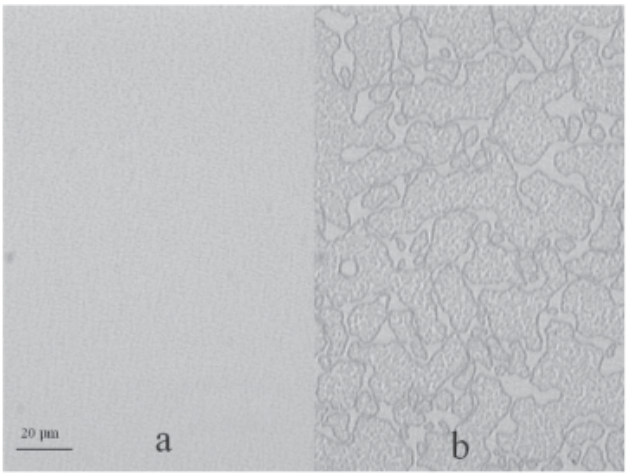

Fig. 11: a) Fully bonded area and b) partially bonded area.

Figure 12 presents the results of the wafer-to-wafer bonding process while in Figure 13 the cross section through a microfluidic channel is presented (at the top corners residual SU-8 can be observed).

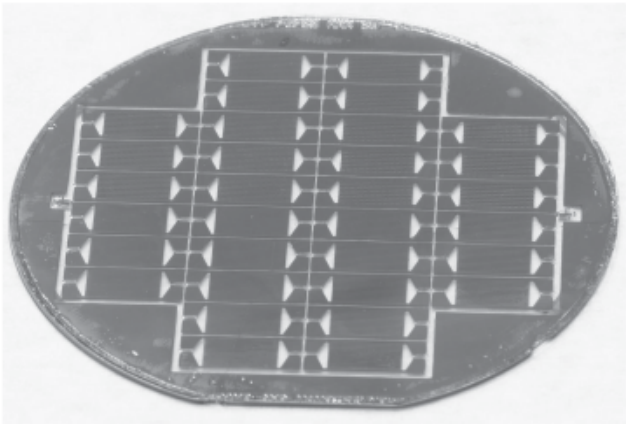

Fig. 12: Bonded wafers using contact imprinting and SU-8 photoresist as adhesive.

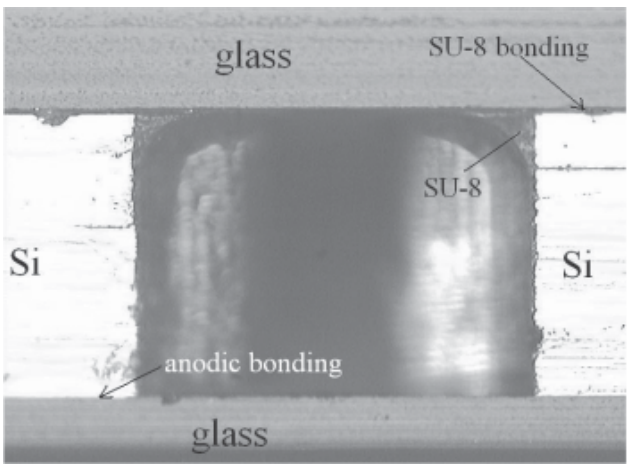

Fig. 13: Microfluidic channel with bulk silicon walls and glass as ceiling and floor.

4. Applications

4.1 Dielectrophoretic device packaged at wafer level

An application of the above describe techniques was the fabrication of a microfluidic device for dielectrophoresis (DEP). The device consists of three functional layers, where two of them are insulators made of glass and the third is a conductive silicon die in which electrodes and the microfluidic channel are patterned. The assembly was performed at the wafer level by anodic bonding. A metallization, performed on the bottom glass layer, provides the electrical connections of the bulk silicon electrodes through via holes etched in the glass with PCB. A detail view of the fabricated device (top with the inlet/outlet tubes and bottom with metallized via holes) is presented in Figure 14. The main steps of the fabrication process are presented in Figure 15.

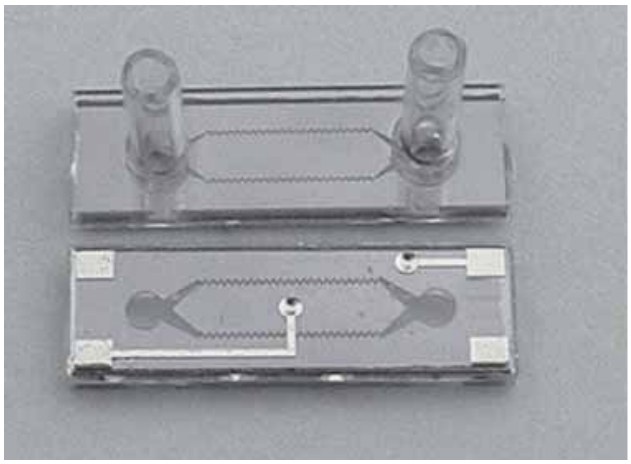

Fig. 14: Photo with the microfluidic DEP device

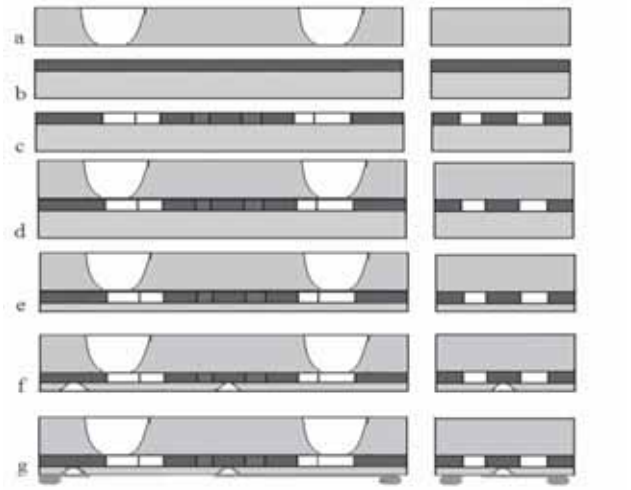

Fig. 15: Main steps of the fabrication process

The fabrication process start with the fabrication of inlet/outlet holes using a low stress a:Si/photoresist mask- Figure 15a. An anodic bonding process is performed between

an 100µm-thick silicon wafer (heavy doped) and a 500µm-thick glass wafer (Figure 15b). In the next step the geometry of the microfluidic channel and electrodes are defined in silicon, using a classical Bosch process on an ICP deep RIE system (Figure 15c). A second adhesive bonding (Figure 15d)-previous described is performed between the wafer with inlet/outlet holes and the wafer with the electrodes. Via-holes must be generated in the bottom glass in order to assure the electrical contact of the silicon electrodes with the PCB. These via-holes are usually defined using a wet etching process. Even the dimension of the mask for via holes is relatively small (diameter of 50µm), due to the isotropy of the process, in a 500µm-thick wafer, the final dimension can reach more then 1 mm. For this reason a thinning process of the bottom glass wafer is required. The bottom glass wafer was thinned from 500µm up to 100µm in the HF/HCl solution -Figure 15e. The mask for via holes was performed using Cr/Au and photoresist. The etching of via-holes (Figure 15f) was performed in the same HF/HCl solution. After the deposition of the metallization layer (Cr/Au) the photoresist masking layer was applied using a spray-coating process (Figure 15g). The device was successfully tested using yeast cells and the results are presented in Figure 16.

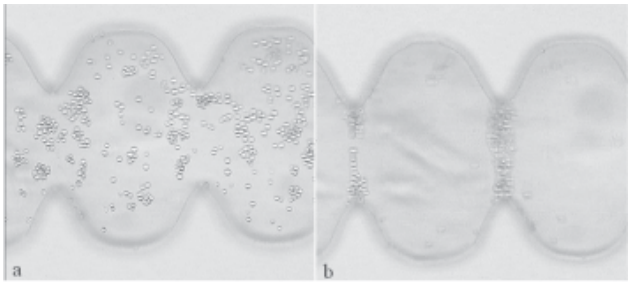

Fig. 16: Trapping of the yeast cells in a DEP device

4.2 Dielectrophoretic filter

The fabrication process of a DEP filter for bacteria trapping will be presented in this section. An 1mm-thick glass wafer was etch-through simultaneous from the both sides using the previous described Cr/Au and photoresist mask. The target was to generate a thick glass frame with metallization on the both sides. After removing the photoresist in a classical resist stripper, and dicing of the wafer, two stainless steel meshes was soldered on the both sides of the frame. Before the second soldering process, the frame was field with silica beads (100µm-diameter). The device acts as an electromechanical filter for cell trapping. An image with the glass frames as well as the funnels used for filter testing is presented in Figure 17.

4.3 Microfluidic device for impedance spectroscopy

In Figure 18 a microfluidic device for electrical impedance spectroscopy analysis of biological sample is presented. The device consist of two glass dies: the top one with in-

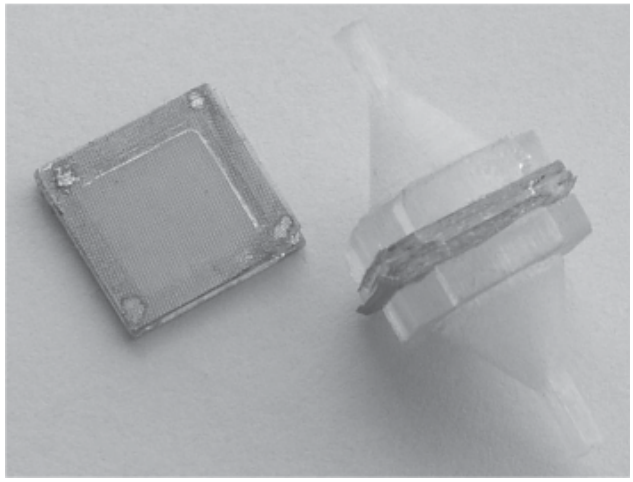

Fig. 17: Image with DEP filter-chip device

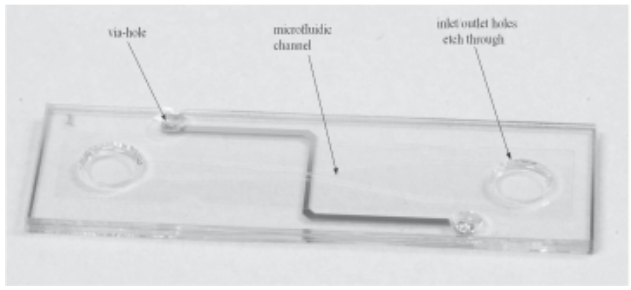

Fig. 18: Image a microfluidic device for electrical impedance spectroscopy

let/outlet holes performed with the technique described before and the bottom die with a 25µm-deep microfluidic channel and metal electrodes. The dies were bonded at wafer level using the adhesive bonding technique previous described.

5. Conclusions

The paper presents two main technologies for fabrication of glass microfluidic devices: wet and dry patterning of glass as well as a new technique for adhesive wafer-to-wafer bonding. Three applications of the described techniques are also presented.

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