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TECHNICAL PAPER

Fabrication of microfluidic device channel using a photopolymer for colloidal particle separation

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Abstract We have developed a method of fabricating microfluidic device channels for bio-nanoelectronics system by using high performance epoxy based dry photopolymer films or dry film resists (DFRs). The DFR used was with a trademark name Ordyl SY355 from Elga Europe. The developing and exposing processes as well as the time taken in making the channels are recorded. Finally from those recorded methods, the accurate procedures and time taken for DFR development and exposure have been found and ultimately been consistently used in fabricating our channels. These channels were patterned and sandwiched in between two glass substrates. In our advance, the channel was formed for the colloidal particle separation system. They can be used for handling continuous fluid flow and particle repositioning maneuver using dielectrophoresis that have showed successful results in the separation.

Keywords Photopolymer · Dry film resists (DFR) · Microfluidic · Dielectrophoresis · Separation and colloidal particles

1 Introduction

Vast interest in Lab-on-chip systems (LoC), which is also known as micro- total chemical analysis system μTAS has grown tremendously in the past decade in the research and industry areas (Manz et al. 1990). The technology allows designers to create small, portable, robust, low-cost, and easy-to-use diagnostic instruments that offer high levels of capability and versatility (Lao et al. 2002; Reyes et al. 2002). LoC systems will decrease reagent consumption and reduce cost per analysis. It also reduces analysis time and provides better controllable process parameters in chemical reactions.

With the recent advances in the synthesis and the characterisation of size-selected particles in the colloidal (submicron and nanometre range), an investigation on their physical and chemical properties has been made possible (Shirinyan and Wautelet 2004). In LoC, a fabrication of microfluidic devices with integrated channels and electrodes of dimensions are made comparable to biological cells or particles size. Additionally, if there is a capability of producing small scale devices, it will allow the development of entirely novel experiments, which currently is still under research. Since then, chip based analytical systems have been developed and applied to a variety of fields such as separation science, chemical production, DNA analysis, medical diagnostics and environmental analysis.

Technology for the separation and manipulation of particles is an essential requirement in chemistry, biology and biochemistry. The separation technique used in this work is dielectrophoresis (DEP). DEP is defined as a force exerted on charge or un-charge particles placed in non-uniform electric fields. When particles are placed in a non-uniform electric field, the particles and fluid will polarise. If there is a difference in polarisability, there will be a net force. The particles will move towards the high field or low field depending on their polarisability. DEP is a non-contact, non-invasive, non-destructive method and does not require labelling for separation of particles in a suspension.

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A microfluidic device has been designed to accomplish DEP separation (Jones 1995; Morgan and Green 2003).

To make LoC comes true; the processes for microchannel fabrication using photopolymer are studied. In order to form a microchannel, some photopolymers such as epoxy has been studied. This polymer is adhesive (Niklaus et al. 2006). The choice of the polymer material depends very much on the thermal, chemical and mechanical requirements of the application. In this work, study on epoxy laminate will be shown. It is a negative photopolymer or sometimes it is called dry film resist (DFR) (Vulto et al. 2005). The commercial name is Ordyl SY355 (50 μm thick, Elga Europe, Italy). Ordyl SY355 is a permanent typed dry film, which is usually used for special MEMS applications. It is in the form of a laminate sheet that can easily be applied on a hot roll laminator. Ordyl SY355 coatings can be processed with proprietary Chlorofluorocarbon (CFC) free chemicals (BMR Developer and BMR Rinse). It exhibits excellent mechanical, heat and chemical resistance. It also has long term stability. Particularly, it shows compatibility with biological fluids. It has strong adhesion to different materials (glass, silicon, epoxy resin, polymers, etc.). It can be used to realise sandwiches with a top plate on the processed film.

Ordyl SY355 resist thickness is 50 μ (Taberham et al. 2008). The storage should be in UV free condition with temperature below 20°C and relative humidity (RH) below 50%. It consists of a resist layer protected on one side by polyester (PET) layer and on the other side by a polyethylene (PE) layer.

2 Materials and methods

2.1 Epoxy laminate

There are a lot of methods and types of materials such as glass, silicon and polymer that can be used to build a channel for microfluidic devices (Stemme 1995; Svasek et al. 1996; Terry et al. 1979). However, here in this section, the advantages of using dry film resist in the fabrication over the other material are emphasized (Renaud et al. 1998; Vulto et al. 2005).

The used of dry film resists (DFR) were originally developed for printed circuit board (PCB) fabrication (Dempsey et al. 1997). It is also available as etching resists and solder mask (Svasek et al. 1996). Although application for MEMS fabrication is still new, DFRs have been reported to be useful for fabricating electroplating moulds, sealing of fluidic channels and as a mask for powder-blasting of microchannels. DFRs offer many advantages over liquid resists, such as a good conformability, excellent adhesion to any substrate, good flatness, no necessity of liquid handling, no formation of edge beads, uniform resist distribution, low exposure energy, low cost, short processing time and near vertical sidewalls. This DFR is a cheap and fast alternative to SU-8, which has been discussed in detail by Vulto et al. (2005).

In addition to that, it can be marked that the setting up cost for DFR processing is significantly lower than for liquid type of resists. Hence, as in this work, Ordyl SY355 dry film resist (Elga Europe, Italy) is believed to be extremely suitable for creating fluidic channel networks. Bonding the second substrate to first substrate under low pressure and minimal heating, without the need for extra adhesives, can perform close fluidic channels. Subsequently, the resist can be transferred into a rigid structure by a post-bake. Biocompatibility of the resist makes it extremely suitable for application in complex biochips (Vulto et al. 2006).

High aspect ratio is the ratio of height to width of the channel in the chip that is fabricated. In this experiment, a device with low aspect ratio will be performed. This is because the strength of the DEP force horizontally can be increased when the height of the channel is reduced. Therefore, the low aspect ratio of the device permits a high-efficiency separation because the range of the DEP force is extended throughout the cross section of the fluid channel (James et al. 2006).

2.2 Substrate material

Glass is preferred to be used as a substrate material and it has been used for over a decade. The advantages of glass in relation to the other materials are; it is chemically inert to most liquid and gases, hydrophilic and optically 100% clear. In this work, a borosilicate glass (Pyrex), which is the type of the microscopic glass slide, is used as the substrate. It can resist strong acids, saline solutions, chlorine, bromine, iodine, and strong oxidising and corrosive chemicals. Therefore, it has been proven to be an ideal substrate for chemical and biological analysis (Simpson et al. 1998; Woolley and Mathies 1994). The other reason for glass to be used as a substrate is because glass is hydrophilic, which attracts and holds moisture. Most plastics, in comparison, are hydrophobic and need treatment to become hydrophilic. If hydrophobic surfaces are needed, a coating to the glass can be applied. Furthermore, there is no interference with any reaction or analysis by any residue, which is known to dissolve out of plastics in some cases. Glass has material properties that are stable in time and it is thus preferable for applications in which devices are used extensively, such as in high throughput screening. It is a reliable shelf life. It is non-porous, implying that small molecules will not be able to diffuse into the material and sometimes even worse, diffuse back into the solution at a later stage, thus can contaminate the experiments.

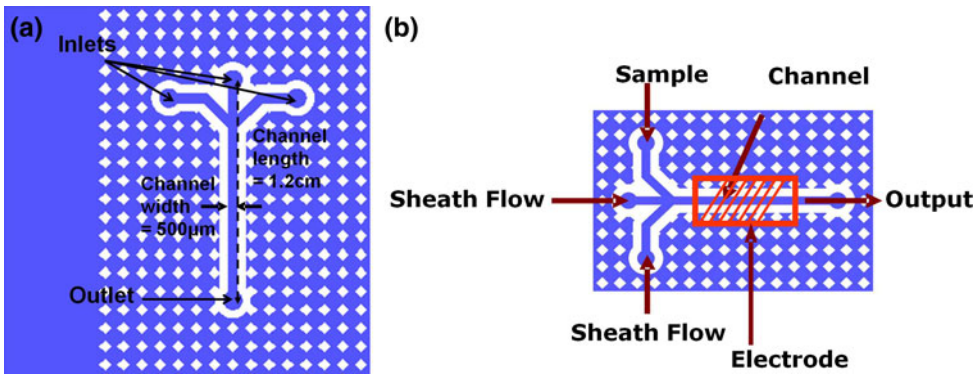

Fig. 1 **a** Channel masks drawn using L-Edit with three inlets and one outlet. The channel width is 500 μm and channel length is 1.2 cm. **b** Illustration of the operational of the angled electrodes array in the channel with the inlets and outlet usage

2.3 Channel design for separation

The channel design begins by drawing a mask layout for the channel. It is drawn using L-Edit Tanner Research. It is the CAD tool that is common for the electrical and electronics engineers in drawing masks. The channel mask drawn will be suitable for the negative photoresist. The channel is designed with several inlets and outlets. These inlets/outlets are for the fluid to move into the channel and out from the channel.

For the channel design shown in Fig. 1a, the channel width is 500 μm wide and the channel length is 12 mm. The arrows show the possible inlets and outlet but it depends on the usage and it can also be vice versa. The diamond shapes are the supporters for bonding process. There are gaps between them to avoid the gas/air trapping while the bonding process takes place. The electrode used on this channel is the set of electrodes that are inclined with certain degree. The illustration of the electrodes on the channel is shown in Fig. 1b.

The electrodes used in the design will be fabricated with gold. Electrodes on the glass produce photo lithographically over titanium seed layer. The titanium layer serves as the adhesion layer between glass wafer and Au electrodes. The Au is conductive and biocompatible. Biocompatible refers to the ability of a material to perform with an appropriate host response in a specific application or the quality of not having toxic or injurious effects on biological systems. The diagram of the microelectrode arrays on the wafer is shown in Fig. 2.

2.4 Epoxy laminate process

As mentioned earlier, the substrate used for the device is a microscope glass and is a 1.0 mm thick borosilicate glass (Menzel-Glaser, Braunschweig, Germany). It is cut to 20 mm × 26 mm and then is placed in the glass slides holder, for cleaning purpose. The holder is filled with 95% pure water and 5% detergent. Then the glasses are ultrasonicated with DI water for 15 min. After sonicated, the

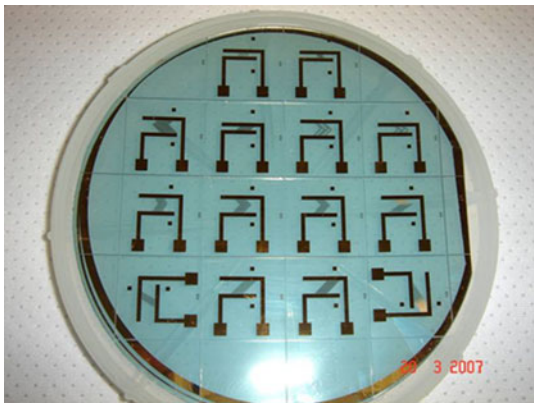

Fig. 2 Microelectrode arrays on the wafer ready to be used

glasses are thoroughly rinsed with DI water followed by acetone, methanol and iso-propanol (IPA) and blow-dried with nitrogen.

After the cleaning process is completed, the glasses are placed in the oven for dehydration at the temperature of 200°C. This step is done to make sure that the glasses used are dried totally so that the attachment of laminate on these glasses will be excellent. The cleaned glasses are left overnight in the oven.

The following day, the process is started by peeling off the protective film, the PE layer, from new sheet of laminate. The resist is applied on the substrate placed on the cardboard. The substrates are put through the hot-roll laminator with speed 1 m/min and at temperature 105–110°C. The protective PET should remain on top of the laminate when the exposure process is performed. The processes such as exposure, developing and rinsing are done accordingly.

2.5 Bonding

There are two appliances that can be used for bonding process in the laboratory. The first one is called the hot press bonder machine and the other is using the oven. The steps taken for the two bonders are almost the same. For the hot

press bonder machine, the temperature used must be ramped up, from 150°C to 200°C. These temperatures are set at a certain time length, respectively. The first temperature of 150°C is set up for 30 min while the 200°C is for 1 h. After this process, the temperature is ramped down slowly to the room temperature. This process takes about 6 h to finish. For the oven, the process of bonding the two laminates on the glass would take longer time. The process is started by setting the temperature up to 150°C for 30 min, and then is ramped down to the room temperature. The next step is to set up back the temperature to 200°C for 1 h and finally it is ramped down again to the room temperature. This process will take one full day to finish.

Subsequently, fluids are flowed through the channels to check for leakage and clogging (Han et al. 2003). Sometimes the channel are tested using fluorescent dyes to confirm a good seal (Satyanarayana et al. 2005). UV glue will be used if the channel has leakage after hot press bonding or oven bonding. UV glue is used to cover the area that is leaked, by permeating it at the side of the glass. The glue will flow by capillary force in between the substrate throughout the diamonds pillars (the supporting shapes for bonding). Afterwards it will cover the whole area around the channel wall. The device is then exposed under UV light for 40 s, and hence the channel will be formed successfully without any leakage.

3 Results

3.1 Channel making

Figure 3a below shows the mask of the channel with the pattern of three inlets and one outlet. The DFR epoxy laminate is exposed to UV light (400 nm) for 9 s through a printed lithographic acetate mask. Illuminated areas would be removed from the glass during development process. The exposure time and the developing time are recorded. Next, it is shown in the graphs that are plotted for the exposure time and developing time taken versus the channel width and laminate thickness.

3.2 Exposure times

The process of defining these patterns on the substrate is known as lithography. Lithography uses photoresist materials to cover areas on the wafer that will not be subjected to material deposition or removal. There are two types of photoresist material, namely, negative and positive photoresist. Since epoxy laminate is the negative resist, it will become less soluble in the developer solution when it is exposed to light, forming negative images of the mask patterns on the substrate. After laminating, the exposure process (photolithography) is taken place. The exposure dose required depends on the thickness of the laminate used. The exposure is performed by placing the channel mask on top of laminated substrate under the light box. This can be illustrated as shown in Fig. 3b. The mask is placed on top of the resist and then it is exposed under the UV light. Since the negative resist is used, those exposed to light will become less soluble in the developer solution. The exposure time period is recorded. This is to find out the accurate time for exposing 50 μm thickness in order to get the accurate channel width after developing and rinsing. An average of channel width versus exposure time is plotted as shown in Fig. 4. It is shown in the graph that the exact time that can be used for exposing 50 μm laminate is 9–10 s.

3.3 Development time

Developing process comes after the exposing process. The process is done by using the chemical named BMR Developer. Developing time has, also, been recorded to ensure the exact developing time that should be performed for having the correct channel width. These data are taken when the exposure time is 9 s. The developed time is taken when the sample is placed in the sonicator bath. The sample is then developed outside the sonicator bath and rinse for 60 s. The average of it is plotted in Fig. 5. In the graph, the region of under develop (UD) and region of over develop (OD) are indicated. In UD region, the widths of the channel are narrower and starting wider but there are still laminate residues in the channel. In OD region, the widths of the channel are

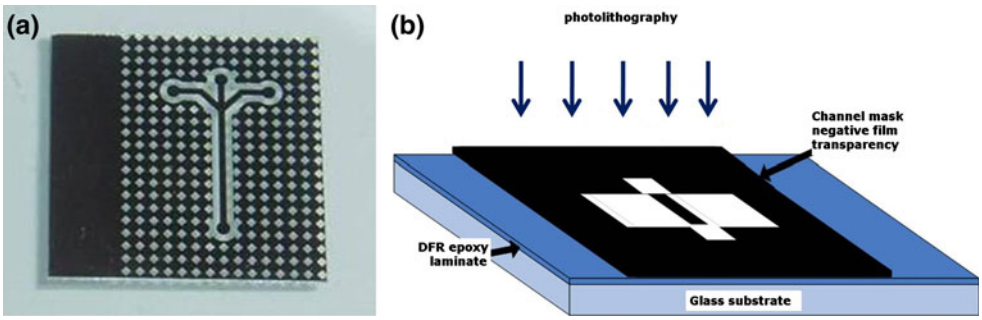

Fig. 3 **a** Acetate mask of the channel. **b** Photolithography technique

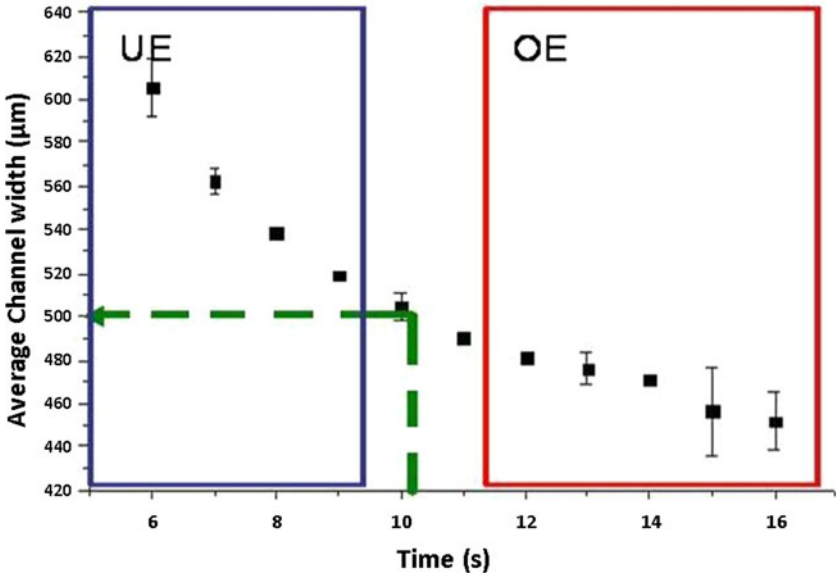

Fig. 4 Average of channel width. The *thick dashed line* indicates the possible time of exposing the resist under the UV light which will produce a good channel with correct width of 500 µm

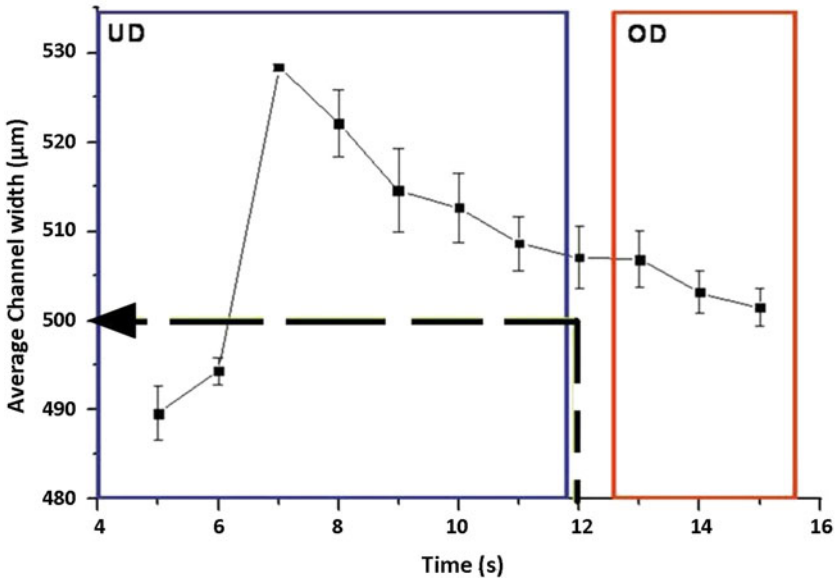

Fig. 5 Average of channel width versus developing time. The thick *dashed line* indicates the possible time of developing the resist in the developer solution which will produce a good channel with correct width of 500 µm

wider but the channel wall becomes non-uniform i.e. not straight. Below 5 s the channel width appeared to be narrowed since there is no formation of the channel at all. In this case, there is not much chemical reaction in between the chemical, BMR Developer and DFR, epoxy laminate.

A graph of laminate thickness versus developing time is plotted in Fig. 6a. In this graph the errors bar is considered. The thickness of laminates is started to decrease when the time of developing increases. The histogram of epoxy thickness versus developing time is shown in Fig. 6b. The graph also shows the log scale of the channel thickness versus develop time. The thickness of the channel at time = 12 s is reduced up to 10% from the original thickness of laminate. In addition to that, the colour of the channel observed is lighter than the original colour (blue), which indicates that the laminate is getting thinner because of the developer.

3.4 The fabrication of the channel

The first attempt in the experiment is to build the channel with two laminates and bond them on top of each other. The step by step process of forming the channel is shown in Fig. 7. First, the electrodes array is chosen to be at one side of the channel. The other side would be a glass with laminate on it to cover the open channel. This in turn will form a close channel.

Since two layers of laminate with 50 µm thick are used and pressed while they are still in the bonding process, the thickness of the laminates is further reduced. The depth of

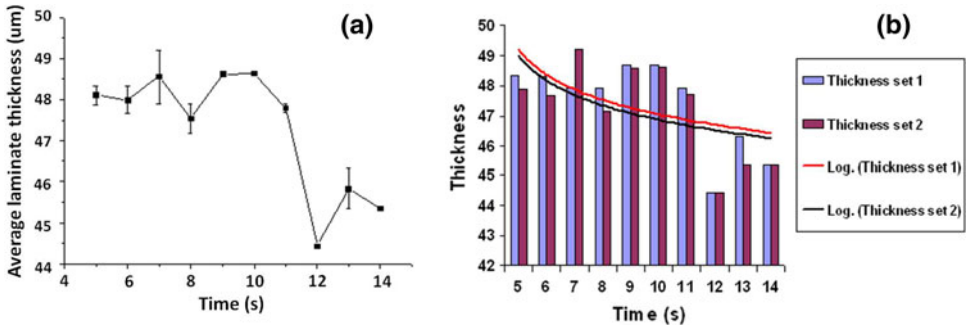

Fig. 6 **a** Average thickness of the epoxy laminate versus the developing time including *error bar*. At time 12 s the thickness of the laminate is 44 μm which will then give the short idea of the thickness of the channel after bonding process. **b** Histograms of the epoxy thickness versus the developing time and the log scale shows as the time increases the thickness of the laminate decreases

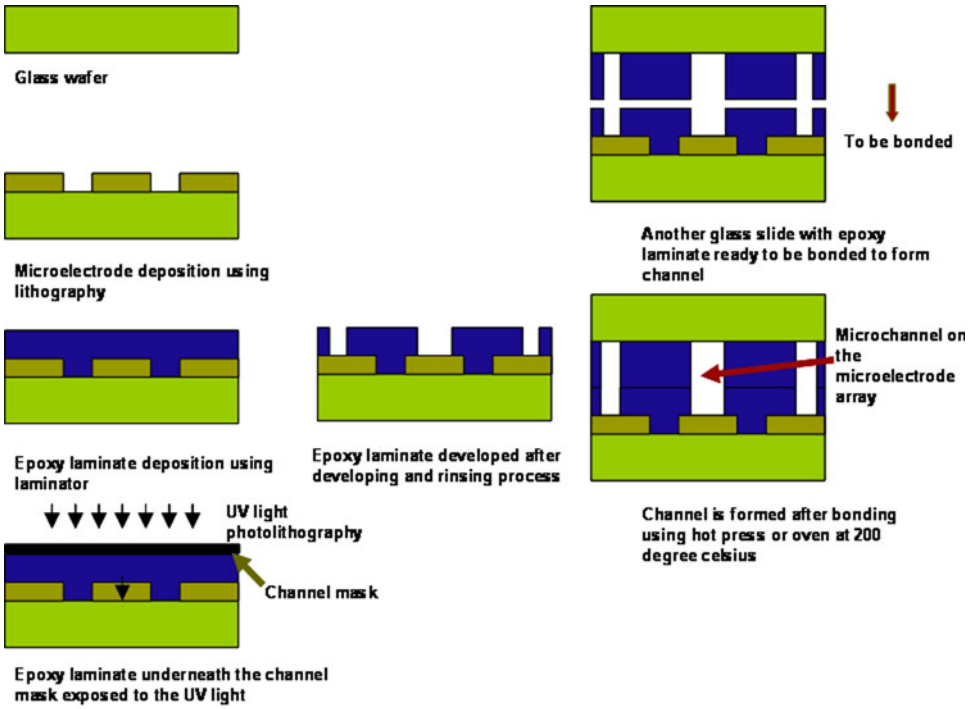

Fig. 7 Fabrication steps for forming the channel with two layers of laminates. From *top* to *bottom* on the *left hand side* of the diagram shows the empty glass wafer followed by the deposition of the microelectrodes using lithography (EBL) and then deposition of the epoxy laminate using the laminator. The process continued with UV light exposure to create the channel pattern and followed by the developing and rising process in the middle of the diagram. At the *right hand side* the two channels from two glass substrates are bonded and hence forming the close channel after placing them in the oven for 2 h at 200°C

the channel is measured by using the microscope set-up with resolution of 0.01 mm. The bonded close channel height is 75 μm. It is shown that the pressed laminate will decrease in thickness from 100 μm to 75 μm, which is a 25% reduction.

The measurement for the thickness of the laminate on the substrate without cover (top substrate) is done 'before' and 'after' it is cured. The uncured laminate with originally 50 μm thick, but it reduces in thickness from 4% to 48 μm because it has been rolled onto the substrate. The cured 50 μm laminate with its thickness is reduced to 41 μm. The percentage of reduction in thickness is approximately 20%. However, when it is pressed during the bonding process, it is reduced even further.

The next fabrication is done with only one side of the glass laminated and with electrodes array on top and bottom of the glass. This is performed due to the fact that the electric fields will become stronger when channel height is reduced. Since only one laminate is used, the thickness or the height of the channel is reduced to 37 μm. The channel height is reduced, compromising the desired separation operation where a device with a low aspect ratio is needed.

3.5 Bonding

The bonding process is a critical process in this fabrication. It will take one full day to complete. Figure 8a–e shows the result of bonded channel on the microelectrode array. If both substrates are manually aligned under a magnifier lamp before they are attached together, there might be slightly misaligned between the resist as shown in Fig. 8c.

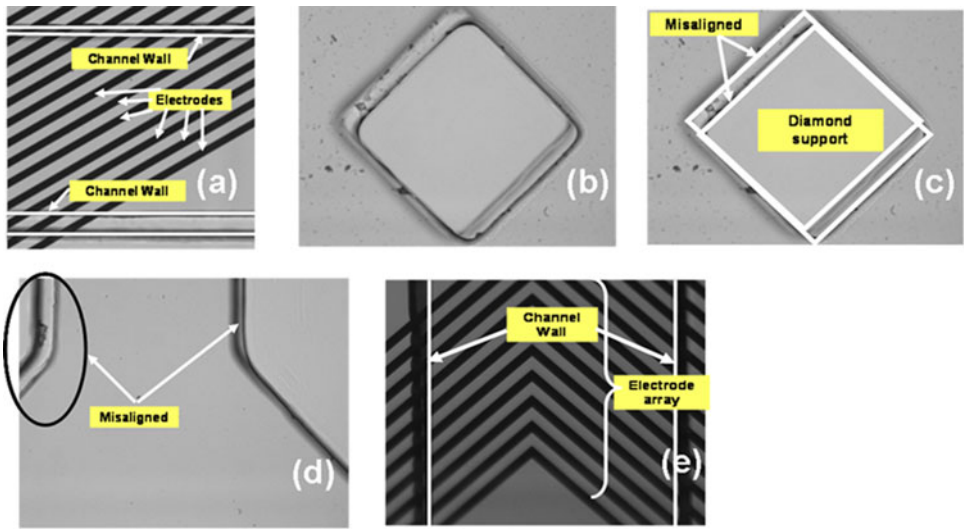

Fig. 8 **a** Bonded channel on angled electrode. **b** Bonded diamond as a support to increase the attachment of the *top* and *bottom* glass substrate. **c** Bonded diamond as a support are misaligned <5 μm from each other. **d** Bonded channel at the junction near the inlets with highlight of misalignment of the channel during bonding process. **e** Channel is shown bonded for V-shape microelectrode array

3.6 Hole drilling

Since the device is small and made of glass, it is important to exercise great caution when handling or drilling holes into the device. The machine used to drill hole on the device is from Proxxon GmbH with main adaptor NG 5/E. The drill bit used is a solid carbide drill, spade type, 60° point for glass from Drill Service (Horley) Ltd.

The process of drilling will generate heat when the drill bit spins and it may break the device. Therefore, to reduce heat, the chip is placed in the petri dish filled with water. The other factor to be considered is that the drill bit must be very sharp to avoid crack on the surface of the glass. The speed and the height of the drill bit must also be controlled.

The process of drilling starts after the chip hole is marked. To make sure that the hole is already drilled, the chip is tilted to see whether the shape of a cone has been formed on the both side near the drilled holes. When the cone is formed, the hole is successfully created. Finally, the channel is produced after the laminating and drilling holes are completed. This channel can be examined by running water into it using the syringe with the silicon tube. This tube is attached to the syringe and it is very flexible.

3.7 The chip

Figure 9 shows one of the bonded angled electrodes array chip that is ready for the experiment. The connecting wires are glued to the electrode. The glue used is the conductive epoxy from Chemtronics. It is cured at room temperature for about 4 h. The glue is recommended to be used if the electrodes array is made from the gold coated with titanium. However, if electrodes array is made from Platinum or Gold without titanium coating, the wire could be soldered directly to the electrode. The thickness of the

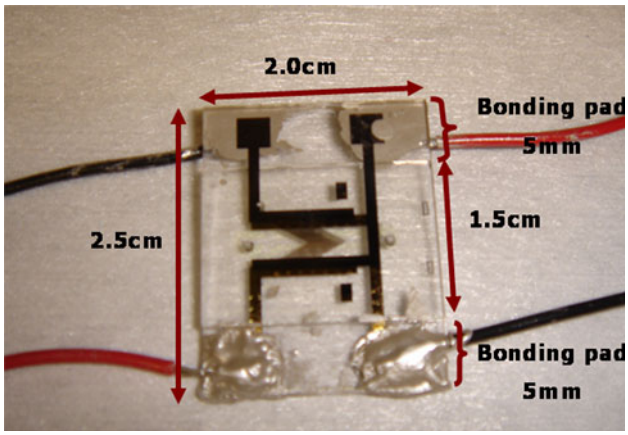

Fig. 9 The bonded angled electrodes array chip with all the dimensions

substrate glass is 1.1 mm. It is measured using digital calliper (0–150 mm) and the microscope camera is also available in the cleanroom Nikon LV-100D, DS-2Mv Digital Camera and using NIS Elements software. Both will give the same measurement of the glass thickness. The total thickness of the bonded angled electrodes array chip is approximately 2.21 mm thick. Figure 10 shows the image of the gap between bonded glasses and the thickness of the glass taken under the microscope camera. The separation of a mixture of colloidal particles was successfully accomplished with this microfluidic device that contains a microchannel as shown in Fig. 11.

4 Discussion

Finally, the channel is made from epoxy laminate. The width of the channel is 500 μm and the height of the channel is approximately 37 μm. Epoxy is a polymer and

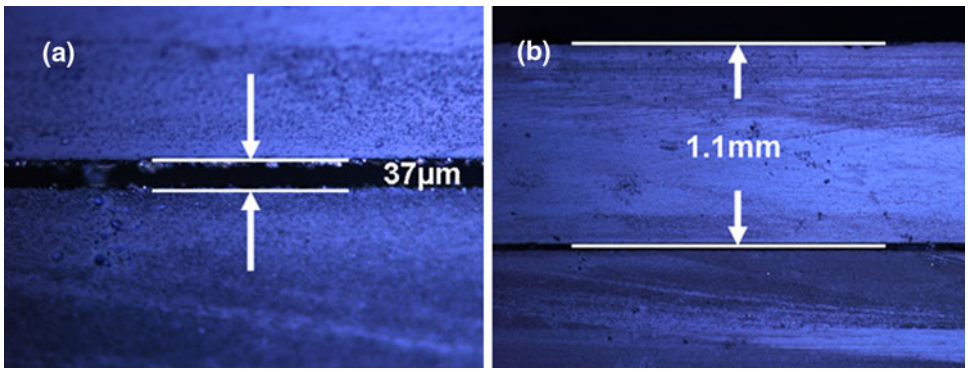

Fig. 10 **a** The gap between bonded glasses. **b** The thickness of the glass substrate scanned under the microscope camera available in the cleanroom Nikon LV-100D, DS-2Mv Digital Camera and using NIS Elements software

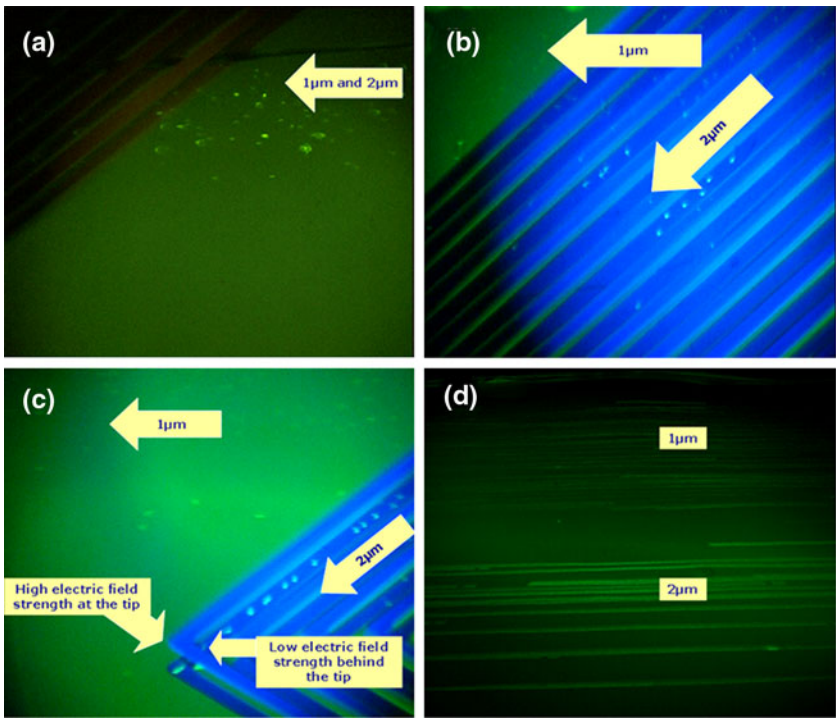

Fig. 11 **a** At the inlet of the channel, two particles are mixed. **b** While flowing along the electrode and experiencing the applied force, two particles starting to be separated. **c** 2 μm particles move towards very low electric field region while 1 μm move almost straight towards outlet. **d** Maximum intensity generated shows the particles were separated at the end of the electrode array

a thermosetting material. It can be cured using the temperature but cannot be dissolved after cured. The bonding process must be done very carefully. This is because in the bonding process, the heat is used to cure the epoxy. For uncured epoxy, acetone is used as a solvent. For cured epoxy, no solvent can be used to eliminate it. However, we tried to remove the unwanted cured channel by putting the device in the oven of temperature up to 500°C. The device is separated, the cured epoxy becomes dark powder, but, the electrode is appeared to be ruined by this process.

The bonding process is much longer if the oven is used. Bonding in the oven requires clips on both sides of the device. The applied pressure is not constant. For example, if each clip does not have specific pressure then pressure given will be varied. In this way, bonding using layers of glasses is performed under the clip. The more glasses used, the more pressure is given to the device. Moreover, for bonding in the oven, the clip must be placed in the middle of the channel. The channel is placed in parallel to the clip. This technique could increase the probability of channel to be bonded perfectly. The optimisation of the bonding process is still under investigation.

After the bonding process the channel is drilled to form a hole. If the channel has many inlets and outlets, more holes are to be drilled. The drilling process is done vigilantly to avoid any crack on the device. If the holes are drilled manually one by one, it might have a different diameter. This will cause some differences in input or output resistance. Once this occurs, there will be a problem if one is working to balance the sheath flow or to sort the particles at the output.

5 Conclusions

In this work, we have performed the microchannel fabrication using epoxy laminate that is rapid, economical and inherently suitable for fabricating disposable devices. These channels constitute a major challenge in the development of affordable (disposable) medical devices. We have also presented a DEP method for separating colloidal particles by using microfluidic device that consists of microchannel and demonstrates that the separation of particles can be accomplished with one layer of epoxy laminate as a channel. Future work will examine new thickness of epoxy laminate to improve the separation of smaller particles.

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