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Removable tubing interconnects for glass-based micro-fluidic systems made using ECDM

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Abstract

Reversible tubing connections for glass micro-fluidic systems are realized using electro-chemical discharge machining of three-dimensional glass vias. The connections reversibly connect standard sized plastic tubing to holes in borosilicate microscope slides. Tubing connections are demonstrated on a sealed, micro-fluidic channel which is fabricated between two glass slides using SU-8. The connections are experimentally tested to withstand up to 30 psi (~206 kPa) of air pressure without leaking.

1. Introduction

Chemical, biological and biomedical applications of micro-fluidics have rapidly increased during the past decade, due in part to the success of gene chip technology. However, very few examples of entirely autonomous micro-fluidic systems have been demonstrated. Most require some sort of fluidic connection to the macro-world, especially for delivery of samples and/or reagents. A variety of interconnection schemes have been introduced in the literature, but most involve permanent attachment of tubing to the micro-fluidic device [1, 2]. A few have addressed reversible interconnects [3, 4]; however, they are for silicon-based micro-fluidics. Glass is the preferred material for many micro-fluidic applications because of its combined properties of hydrophilicity, electrical insulation and transparency. However, the attachment of small diameter plastic tubing to small holes in glass has not evolved much beyond simple gluing with silicone rubber or epoxy. Adhesives pose numerous problems, including contamination and non-reversibility, in addition to the difficulty in minimizing the size of the connection while maximizing the seal pressure. In this paper, the fabrication and testing of removable and re-attachable interconnections to glass-based micro-fluidic systems are investigated. This feature is useful for prototype development of micro-fluidic devices as well as for disposable and modular systems.

The fabrication of glass-based interconnects involves machining vias with three-dimensional features into glass slides. The most common methods of machining glass are by ultra-violet (UV) laser ablation and wet etching in hydrofluoric (HF) acid. Laser ablation can produce high-aspect ratio features, but it requires expensive lasers and optics. HF etching is a low cost method, but it cannot produce high-aspect ratio features in glass. Electro-chemical discharge machining (ECDM) of glass is a relatively new, alternative machining method that is both low cost and capable of producing high-aspect ratio etch profiles. Previous research groups have used this method to machine holes and micro-fluidic channels in glass [5]. However, to the authors' knowledge, this is the first time it has been used to machine three-dimensional, complex structures in glass. Specifically, in this paper, we exploit the effects of ECDM to machine hourglass-shaped vias and threaded vias in glass.

2. Electro-chemical discharge machining (ECDM)

The mechanism of ECDM follows directly from the more familiar electro discharge machining (EDM). In EDM, a high voltage is applied between two conductors submersed in an insulating medium. This initiates an electrical discharge, or spark, when the conductors come into close proximity. The bulk of the discharge current is usually carried by electrons and does not contribute to material transport, i.e. machining. However, positively charged atoms sputtered from the electrode surface results in material transfer into the liquid. Electro-migration of the electrode material takes place within the conductive plasma of the discharge. By

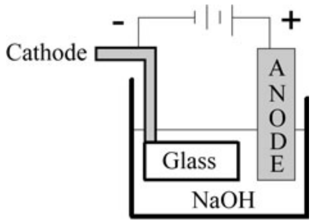


Figure 1. Basic physical set-up for ECDM.

proper choice of polarity and electrode material, one electrode is selectively machined and the other serves as the die or machining electrode. Carbon is typically the material of choice for the machining electrode because of its low electromigration and relatively high conductivity [6].

Although there are similarities between ECDM and EDM, the detailed mechanisms involved in ECDM are not well understood. ECDM employs an electric discharge, but the machined substrates are typically insulating. The physical set-up for ECDM is shown in figure 1. The cathode and anode are immersed in a highly conductive, aqueous electrolyte, such as sodium hydroxide (NaOH), and a voltage (20–60 V) is applied between them. Machining is a combination of both physical and chemical reactions. By applying a voltage to the electrodes, the electrolysis of water takes place. Electrolysis generates gas bubbles at both electrodes: H_2 at the cathode and O_2 at the anode. The cathode is then brought into direct contact with an insulating substrate, such as glass, and an electric discharge occurs within the electrolysis bubbles. It has been postulated that the discharge induces removal of the substrate material, which is then complexed and solubilized by the NaOH [5]. In addition, NaOH in solution increases the hydroxide concentration, improves the conductivity of water, and therefore, increases the rate of this reaction. How the discharge 'induces' silicate removal is open to some debate. Localized Joule heating may 'melt' or soften the silicate. Ghosh [7] reported temperatures between 8000 K and 10 000 K, using thermocouples to measure the localized temperature. However, there is some question whether Ghosh measured the temperature of the glass or of the discharge. Local sputtering through the gas phase is also a suggested mechanism. Additionally, machining is polarity dependent, which implies that spark ignition of electrolysis-generated hydrogen gas could also assist in material removal.

3. ECDM experimental set-up

ECDM depends on several parameters, such as the electrolyte solution, the electrode material, the size of the electrode and the voltage applied to the electrodes. Therefore, preliminary experiments were performed to determine the critical voltage value when sparking is seen and glass machining begins (table 1) for different electrolyte concentrations and electrodes. The results of these experiments confirmed prior reports [8] of the dramatic increase in etch rate with the onset of sparking as well as the reduction in sparking voltage with decreasing electrode diameter. In addition, table 1 shows the dependence of etch rate on electrolyte concentration below and at the sparking voltage. A maximum etch rate is obtained at 10% NaOH concentration; therefore, it is used for all subsequent ECDM experiments. The cathode electrode material is chosen based on the surface quality of the through-hole during machining as explained below. A 1 inch square nickel gauze is used as the anode electrode.

Via formation begins with the machining of through-holes in borosilicate glass. This is done using a 0.5 mm diameter platinum wire as the working (cathode) electrode. The glass substrate to be machined is submerged and suspended within the NaOH solution. There is about 2 mm of solution above the glass slide. The working electrode makes direct contact with the substrate during the machining process. The electrochemical discharge begins when the voltage applied to the electrodes reaches a certain critical value [7], at which point, the working electrode will begin to spark with a bright, orange glow. With a 10% NaOH solution and a 0.5 mm diameter Pt cathode, the critical voltage is observed at 47.8 V.

A problem, encountered during initial trials and discussed in prior reports [9], is the accessibility of the electrolyte at the working site as the drill depth increases. If the electrolyte cannot reach the working electrode, the electrochemical reactions cease and glass machining terminates. In addition, a temperature increase, associated with the lack of electrolyte at the working site, can cause the glass sample to crack. To ameliorate these issues, tool holders capable of vertical motion are implemented. By vertically moving the working tip into and out of the working site, fresh electrolyte is constantly supplied and the electrochemical reactions are able to continue at a near constant rate each time the tip is brought into contact with the surface. Vertical motion is provided in our

Table 1. Experimental test data which compare electrode material, electrode diameter, NaOH concentration, no sparking/sparking voltage and etch rate in borosilicate glass.

Electrode material	Electrode diameter (mm)	NaOH concentration (%)	No sparking/ sparking	Voltage (V)	Etch rate (mm min^{-1})
Nickel	0.38	30	No sparking	23.0	0.0088
Nickel	0.38	30	Sparking	27.7	0.042
Nickel	0.38	25	No sparking	25.3	0.0122
Nickel	0.38	20	No sparking	35.4	0.0128
Nickel	0.38	20	Sparking	39.0	0.102
Nickel	0.38	10	No sparking	31.1	0.0148
Nickel	0.38	10	Sparking	48.4	0.130
Nickel	0.25	10	Sparking	43.3	0.160
Nickel	0.38	8	No sparking	32.8	0.0137
Nickel	0.38	5	Sparking	51.1	0.100
Platinum	0.50	10	Sparking	47.8	0.133

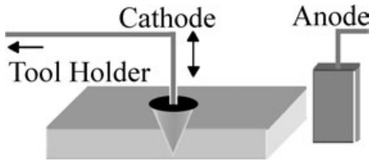
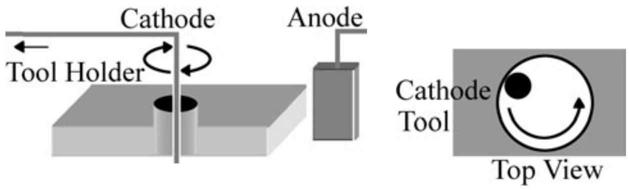


Figure 2. ECDM set-up with vertical moving tool.



(*a*) (*b*)

Figure 3. (*a*) ECDM set-up with a moving working tool, and (*b*) top view indicating cathode circular motion inside hole.

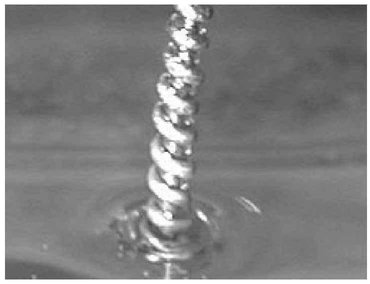


Figure 4. Photograph of a working tool for forming threaded interconnects.

set-up by means of a solenoid driven tool holder. Custom drive circuitry provides control of cycle rate, dwell time and amplitude. The tool is driven at 1 Hz, with a displacement amplitude of about 1 mm. With this set-up, tapered through-holes up to 2 mm deep are readily achieved (figure 2).

The surface quality of the machined hole is strongly dependent on the type of working (cathode) tool used. Holes machined with nickel wire have very rough surfaces with frequent instances of surface cracking. In comparison, holes machined with platinum wire appear to be very smooth with no instances of cracking. Possible explanations for this behavior may be derived from the observed differences in the rate of electro-migration of the two metals when used as the cathode during ECDM. When sparking, and thus ECDM commences, platinum begins immediately to erode away, leaving a visible trail of black platinum-containing particulates in the solution. Conversely, nickel does not visibly erode during the ECDM process. One speculation for the difference in etch surface quality is that the platinum removal dissipates and distributes the electrical energy over a larger area, such that the glass surface sputter rate is lower and more uniform. Earlier literature reports that smooth surfaces resulted from the complete complexation of silicate [5] during the chemical reaction between the sodium hydroxide and the glass,

$$2NaOH + SiO_2 \Rightarrow Na_2SiO_3(s) + H_2O.$$

Complete complexation is likely to be more difficult to achieve during a high removal rate of silicate, as in the case of a high rate of localized sputtering, if there is limited availability of the electrolyte. This may be what occurs during ECDM with a nickel cathode.

With the set-up shown in figure 2, a tapered hole results. This is because the machining rate is determined in large part by the field distribution between the working electrode and the glass surface just prior to discharge. As the tool is moved vertically out of the hole to allow fresh electrolyte to flow, no vertical etching occurs. This is due to the increased distance (reduced field) between the tip and the glass surface at the bottom of the hole; however, horizontal etching of the sidewalls can still take place. When the tool is lowered to the bottom of the hole, the highest field, and therefore highest etching rate is at the tip. As a result, a conical-shaped hole forms, where the entering hole size is considerably larger than the exiting hole size. For example, a through-hole, formed by a 0.5 mm Platinum wire, in a 1.0 mm thick glass slide will have an entry hole diameter of 0.65 mm, versus an exit hole diameter of 0.25 mm (taper = 21.8°). To minimize side-wall etching, the voltage supply is turned off as the working tool moves upward and is turned on again as the tool moves downward.

In order to machine cylindrical, smooth sidewalls (which are needed for the imprinting of threads), a cone shaped, through-hole is first formed using the set-up described above. Then, the sample is inverted, and the working electrode is placed inside the through-hole and moved in a circular motion (figures 3(*a*) and (*b*)) around the edge of the hole. In this set-up, no vertical motion is used. The circular motion is imparted to the tool through a manual joystick actuator. An adjustable conical limit stop on the joystick arm provides repeatable rotational displacement magnitude to the tool according to its vertical position on the arm. A 0.25 mm platinum wire is used as the working electrode with 40.0 V applied voltage. Since the working tool is placed within a through-hole, fresh electrolyte should ideally reach the working site. However, since the diameter of the exit hole (0.25 mm) is very small, a lack of enough fresh solution re-circulating around the hole is possible, which can lead to overheating and cracking of the glass at the working site. To allow more time for the electrolyte to re-circulate, the electrode voltage is pulsed on and off at 1 Hz during ECDM machining. Under these conditions, ECDM continues until the through-hole achieves a cylindrical shape with diameter of approximately 1.0 mm. This typically takes 3 min.

The ECDM set-up shown in figure 3 is also used to imprint threads on the inside walls of a hole. All of the experimental parameters remain the same except for the type of working electrode. The platinum wire used to make a smooth, cylindrical hole is replaced with a coil made of nickel wire (figure 4). Initially, the use of a coil consisting of platinum wire was attempted; but, the threaded patterns did not transfer as sharply as those made with the nickel wire. To form the coil for the thread imprinting tool, a 0.25 mm nickel wire is wrapped around another 0.25 mm nickel wire. This wire is then attached to the tool holder and moved in a circular motion, with an applied voltage of 40.0 V, for approximately 2.5 min, to transfer the threaded pattern to the glass.

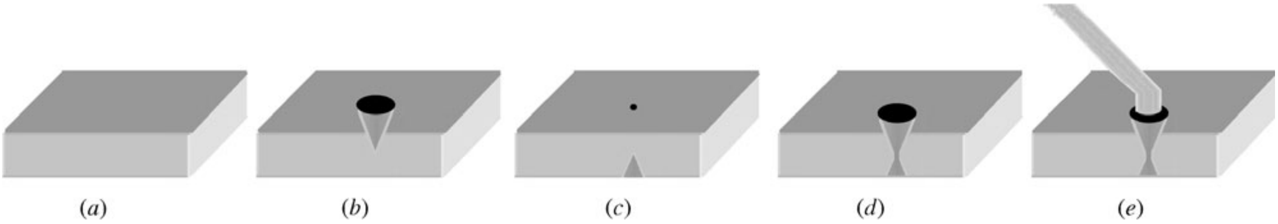


Figure 5. Hourglass fabrication process: (*a*) clean glass slide, (*b*) use ECDM to machine a hole, (*c*) flip glass slide over and mark, (*d*) use ECDM to machine another hole, and (*e*) secure tubing.

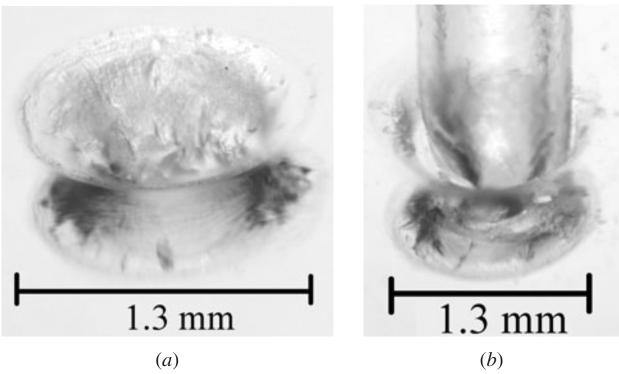


Figure 6. Hourglass interconnect: (*a*) detailed view of hourglass and (*b*) hourglass with tubing attached.

4. Fabrication and assembly of interconnects

4.1. Hourglass interconnect fabrication

This interconnect employs an hourglass-shaped via in a glass substrate to secure flexible tubing. It is a simple structure to fabricate and use, but it is limited in working pressure, and therefore, application. Figure 5 illustrates the fabrication process. The hourglass-shaped vias are machined on standard 1 mm thick microscope slides (Fisher Scientific).

The fabrication begins with cleaning the glass slides in acetone, followed by rinsing in methanol and de-ionized water. In terms of functionality, smooth sidewalls are not necessary, therefore a nickel wire, which has a longer lifetime and lower cost, is used as the working tool. The set-up shown in figure 2 is used to make a conical hole, using a 0.38 mm diameter nickel wire and 48.4 V. The rate of ECDM using these parameters is about 2.16 μm s^{-1} (0.65 mm/5 min). The glass slide is then flipped over and a second hole is machined to meet the first. The 'waist' diameter of the via increases as machining time increases. A photograph of the completed via is shown in figure 6.

Flexible plastic tubing, with an outside diameter (OD) that is slightly larger than the 'waist' diameter of the via, is pressed through the via hole. This tubing becomes compressed at the center of the via and is held in place. Since no adhesive is required, the tubing can be removed and replaced as desired. Silastic tubing (Fisher Scientific) is chosen because of its elastic properties and large working temperature range. For a via with a waist diameter of 0.565 mm, Silastic tubing of 0.96 mm OD is used. To facilitate insertion of this very flexible tubing, the end of the tubing is cut diagonally, to produce a sharp tip. After pressing through the via, the protruding end is cut off, flush with the glass slide. Because of the large working temperature range of Silastic tubing (−54 to 249 °C), tubing attachment, prior to adhesive bonding of the glass slide to a micro-channel (as described later), is feasible. Stiffer tubing, such as Teflon tubing, can also be effectively pressed into the hourglass vias of a sealed channel, i.e. after bonding.

4.2. Threaded interconnect fabrication

Threaded vias are machined on standard 1 mm thick microscope slides (Fisher Scientific). The process is illustrated in figure 7.

The glass slides are cleaned in acetone, followed by a rinse in methanol and de-ionized water. A permanent marker is used to mark the desired location of the holes, for example to correspond with fluidic micro-channel inlet and outlet reservoirs. A 0.5 mm diameter platinum wire is used as the working electrode. The microscope slide is submerged into the NaOH solution with about 2 mm of solution above the slide. A voltage of 47.8 V is applied to the electrodes for 7 min to initially drill conical through-holes with smooth sidewalls. The ECDM drilling rate for these holes is about 2.38 μm s^{-1} (1 mm/7 min). The length of platinum wire that is typically consumed during ECDM machining, through a 1 mm microscope slide, is about 0.5 mm. The glass slide is then flipped over and a 0.25 mm diameter platinum wire is aligned and inserted into the center of the small, backside hole. Using the tool holder with circular motion and a pulsed power supply signal, 40.0 V is applied to the electrodes for 3 min to enlarge the backside hole, and to smoothen and straighten the sidewalls to form a cylindrical via.

To imprint the threads, a coil of nickel wire (shown in figure 4) is employed as the working tool. The coiled wire is aligned to the center of the glass through-hole. The 1 mm microscope slide is then submerged 1 mm into the solution such that hardly any solution resides above the slide. With an applied, pulsed voltage of 40 V for about 2.5 min, the tool holder is moved in circular fashion and makes direct contact with the through-hole's sidewalls. Photographs of the resulting smooth cylindrical via and threaded via are shown in figure 8.

Using the threaded via as a master mold, Kynar™ (Polyvinylidene Fluoride, PVDF), a thermoplastic, is melted and compression-injected [10] into the master to form mating plastic screws. The process is illustrated in figure 9. First, the microscope slide containing the threaded vias is placed on a temperature-controlled hotplate, and heated to 245 °C. In order to create a 'thumb screw', where the head of the screw is large enough to be easily handled, a 7/32 inch OD (3/16 inch ID), metal tube is centered over the threaded via to size the head. The Kynar™ thermoplastic is shredded into tiny shavings to expedite complete melting. The shavings are

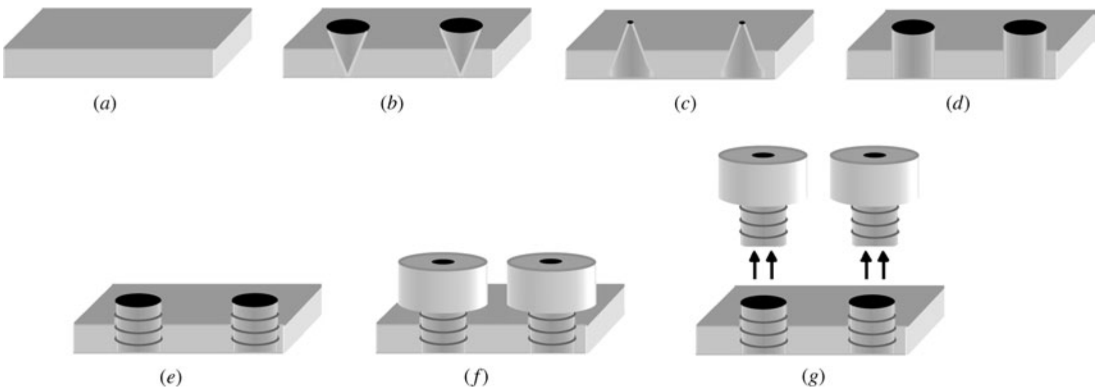


Figure 7. Threaded interconnect fabrication process: (*a*) clean glass slide, (*b*) use ECDM to machine conical holes, (*c*) flip glass slide over, (*d*) use ECDM to machine vertical sidewalls, (*e*) use ECDM to imprint threads, (*f*) mold plastic screws, and (*g*) remove plastic screws.

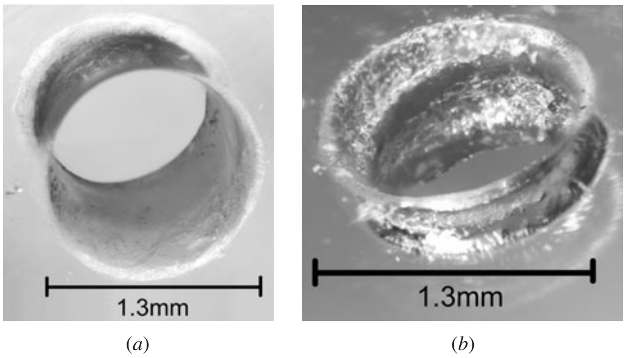


Figure 8. Threaded interconnect: (*a*) cylindrical hole and (*b*) final threaded interconnect.

placed into the metal tube and allowed to melt for 2 min. A 3/16 inch diameter, solid metal rod is then placed into the hollow tube and a slight pressure is applied to the rod for an additional 2 min. This forces the melted plastic shavings into the threaded via master mold. While continuing to apply pressure to the solid metal rod, the microscope slide is removed from the hotplate and allowed to cool. The result is a molded, plastic screw that is mated to the threaded via.

In order to use the plastic screw as a removable, micro-fluidic interconnect, a through-hole in the plastic screw is necessary. This is accomplished by placing a 0.25 mm metal wire in the center of the master mold prior to heating. During the heated compression, the wire reserves a space for the through-hole. After the molded, plastic screw cools, the wire is easily removed since the thermoplastic does not adhere to metal. This completes the fabrication of the threaded interconnects. Photographs of the molded plastic screw are shown in figure 10. The screws are removed from the microscope slide, and a milling machine is used to carefully drill an enlarged hole (1.2 mm) inside the head of the plastic screw. The plastic tubing can then be inserted and secured to this enlarged hole. Of course, the head, the plastic threads and the center hole can be formed in one step, by using an appropriately machined mold in place of the metal tube and wire.

To finish the assembly of the threaded interconnect, tubing must be attached to the plastic screw. Using a soldering iron, the ends of a 1.22 mm OD polyethylene tubing (Intramedic) are heated to create a flared shape. The tubing is then inserted into the hole previously milled in the top of the screw's head. A 2-part epoxy (2 Ton^{TM} Clear Epoxy) is used to affix the tubing to the plastic screw. Once the epoxy has fully set, this interconnect can be screwed back into the threaded hole on the sealed channel.

Small, silicon rubber gaskets are made and inserted between the plastic screw head and the top glass surface to improve sealing. Using a flat, metal plate, a drop of re-flowable silicon rubber is compressed between two Mylar sheets. After the silicon rubber sets, it is peeled off from the Mylar sheets and cut into tiny, rubber gaskets. The gasket slips over the threads of the plastic screw before it is inserted into the via.

4.3. Micro-channel fabrication

A micro-fluidic channel with tubing interconnects is fabricated by the bonded assembly of two glass slides: one supporting a micro-channel defined in SU-8, and the other containing either the threaded vias or the hourglass-shaped vias. The process is illustrated in figure 11. Channel fabrication begins by cleaning a standard 0.15 mm thick, glass cover slide in acetone, followed by rinsing in methanol and de-ionized water. The cover slide is then dehydrated at 200 °C for at least 5 min on a hotplate. Photo-curable epoxy, SU-8 2015, is dispensed onto the cover slide and spun at 2000 rpm for 30 s, resulting in an SU-8 layer thickness of 15–20 μm. The coated cover slide is then soft-baked for 1 min at 65 °C, and then further soft-baked for 3 min at 95 °C. Next, the coated cover slide is exposed to UV through a photo-mask containing the micro-channel and reservoir pattern. After exposure, a hard-bake at 95 °C for 1 min is performed to cross-link the exposed SU-8 regions. The fabrication of an open micro-channel is complete after development of the channel in SU-8 developer for 3 min.

The micro-channels are sealed by bonding the via-containing glass slide, hereafter called the 'glass cap', over the open micro-channel. First, the glass cap is carefully cleaned using a piranha bath solution (3:1, H_2SO_4:H_2O_2) at 80 °C. This insures thorough removal of any thermoplastic that may have stuck to the surface during molding, and guarantees good adhesion between the SU-8 and the glass surface. A lower viscosity epoxy, SU-8 2002, is spun onto the glass cap at 1000 rpm for 30 s, resulting in an SU-8 layer thickness of 3 μm. The glass cap is soft-baked for 1 min at 65 °C, and

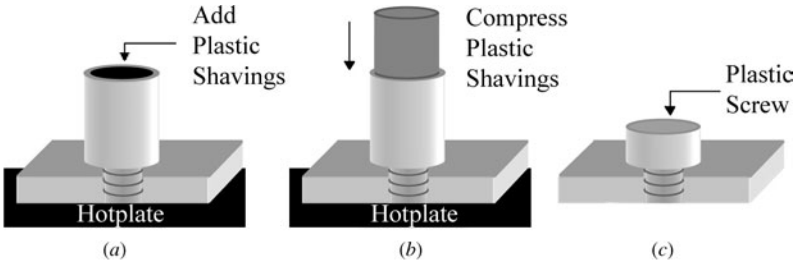


Figure 9. Molding process: (*a*) a metal tube is centered over the threaded via and plastic shavings are added. (*b*) Plastic shavings are compressed using a solid metal rod. (*c*) The final result is a molded, plastic screw.

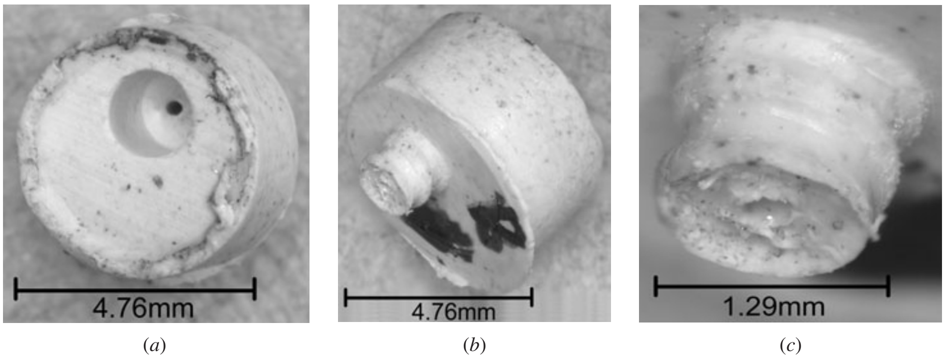


Figure 10. Plastic interconnect screw: (*a*) top view, (*b*) side view and (*c*) detailed view of threads.

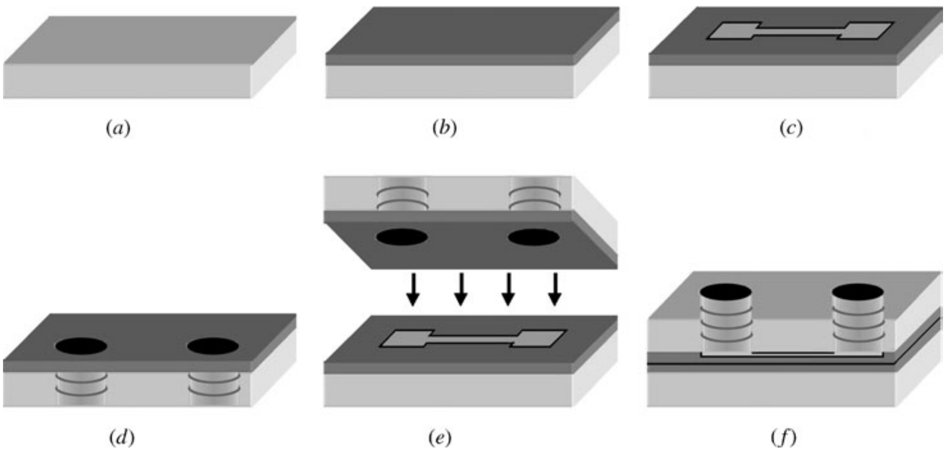


Figure 11. Micro-channel fabrication: (*a*) clean glass slide, (*b*) spin on SU-8 2015, (*c*) expose, bake, and develop, (*d*) spin on SU-8 2002, (*e*) align threaded holes to micro-channel, (*f*) bond slides.

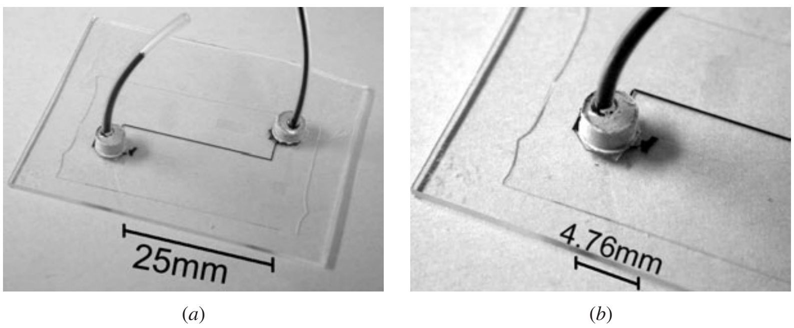


Figure 12. Micro-fluidic device: (*a*) entire view of device and (*b*) detailed view of threaded interconnect.

further soft-baked for 2 min at 95 °C. Following the soft-bake, the vias in the glass cap are aligned to the channel reservoirs on the micro-channel slide. The two, aligned glass components are then pressed together and heated to 100 °C on a hotplate. The thin, unexposed SU-8 layer adheres the glass cap to the micro channel slide. A similar bonding technique is previously described in the literature [11, 12]. The bonded slides are then exposed through the photo-mask and hard-baked,

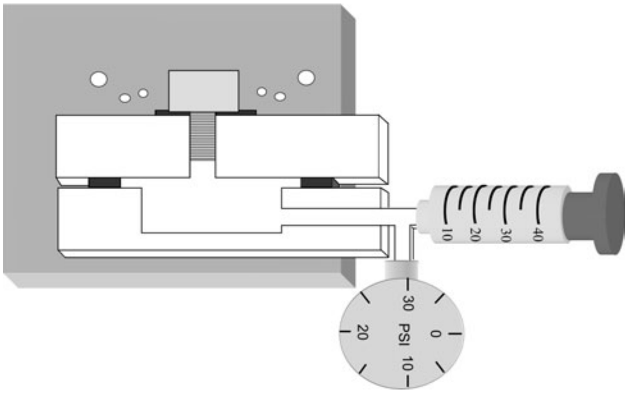


Figure 13. Cross-section of the leakage test set-up. A syringe, loaded with air, is connected beneath the air chamber. A plastic screw in threaded glass is placed over the O-ring seal and is mechanically clamped over a small chamber.

cross-linking the exposed SU-8 regions. Exposure takes place through the 150 μm glass cover slide side, to minimize exposure distance. The fabrication of the sealed channel is complete after developing the channel in SU-8 developer for 3 h. This long developing time is necessary to insure complete removal of the unexposed SU-8 residing inside the sealed channel. The development time can be shortened by agitating the developing solution; however, it should be noted that this process was not optimized for patterning and bonding. The goal was to simply obtain a clean channel for experimental testing of the interconnect technologies.

5. Experimental testing and results

5.1. Threaded interconnect

An assembled, micro-channel device with threaded interconnects is shown in figures 12(*a*) and (*b*). The tubing and channel are filled with water and food coloring for visual clarity.

Experimental testing of the threaded interconnects consisted of a leakage test under pressure. In an attempt to determine the maximum pressure that the threaded interconnect could withstand before leaking, a plastic screw without a center-hole was inserted into the threaded via and the glass slide was then mechanically clamped over a small chamber and sealed by an O-ring (figure 13).

A syringe, loaded with air, was connected to the chamber, and the glass, with the screw, was immersed in DI water. As the syringe was compressed, the increase in air pressure inside the chamber was monitored by a pressure gage. When air bubbles were first observed to appear around the screw, the pressure was recorded. Six plastic screws were fabricated and tested in this manner for leakage. All six screws performed satisfactorily (no bubbles) up to 30 psi (~206 kPa), which is the maximum pressure that could be safely applied in this set-up.

5.2. Hourglass interconnect

The hourglass-shaped interconnect was pressure tested using the same technique as above. In this case, the tubing was clamped shut during testing. A syringe, loaded with air, was again connected beneath the chamber, and the topside of the glass was immersed DI water. The pressure at which these interconnects failed (bubbles appear) was measured to be 15 psi (~103 kPa).

6. Conclusion

Reversible interconnections for glass-based micro-fluidic systems have been developed using ECDM to machine three-dimensional vias in glass substrates. Plastic tubing can then be reversibly attached to these vias. Two types of vias were demonstrated: an hourglass-shaped via and a threaded via. Mating plastic screws (for the threaded interconnects) were made using compression molding. Experimental testing demonstrated that the threaded and hourglass-shaped interconnects withstood air pressures up to 30 psi (~206 kPa) and 15 psi (~103 kPa), respectively, without connection failure (leakage).

Acknowledgments

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