

Toward the Fabrication and Testing of All-Polymer Micronozzle

by

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Abstract

A disposable all-polymer micronozzle was designed and fabricated by merging the two different technologies of microfluidics and microneedles together. Polymer micronozzles (polyimide and SU-8) were fabricated using different steps of spin casting and one step of photolithography.

Microfluidic devices consisting of one input channel and one output channel each with a 500 μ m diameter, and connected with a channel 100 μ m in width, were fabricated using the PDMS polydimethylsiloxane (PDMS) casting. To achieve a thin PDMS membrane, spin casting of PDMS over the mold is required. The fabricated thin PDMS microfluidic layers were bonded to polymer nozzles using oxygen plasma treatment and precisely aligning the two layers together.

The resulting polymer nozzles were connected to the pressure system of a custom made inkjet printer, by the means of a plastic holder device. The holder device was designed in SolidWorks and printed using a 3D printer. Finally a solenoid actuator was attached to the setup. Different solenoid plunger tips were designed to maximize the deformation of the PDMS membrane which is used to attempt liquid ejection and printing. First the internal pressure was tuned. The effect of frequency, duty cycle and input voltage of the solenoids input pulse on the created pending droplet's volume was characterized experimentally. The maximum displaced volume was found with actuation for a 12V input pulse with 10% duty cycle. For a 50 μ m nozzle diameter this volume is 4.78×10^{-11} L and for a 200 μ m it is 3.83×10^{-10} L. Reducing the surface tension of water using surfactant resulted in flow of ink onto the hydrophilic plasma-treated SU-8 surface, and total surface wetting.

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To my beloved parents,

1. Introduction

1.1. Background

1.1.1. Inkjet Printing

Micro-drops, i.e., small droplets with diameters ranging from a few microns to several hundreds of microns, have been applications in different research areas such as fluid dynamics, chemistry [1]. In addition to these, micro-drops have the potential to revolutionize a vast range of other research areas such biotechnology and biomedical engineering applications (Cell sorting [2], DNA microarrays [3] and DNA synthesis [4], drug discovery [5] and medical therapeutics [6]). In addition to biotechnology and biomedical research, micro-drops have made their way into improving different areas in engineering and manufacturing. Optics [7], droplet-based manufacturing [8], inkjet soldering [9], IC manufacturing [10], precision fluid deposition [11], large area displays and many other applications fall under this category [1]. For all of the above applications, the generated droplets should be formed in uniform volumes. Micro-drop ejectors with the capability of producing monodisperse droplets have one characteristic in common; they all take advantage of high-speed fluid jets of nearly the same diameter as the desired micro-drops. The created fluid jets are then disturbed with an actuator pulse that creates sufficiently high kinetic energy to create microdrops.

The most common method of creating micro-droplets in all of the above applications is inkjet printing. Inkjet printing can yield thicknesses less than $0.5\mu\text{m}$ and feature sizes between 20-50 μm . The inks can include polymers, composites, solvents, living cells, and a wide variety of functional materials including conductive materials such as silver particles. They have been used for printing Microelectromechanical (MEMS) systems [12], conductive traces for circuits [13], electronics [14], micro-electronics[15] and bio-printing[16]. Inkjet printing is considered to be a

low cost fabrication method for fabricating complex integrated circuits consisting polymeric thin-film transistors (TFT) [17]. One of the most recent advances in inkjet printing was in the area of medicine and biomedical engineering. This technology has been adapted to applications such as drug screening, genomics and biosensors. High density micro-arrays of cells and DNA have been printed using high resolution commercial glass nozzles [18]. Inkjet-printing of proteins on cellulose paper has been previously reported [19]. In addition to cells and proteins, biosensors have been created by inkjet-printer deposition on glass [20]. There are reports of 3D cellular structures created by inkjet printing which is called Organ Printing [21]. Organ printing and drug screening are among the most recent applications of inkjet printing. For this purpose all the supportive materials for the living cells should be simultaneously delivered in the right environment for the cells. One of the necessities among many others for printing tissues is that all the cells are printed and kept alive during the process.

The most common inkjet printing technique is the DOD (i.e., Drop-on-Demand) inkjet printing in which each voltage pulse corresponds to one droplet being ejected out of the nozzle. There are different actuation types used for DOD inkjet printing such as thermal and piezoelectric [22].

1.1.2. Microfluidics

Microfluidic devices are used for precise control and manipulation of sub-microlitre volumes of liquids [23]. The use of microfluidic devices has revolutionized biomedical research. Since most of the reagents used for biomedical engineering purposes are expensive, ability to handle them in nanolitre-sized quantities is a great advantage to this research area. The fabrication techniques for these microfluidic devices are amenable to mass production. It is possible to perform different functions on the same chip in as analogous manner to an integrated circuit. A majority of research microfluidic devices are fabricated from polydimethylsiloxane (PDMS). PDMS is

transparent, non-toxic, non-flammable and also inexpensive for fabrication. Its unique characteristics have made it a great candidate for developing microfluidic devices using soft lithography [24], the primary technique for fabricating micro-channels and microstructures out of PDMS.

1.1.3. Microneedles

Another recent development in biomedical applications in recent years has been the fabrication of microneedles for transdermal drug delivery into the human body [25]. There are several techniques and materials used for fabricating the microneedles. Some of the materials include silicon [26], metals [27], polymers [28] and other materials with feature sizes ranging between sub-micron to millimetre dimensions.

Several reports of cell printing using different non-polymer commercial nozzles have been published in literature [18][29][30]. The nozzles mentioned in these applications are not disposable. The prefabricated glass and metal nozzles are expensive and require delicate handling [22]. In this project a novel all-polymer micronozzle has been introduced that is aimed to overcome these problems.

1.2. Research Objectives

In this project we design and fabricate an all-polymer micronozzle design that is supposed to handle a much broader range of liquids including complex inks and biological materials with respect to previously developed techniques. By taking advantage of microfluidics technology and combining it with the microneedle technology we introduce a new fabrication process aimed for manipulating and printing those materials in real time. The proposed ejection system is intended to be high throughput and cost effective. The disposable all-polymer micronozzle is fabricated by

combining three main parts of polymeric micronozzles, microfluidic channels and a separate solenoid based actuation system. The microfluidic system can have modular designs to meet the specifications of various applications.

Using the new fabrication method for polymer micronozzles [28], a wide range of high resolutions (between 30 to 200 μm) can be achieved. The microfluidics will give the system the ability of on-chip analysis and handling of a wide range of liquids with different material properties. All of the materials used in this device are considered low cost, with the aim of allowing the user to dispose of the microfluidic chip and nozzles after each use and therefore limiting cross-contamination.

The main objectives of the present project are:

- design and fabrication of polymer micronozzles with different nozzle diameters
- design and fabrication of microfluidic channels matching the micronozzles design
- developing a technique to bond between the microfluidic device to the micronozzles
- choosing the actuator which is separate from the micronozzles (it does not add to the fabrication cost)
- tuning the internal pressure of the micronozzles to create a stable meniscus
- tuning the input voltage signal of the actuator in order to eject liquids out of the nozzle
- determining the effect of surfactants on the printing behaviour of the micronozzles

Figure 1.1 depicts the general design of this cartridge.

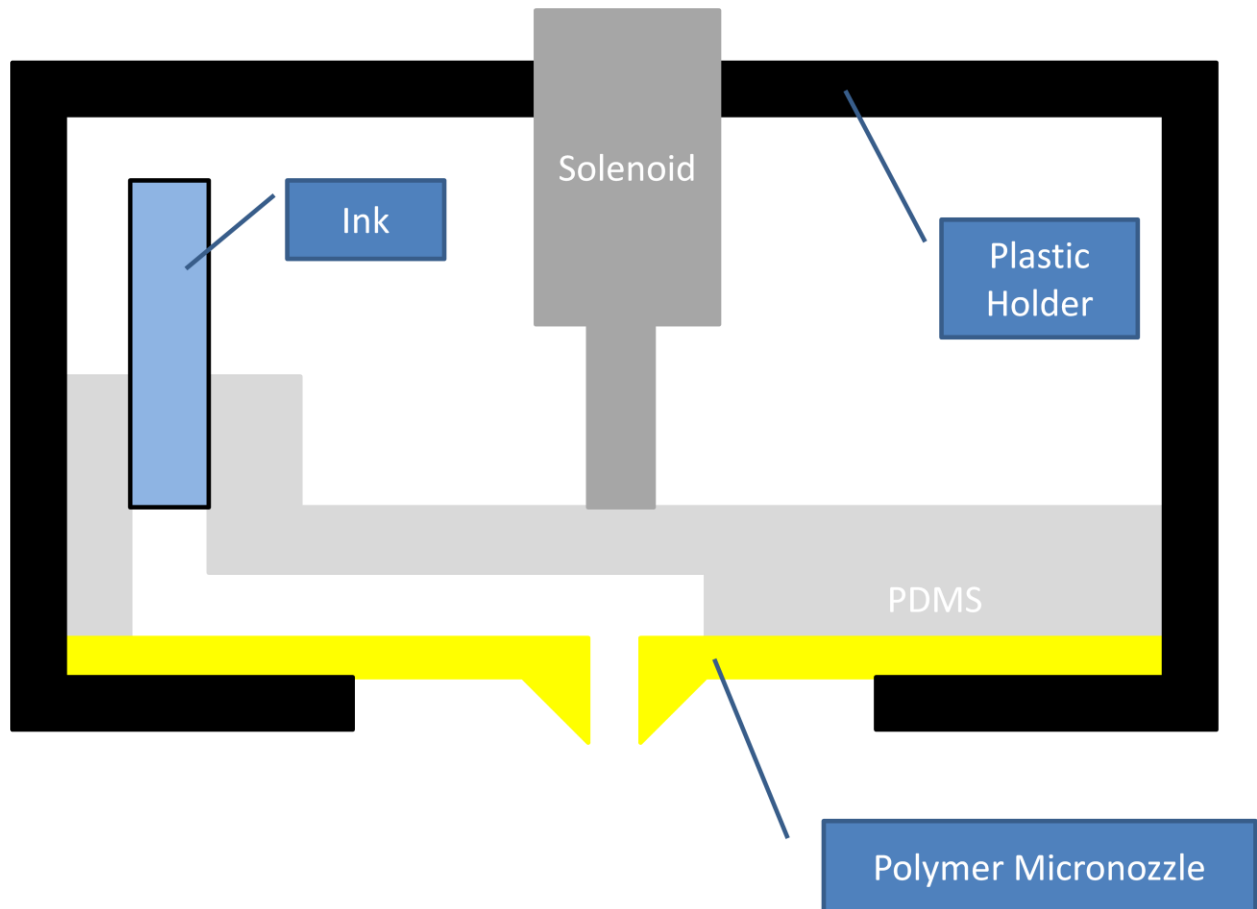


Figure 1.1 Single nozzle disposable cartridge actuated by solenoids

Different experiments are designed to compare the performance of the newly developed microfluidic polymer nozzle with the previous commercial and non-commercial nozzles. The experiments include setting the internal pressure of the system, tuning the input voltage (amplitude, duty cycle and frequency) and characterising the effect of different nozzle sizes on

the performance of the system. In addition the effect of using surfactants to lower the surface tension of the system is tested.

1.3. Thesis Format

The remainder of this thesis continues with four chapters. **Chapter 2** reviews the published literature in microdrop generation especially using the inkjet printing method. Different fabrication techniques for fabrication of microneedles are reviewed and compared. A brief review on microfluidic devices is described. Applications of inkjet printing in tissue engineering and drug screening are then described. In **Chapter 3** several design considerations of the device are expressed. In **Chapter 4**, fabrication of the disposable inkjet print head is explained in detail. First the fabrication of microfluidic devices is explained. Then the fabrication of microneedles is described. Following are the actuators and their performance. Then the experimental setup for this project is described. **Chapter 5** includes the results and discussions of the different experiments performed on the fabricated micronozzle chips and their performance. Finally the thesis is concluded in **Chapter 6** with the general discussion of the results and the recommendations for future work. This thesis is written in the traditional format.

2. Literature Review

2.1. Microdrop Generation and Applications

2.1.1. Inkjet-Printing

Different printing technologies have been developed to date, such as screen printing, gravure printing, offset, flexography and inkjet printing [31]. Among all of these technologies inkjet printing has the advantage of having respectively high resolutions [32]. Inkjet printing has seen applications in a variety of new and interesting areas such as inkjet printing of functional materials. In these applications, usually an inkjet printer is modified to deposit a variety of inks with specific electrical, structural, optical, chemical and biological properties [33]. Inkjet printing is one of the most widespread techniques for commercial document printing such as printing high resolution posters. The main drawback of this technique with respect to other printing technologies available such as screen printing is that it is not a high throughput fabrication technique ($0.01-0.05 \frac{m^2}{s^{-1}}$).

Other than graphic arts, applications of inkjet printing are printing thin film transistors [34], light-emitting devices [35], solar cells [36], memory and magnetic applications [37], contacts and conductive structures [38], sensors and detectors [39], and biological and pharmaceutical applications. Inkjet-printing has also been successful in fabrication of LCDs [40] and RFIDs [41]. Inkjet printing systems allow deposition of bioactive fluids and integration of electronic, photonic, sensing and structural features, which is not generally possible with regular microelectromechanical system (MEMS) fabrication methods. The main reason is that the biomaterials especially the materials used in tissue engineering are not able to survive the optical and chemical exposure of the lithographic process.

Inkjet printing as a fabrication method has several advantages. The most important advantage is that it is a non-contact additive approach, which does not require a mask and photolithography process. In addition with respect to other fabrication technologies, inkjet printing is considered to be low cost [42] as there is the opportunity to minimize waste and also reduce the environmental impacts. The inks and materials which are used in inkjet printing are either solutions or suspensions. One of the advantages of inkjet printing in various applications is that it provides the ability of depositing microlitre-sized droplets in precise locations using computer aided design and it permits operation at low temperatures that are not harmful to most of biological and functional materials.

One important outcome of inkjet printing is the behaviour of the droplets after being ejected. In [43] the author has demonstrated inks with higher viscosity create smaller droplets with a longer liquid column, droplet size, and the ink material, surface energy of the substrate. The diameter of the nozzle set a limit on how thin a line can be printed. The desirable inks for inkjet printing are the ones that have lower viscosity, high surface tension and in addition to these properties the inks are Newtonian.

Two different modes for inkjet printing exist; continuous mode and drop-on-demand mode [44]. In continuous inkjet printing pressure forces the ink out of the inkjet-printer nozzle. An electromechanical device for example a piezoelectric transducer can be used to create the necessary pressure changes in the ink. In drop-on-demand inkjet printing each droplet is created with each actuator pulse. The actuator is usually made from piezoelectric materials or thin resistors. The resistors will change the temperature to vaporize the nearby liquid. Drop-on-demand systems usually have a simpler design but they consume more energy. Piezoelectric drop-on-demand inkjet-printing technology has several advantages over continuous and heat

mode. First of all it does not heat the fluid to cause any damage to the material. Also it is easier to achieve small drop diameters and use inks with lower viscosities with piezoelectric drop on demand. In thermal DOD inkjet printing droplets are generated by heating the wall of the chamber causing formation of vapour bubbles and ejection of droplets through the nozzle orifice. In piezoelectric DOD inkjet printing a voltage pulse is applied to a piezoelectric actuator causing a pressure wave in the ink chamber that results in formation of droplets at the nozzle. Usually it is the change in the volume of the corresponding ink chamber which causes a fixed volume of ink to be ejected out of the nozzle. For example for a chamber which is actuated using a piezoelectric actuator applying a voltage contracts the chamber causing a shockwave in the liquid [44].

In the following section a review of different inkjet printing nozzles and printer systems is provided. The focus is on different nozzles which have been used in different applications of inkjet printing for functional materials.

2.1.2. Inkjet Printers in Different Applications

A wide variety of commercial inkjet printer heads with different nozzle types have been fabricated. For the purpose of inkjet printing of functional materials, cells and biomaterials inkjet nozzles are necessary that are compatible with polymers, biomaterials, bioinks and conductive inks.

Commercially available printers have been modified for high throughput cell patterning. In [45] the authors have modified an off the shelf desktop printer (Canon BJC 2100) and printed cells. The nozzle size of this printer is 20 μ m-30 μ m and the droplet size is 10-20 pL. Also in this publication it has been mentioned that available printers with high resolutions of about 1 μ m are

able to print cells with a resolution of about 10-20 μ m. The downside of this technique is that the Canon printer is thermally actuated which will heat the cells and biomaterials up to 300°C.

Different types of inkjet printer heads were used for preparation of polymer microarrays [46]. For printing polymeric compounds an important requirement is that the inkjet nozzle segments that are in contact with the polymer should be completely resistant to organic solvents. Numerous commercially available desktop inkjet printers have been used for the purpose of printing polymers. Epson Stylus color 670 and 480 SXU [47][48] was used for printing polymeric passive RC filters. Binders for ceramic components were printed using HP Deskjet 400 [49]. Nanosized silver colloids have been printed using the ordinary commercial Epson R210 printer which consists of a piezoelectric head and the resolution is 5760 dpi, the droplet size is in the order of 3 pL [50]. The advantage of desktop inkjet printers for depositing functional materials is that they are commercially available at a low cost.

For inkjet-printed light emitting polymer displays [35] a high frequency driven piezoelectric drop-on-demand inkjet printing head has been used. This printer head comprises a multilayered PZT (Lead zirconium Titanate) ceramic. The several layers of PZT produce a stronger force to push the liquid outside of the nozzles comparing to just one layer.

Another option for printing polymeric devices is the use of inkjet dispensers. Different manufacturers have fabricated this kind of dispensers for scientific purposes. Microdrop, Microfab, Jetlab, GeSim, Packard have introduced various devices for this purpose. These commercial inkjet dispensers have been used to fabricate polymer microarrays [46].

Authors in [51] have introduced an electrostatically induced inkjet head system for printing conductive inks. The suggested system comprises two electrodes, a microsyringe pump, a fluid

chamber and a glass capillary. The electrostatic force between the two electrode plates results in formation of a droplet. This happens when the surface tension of the ink meniscus is smaller than the electrostatic force which is applied between the two plates. In another publication the authors propose a multinozzle printing system [52]. The authors have proposed a system that is able to print different materials using a set of different nozzles. Two different piezoelectric glass nozzle and a solenoid actuated microvalve print head are used in this study. The solenoid print head from Lee Company opens the orifice of the nozzle with the help of the solenoid actuation. Then the backpressure will force the liquid out of the nozzle. A glucose biosensor [53] has been printed using an array of nozzles which are thermally actuated. The authors have modified a commercial printer by Olivetti Tegnost (Ivrea, Italy). The head consists of 208 nozzles. The resulting droplet size is 10-12 pL. IBM Colorjet was used to print PZT pillars [54].

In addition to the non-biological applications that were described in this section, some of the biological applications of inkjet printing are described in the next section. In the mentioned applications, either a desktop piezoelectric inkjet or thermal inkjet printer and electrostatic actuation have been used to print cells and biomaterials. These techniques have several drawbacks for printing cells and biomaterials. High temperatures and actuation techniques are used which will be harmful to these materials.

2.2. Biological Applications of Inkjet Printing

The word “tissue engineering” has been introduced in 1987 and the word “organ printing” in 1999. The organ printing technology prints successive layer of cells and biomaterials on top of the previous layer that has been printed, in this way an organ is formed. The basic assumption for printing organs and tissues is that the cells can be suspended in a solution and then formed into a tissue [55]. One of the challenges encountered in tissue engineering is growing cells and

scaffolds into the desired architecture to keep the cells' biological function and also most of the biomaterials such as DNA and proteins are not easy to handle.

Different inkjet printing techniques have been used to print three dimensional tissues. In [56] the authors have used thermal drop-on-demand inkjet printing to print 3-D organs. They have modified a commercially available printer Hewelett Packard (HP) 550C and an HP5162a ink cartridge in order to print the required materials. This process needs time consuming cleaning of inkjet printer cartridge using ethanol, each time the printer is used. Hewelett Packard 2225C and 7970A commercial inkjet printers were used to print human cells [57]. In another research high throughput inkjet printing has been used to deposit collagen [58] using the modified off-the-shelf Canon Bubble printer cartridge.

A high resolution inkjet printing technology was developed using an ordinary inkjet printer to print DNA arrays [59]. Control of neural stem cells has been performed using a commercial Canon inkjet printer to print active macromolecules on hydrogel substrate [60].

In one study [61] piezoelectric drop-on-demand inkjet printing was used to print suspensions of human fibroblast cells and the authors have demonstrated that the amplitude of input voltage pulse to the piezoelectric actuator does not have a substantial effect on the cell survivability.

2.3.Other Methods

Other than the common inkjet printing methods that were described in the previous section, several other methods were introduced to create micro-droplets. XEROX PARC has introduced a new technology to create microdrops. In this technology focused acoustic beams are used to eject droplets [62]. The advantage of using ultrasonic waves to create and eject droplets is that this technique prevents particle jamming. Ultrasonic actuation gives the user the ability to change the

droplet size without changing the diameter of the output nozzle. The major disadvantage of this technique is that the energy needed to focus the beams to create the droplet is high. This high amount of energy may damage the inks that are being used especially if they contain any kind of cells and biomaterials.

A novel technology has been introduced to create microdrops from microarrays [63]. In this method the required amount of liquid is drawn into the ink chamber. Droplets are created and pushed out of the output hole using different actuation methods. In the first actuation method a piezoelectric actuator is used to push a membrane on a chamber filled with gas. Instead of gas chamber a deformable elastomer can be used. In another method of actuation, inertia is used to push the droplets out of the nozzle. In this method the whole nozzle is moved with a high velocity to force the liquid out of the nozzle as opposed to other methods that the nozzle is fixed and the liquid is pushed using different actuation methods.

2.4.Microneedles as Nozzle-Shaped Structures

In this section, a literature review is performed to choose a suitable fabrication technique to create nozzle-shaped structures. The micronozzles should be fabricated in small diameters and also the fabrication technique needs to be cost effective. One of the interesting recent developments in drug delivery systems has been the fabrication of microneedles which provides a painless and safer approach of injecting drugs into human body as a way of transdermal drug delivery [25]. The idea in this project is to use the same fabrication technique that is used to fabricate these microneedles, to fabricate nozzle-shaped structures.

There are two types of microneedles hollow and non-hollow [64]. One purpose of fabricating non-hollow microneedles is to increase skin permeability. Since we are going to take advantage

of microneedles as a micronozzle for our printer, we will focus on different approaches to fabricate hollow microneedles. Hollow microneedles have been fabricated using several different materials such as silicon and silicon dioxide, metals, glass and a large variety of polymers. According to literature they have been fabricated in a wide range of lengths varying from 30 μ m to 1 mm, and diameters ranging from 3 μ m to 100 μ m. In general two types of microneedle designs are created so far. The first design is in-plane microneedles. In this type of design the needles are parallel to the surface. The second type is out of plane microneedles. These microneedles are perpendicular to the fabrication surface [65] .

Polymer microneedles have been fabricated and characterized using various methods. Polymer microneedles are considered to be cost effective with respect to other types of microneedles. In [66] the authors have fabricated an array of vertically spaced microneedles out of PDMS. The process consists of one step of UV exposure and two steps of PDMS casting. Two steps of deep X-ray lithography of Polymethylmethacrylate (PMMA) have been used [67] to fabricate hollow microneedles.

In [68] the authors have fabricated microneedles out of chromium masking material deposited on a silicon wafer. The process consists of a deep reactive ion etching process step. The diameter of fabricated microneedles is 80 μ m.

Hollow glass microneedles have been have been fabricated in several different researches [69][70][71]. The process is a single mask process. A silicon dioxide layer is exposed to UV to create holes and then etched by DRIE. Several etching steps are used in this process. These glass microneedles are 5 μ m in diameter and 30 μ m in height. Hollow microneedles using laser

micromachining have been fabricated [72]; the molds are made out of polyamide, polyethylene terephthalate and titanium.

In [73] the authors have fabricated a single glass microneedle. Thos microneedle is $60\mu\text{m}$ in diameter and range between $500\mu\text{m}$ to $800\mu\text{m}$ in height. Stoeber and Liepmann in [74] have fabricated microneedles with wide base and pointed tips. The authors have used both isotropic and anisotropic etching. The diameter of microneedles in this case is $40\mu\text{m}$ and their height is $200\mu\text{m}$. Stoeber and Liepmann [75] have developed the fabrication process for single crystal microneedles by using deep reactive ion etching (DRIE) of silicon and a step of isotropic etching.

In [76] the authors have fabricated short microneedles of $9\mu\text{m}$ in length that are $50\mu\text{m}$ in width, using a combination of bulk and surface micromachining. LPCVD and different etching steps are performed in order to produce these microneedles.

In one publication [26] a process of DRIE, anisotropic wet etching and conformal thin film deposition is used to fabricate hollow silicon microneedles with maximum width of $250\mu\text{m}$ and height between $150\mu\text{m}$ and $350\mu\text{m}$.

An array of hollow microneedles [77] has been developed using backside exposure of SU-8 and electrodeposition of metals. The microneedles width ranges from $33.6\mu\text{m}$ to $101\mu\text{m}$ and the thickness is either $200\mu\text{m}$ or $400\mu\text{m}$.

An array of ultra sharp microneedles with a tip radius smaller than 100nm has been fabricated [78]. The process includes two steps of mask lithography of silicon. A combination of DRIE and isotropic etching (RIE) has been used in this fabrication. The height of the resulting array of

microneedles is 400 μ m. Another group of researchers [79] were able to produce long microneedles nearly 400 μ m in length with DRIE and anisotropic etching.

Recently a simple process for fabrication of hollow and out of plane polymer microneedles has been developed. The process includes solvent casting and requires only one step of lithography. The molds that result from this technique are usually reusable which saves a lot of time and reduces the fabrication expenses [28]. According to our intended application needs this process is the most convenient process to fabricate polymer micronozzles for our device.

2.5. Microfluidics

Microfluidic devices are used to guide small amounts of liquids in submillimetre-sized channels. The current applications of microfluidics are in the fields of chemical synthesis, biology and even information technology [80]. For analysis applications, microfluidics offer advantages of using small amounts of material. Microfluidic devices fabrication is easy and low cost. High throughput screening in drug development [81] and cell sorting [82] are among the many applications of microfluidic devices [83]. Cell biology [84] and chemical synthesis [85] are among other emerging microfluidic applications. The science of microfluidics is used in tissue engineering applications to mix and route cells [86].

An important factor in fabrication of microfluidic devices is to choose the material with the desired surface chemistry properties. The most popular material in fabrication of microfluidic devices is PDMS. PDMS has the ability to replicate features down to nanoscale [87]. PDMS is the most widely used silicon-based organic polymer and belongs to a group of polymeric compounds known as silicones. Silicones are polymers that include silicon together with carbon, hydrogen, oxygen and sometimes other chemical elements [88]. PDMS is hydrophobic by

nature; however after it is exposed to oxygen plasma the surface will temporarily change to being hydrophilic so the liquids easily wet the surface of the microchannels which is desirable. Other options for elastomers used in fabrication of microfluidic devices are polycarbonate [89] and polyolefin [90]. Other than elastomers, other options that exist for fabrication of microfluidic devices are silicon [91] and glass [92].

Previously, microfluidic devices have been fabricated from glass and silicon using the process of photolithography. For analysis applications, silicon as the material which is used in fabrication of microfluidic devices has the disadvantage of opaqueness to light. Being opaque will prevent using the conventional detection methods with light. In addition to this problem, silicon is considered to be an expensive option. On the other hand, glass and silicon are thermally stable and this characteristic makes them suitable for special microfluidic systems that need to be thermally stable [93].

In addition to fabrication of simple microfluidic channels, other components are necessary on a microfluidic chip. These components are used for analysis, mixing and routing of liquids on a small microfluidic chip. In [94] and [95] microfluidic valves fabrication has been explained. In [96], [97] and [98] different microfluidic mixers are presented. Microfluidic pumps are another important component of microfluidic devices [99].

Soft lithography techniques, replica molding and rapid prototyping are a common approach to fabricate microfluidic devices [100]. In rapid prototyping a CAD design is transferred to a mask, which using UV exposure will be transferred to a negative or positive photoresist on a silicon wafer in order to make the master mold. PDMS will be spin coated or cast (replica molding) on top of the master mold to make the desired microfluidic components such as channels, pumps

and valves. In the next step, the microfluidic chip is sealed with taking advantage of the same material or any other suitable material. Using oxygen (air) plasma on both surfaces is one option for sealing the device. The process of changing the surface properties of PDMS is irreversible. Exposing the PDMS surface to plasma creates polar groups, when they are in contact with the other surface the reaction of the two surfaces will create strong covalent bonds. PDMS can be bonded to a variety of surfaces such as PDMS, glass, silicon, silicon dioxide, quartz, silicon nitride, polyethylene, polystyrene and glassy carbon. However PDMS does not bond to a variety of surfaces such as some polymers [101].

Several challenges exist in the fabrication of microfluidic devices. The most important of all is the complex microfabrication procedure needed to make a microfluidic system which includes all the different components (mixers, pumps, valves) to perform different functions on one chip. The required lithography masks used for fabrication of these microfluidic devices can require several layers. In this case the alignment of each layer will be an important issue. Also in microscale the effect of surface forces is higher which will introduce new problems to the system [102]. One difference between handling macroscopic scales of the liquids with microscopic scale is that inertia has a larger effect on the liquid flow in macroscopic fluids [80].

2.6. Actuators

Different actuators are used in inkjet printing. Each of them has different properties that make it suited for different applications with different limitations. The most common actuation method for printing functional devices using inkjet printing for example printing electronic materials is drop on demand inkjet printing.

Push type cylindrical solenoids have been used previously for opening and closing a valve [103]. By applying a voltage to the actuator the plunger of the solenoid is pushed down which will completely close the channel. PDMS being elastic, after the voltage pulse goes back to zero, it will push the plunger back up.

Another technique similar to thermal inkjet printing has been developed [104] which uses an electrical discharge to create a pressure bubble inside the ink chamber. High voltage electrodes are attached between the fluid and the nozzle. High current density will pass near the output hole and creates a vapour bubble. The other disadvantage is that this technique may harm the living cells and biomaterials that are used in the ink.

Using electric fields to create droplets is another method used in inkjet printing [105]. First a meniscus is created at the output of the device. The electric field will elongate the meniscus. A high enough electric field will overcome the surface tension of the liquid and break it into droplets. The diameter of the created droplets in this technique is significantly smaller than the diameter of the output hole. The inks that are used in this technique need to have a large dielectric constant and also low surface tension. Other inkjet technologies have been developed that are not as common as the aforementioned technologies; Electrorheological inkjet printing and liquid ink fault tolerant processes [106].

A novel technology called TopSpot Vario that is used to create droplets out of a micromachined microarray has been developed in [107]. The authors have used a piston and a PDMS elastic membrane to eject the droplets out of the array. A piezoactuator actuates the piston which will deform the elastomer and finally force the liquid out of the nozzle.

3. Design Considerations

The objective of this project is to build a disposable micro-nozzle by merging the different technologies of inkjet-printing and modular microfluidics together. In the initial experiments, design and fabrication of polymeric micro-nozzles with different orifices and arrangements has been performed. Choosing the right polymer for fabrication of this device is essential. In the following step of this project, design and development of different microfluidic chips meeting the needs of our intended applications are performed. The position of reservoirs in the microfluidic chip and the layer containing the micronozzles should align. After developing the micro-nozzles and the microfluidic chips, various bonding techniques have been tested. As the next step, the necessary actuating system for the device has been designed. It is important that the actuator is not attached to the polymer micronozzles. In this way, it will not add to the cost of fabrication of the device. Development of a holder system for the nozzle which will connect to a syringe pump system is the final step before testing the hardware of this device. With each voltage pulse the volume change above each micronozzle forces the inks out of the ink chamber and into the orifice of each nozzle. In addition to these general design considerations, in this chapter several design parameters regarding the micronozzle are described.

At the microscale, surface tension and capillary forces have an increased effect compared to macroscopic scale. At the macroscopic scale, pressure and gravity will have the larger effect. Surface tension is a measure of the cohesive energy present at an interface. Surface tension has the dimension of force per unit length and surface energy has the dimension of energy per unit area. Surface tension is defined as

$$\sigma = \frac{U}{2R^2} \quad (3-1)$$

where R is the characteristic molecular dimension and U is the total cohesive energy. As an example, the surface tension of water in contact with air is $72.8 \frac{N}{m}$.

In the experiments section Pluronic F68 is going to be used as a surfactant to reduce the surface tension of the ink (water and food color). The characteristic of surfactants is that they consist of amphiphilic molecules. Each amphiphilic molecule consists of a hydrophilic head and hydrophobic tail. A surfactant will reduce the surface tension of a liquid by gathering on the liquids surface because of its amphiphilic properties [102].

According to the nature of a solid surface, how liquids spread is different. Partial wetting corresponds to the liquid creating a droplet on the surface. The other case is total wetting, which happens when the liquid spreads in a thin layer on the surface of a solid. In total wetting the liquid will decrease the energy of the system.

For determining whether the liquid spreads partially or totally, the spreading parameter S is used. The spreading parameter is defined as

$$S = \gamma_{SG} - (\gamma_{SL} + \gamma_{LG}) \quad (3-2)$$

Where γ_{SG} is the surface energy of the dry solid surface, and $\gamma_{SL} + \gamma_{LG}$ is the surface energy of the wetted surface. γ_{SL} is the surface energy at the solid-liquid interface and γ_{LG} is the surface energy at the liquid-gas interface. A positive number for S corresponds to total wetting of the surface while a negative number corresponds to partial wetting.

In partial wetting two different situations occur. In the first case the contact angle with the substrate is more than 90° which is called a hydrophobic contact. In the second case the contact angle is less than 90° which is called a hydrophilic contact. According to [108] plasma treated

SU-8 has a high surface energy of about 75 mN/m. In the case of plasma treated SU-8 surface with water γ is nearly 7mN/m. This positive number suggests that water will totally wet the plasma-treated SU-8 surface.

The shape of the droplet results from the competition of gravity and surface tension. Surface tension forces the droplet to form a sphere while gravity is trying to lengthen the droplet [102].

To create monodisperse droplets using the drop-on-demand technique, certain conditions apply. There are several tuneable factors in order to create monodisperse droplets with each voltage pulse. The amplitude of the input pulse of the actuator is one important factor. A high positive voltage will force the liquid out of the nozzle. A higher voltage will create a stronger magnetic field inside the solenoid coils. The high magnetic field will create a larger force on the iron plunger of the solenoid.

The other important factor is the shape of the input pulse [109]. The shape of the pulse is critical to the production of drop-on-demand droplets. The most important factor in the input pulse is the pulse width. In addition to creation of a single droplet by tuning the pulse width, shorter pulse widths create smaller drops. Also higher amplitudes are required when the pulse width is shorter. For certain ranges of pulse width, there is no specific amplitude that will create micro-droplets. To find the right pulse width for the input voltage of the actuator, the lowest possible pulse width should be chosen and then the voltage is tuned. If there was no voltage that would work in this specific pulse width, the pulse width should be increased and process needs to be repeated until the right amplitude and pulse width is found.

The rheological characteristics of the fluid and even the different levels of fluid inside the nozzle require different settings. There is a range of fluid characteristics that make that fluid and

ejectable ink. Regarding internal pressure of the system, negative pressure is needed to eject the drop. The reason that a negative internal pressure is preferred is that a positive internal pressure of the fluid creates a meniscus build-up outside the nozzle. The liquid jet should overcome this liquid build up to push a droplet out of the nozzle. Since water has high surface tension, positive internal pressure is needed. Water does not flow out of the nozzle and does not break its meniscus.

In the next chapter fabrication of the all-polymer micronozzle is presented. In addition to the fabrication process, the micronozzles are connected to an experimental setup and different characteristics of the device are tested.

4. Experiments

4.1.Fabrication of the Disposable Inkjet Print Head

4.1.1. Fabrication of Polymer Microneozzles

The first step toward fabricating the polymer microneozzles is to choose the proper material that will meet the application needs. One of the most important requirements in choosing the right material is that the microneozzle surface should not have any reactions with the fluids that are passing through the nozzle. Another important characteristic of these microneozzles is their strength under the pressure of the actuator. The microneozzles should withstand the pressure from the actuator without bending or breaking. Any cracks in the microneozzle chip surface will cause liquid leakage. In addition to these characteristics, a flat surface on the backside of the nozzle chip is required. A curvature in the microneozzle chip will cause difficulties for the alignment of the top PDMS layer. In addition to the alignment problems, the curvature will cause difficulties when the device is going to be connected to the experiment setup and the actuators that are designed for a flat microneozzle chip surface. In the literature review section different fabrication methods for microneedles from various methods and materials have been reviewed. A new solvent casting process developed by Mansoor and Stoeber for fabrication of hollow out of plane polymer microneedles is used for fabrication of microneozzles [75]. This process requires just one step of photolithography which makes it more convenient with respect to other methods. Polyimide and SU-8 are two options that have been considered as mentioned for the microneozzles used in this work. Both polyimide and SU-8 possess their own advantages and disadvantages. At first polyimide was tested as the structural material for the fabrication of microneozzles. Polyimide microneedles have the advantage of high strength under pressures and

deformations. Figure 4.1 depicts the fabrication process for polyimide microneedles. The fabrication is described in detail in Appendix A.

According to [110] plasma bonding of both PDMS and polyimide will create a weak bond between the two materials in a way that the polyimide layer can be easily removed from the PDMS layer by applying a small pulling force. Based on the experiments that I performed 40 seconds of oxygen plasma treatment at 26.9W on both surfaces followed by two hours of post-bake in 70°C convection oven will bond the two surfaces together. A small amount of water was pushed through the microchannels using a syringe. The strength of the bond between polyimide and PDMS using this technique is too weak to withstand the liquid pressure in the channels. The bonding of polyimide to PDMS in all of the tests that were performed was unsuccessful, indicated that using an adhesion layer between PDMS and polyimide is necessary to bond the microfluidic device to the microneedles.

VM651 and VM652 are adhesion promoters that are used to improve the adhesion of polyimide with different materials such as silicon dioxide and silicon nitride. VM651 was diluted in DI water (0.1% solution) and was used to bond polyimide to PDMS. First the surfaces of the PDMS and polyimide were cleaned with acetone and dried with a nitrogen gun. Since both of these surfaces are hydrophobic first a 40 second oxygen plasma treatment is required on both surfaces. The next step is to deposit a thin layer of the diluted VM651 on either the PDMS or the polyimide layer. After the treatment 10 μ L of the solution was deposited onto the polyimide surface and the microfluidic thin layer was aligned with the nozzle and bonded to it. The sample was left for 2 hours in 70°C convection to allow for the bond to form. In other tests the sample was left out in room temperature for 16 hours to prevent the curvature caused in polyimide from heat. In other experiments instead of depositing the diluted VM651 on the polyimide

microneedles it was deposited on the PDMS layer .In most of the cases either the channel or the nozzle was clogged with the VM651 material which prevented the liquid from going through the nozzle. In some cases non-uniformities caused by the evaporation of the VM651 allowed for leaks in the layer between the PDMS and polyimide.

In addition to several bonding problems, another important problem was encountered during the process of fabrication and bonding the polyimide to the microfluidic device. For adding the required stiffness to polyimide, different concentrations of clay were added to the polyimide and tested. Polyimide with 2%, 5% and 10% clay was tested. It was found that increasing the percentage of clay in polyimide also increased the curvature in the micronozzle. It was noticed there may be a trade-off between the stiffness of the polyimide micronozzle and the unwanted curvature of the device; however the details were not explored. The unwanted curvature in the micronozzle resulted in problems during alignment of the device and also connecting the micronozzle to the experiment set-up and the actuator. After several experiments with polyimide microneedles, the second option, namely SU-8, was used as the alternative structural material.

SU-8 micronozzles have the disadvantage of being fragile. Handling the SU-8 microneedles especially under the actuation forces causes the microneedles to crack or break. SU-8 micronozzles chips have less curvature with respect to polyimide micronozzles and also do not require any adhesives to bond to the PDMS microchannels. 30 seconds of oxygen plasma treatment, followed by 10 minutes of baking on a hotplate at 120°C was sufficient to create a strong bond between these two surfaces. An additional step exists in choosing SU-8 micronozzles over polyimide as a result of the additional exposure to UV light that is required in fabricating the SU-8 micronozzles.

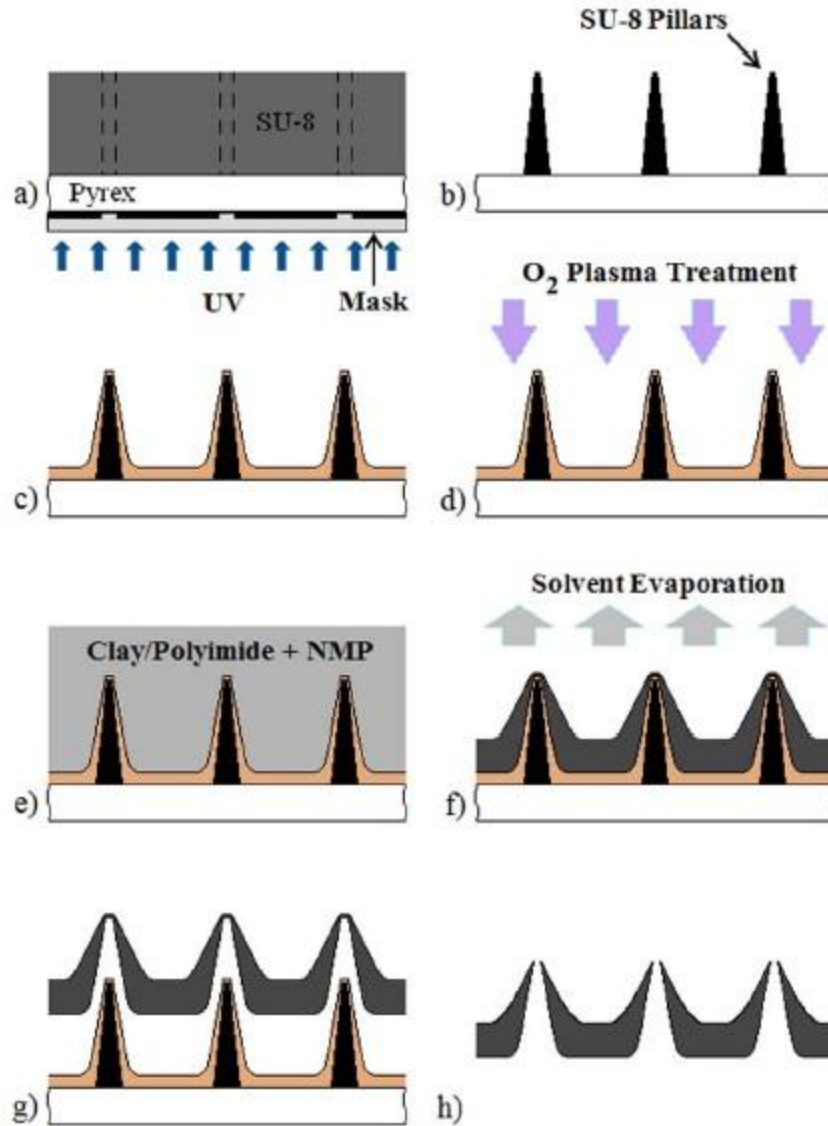


Figure 4.1 Process flow for fabrication of polyimide microneedles, copied with permission from Iman Mansoor [28] a) SU-8 spin casting and photolithography b) Developing the photoresist c) PDMS deposition d) PDMS plasma treatment e) Polyimide/Clay deposition f) evaporation g) releasing the microneedles h) the fabricated microneedles

The process of fabricating SU-8 microneedles is similar to that for fabricating polyimide microneedles. The first step in fabricating microneedles is to make a mold. For purpose of testing these devices as a nozzle for inkjet printer, only one needle pillar is sufficient. The masks are attached to Appendix C. Also Appendix A describes the detailed mold fabrication for these

polymer micronozzles. The next step of the fabrication is to deposit a layer of sacrificial material. The material used in this step is dependent on the polymer that is going to be used for the structural material of the needles. After the evaporation of the solvent, which was used as a sacrificial layer on the mold, the second layer of the polymer which corresponds to the microneedle structural material will be deposited.

SU-8 2075 which is a negative photoresist by Michrochem was used as the material for the mold. The first step is to spin coat a 350 μ m layer of SU-8 2150 or 2075 on a 300 μ m (or 500 μ m) Pyrex glass wafer. The resulting wafer is soft baked at 65°C for 10 minutes and 95°C for 2 hours (more details in Appendix A). In this technique 2484 $\frac{mJ}{cm^2}$ exposure of UV on the sample has been used. The exposure should be performed with 3 minute steps with a 10s interval in between. A base glass substrate is used to make a gap between the PR and the mask which will cause a light diffraction. This set-up for exposure will result in cone-shaped pillars (sharp at the tip). Another soft bake step is needed after exposure. In this case the samples are kept on a hot plate for 5 minutes at 65°C and 30 minutes at 95°C. The next step is to develop the SU-8 photoresist by immersing it in SU-8 developer for 45 minutes. Then the sample is washed with isopropanol for 1 minute.

After the mold is ready, it should be coated with a thin layer of parylene C. This layer is added to help the needle pillar adhere to the substrate. The sacrificial layer that was used was 4% polyvinyl alcohol (PVA) in water. PVA was deposited on the surface of the mold. Then the solvent of PVA should evaporate on a hot plate for 2 hours at 70°C.

After the sacrificial layer is ready the structural layer should be deposited. A mixture of SU-8: cyclopentanone at 1:3 by volume is deposited. After depositing this mixture the evaporation

process is performed at 45°C on a hotplate for 1 hour. After this step the whole wafer is exposed to $1000 \frac{mJ}{cm^2}$ of UV light and in this way the structural layer of the microneedles is cross-linked.

An etching step is required to remove the remainder of the SU-8 from the top of the needles.

The etching is performed by using the oxygen plasma for 30 seconds at the power of 100 W and pressure of 200mTorr. For removing the SU-8 structural layer from the mold we need to dissolve the PVA in water. At first a solution of water was used at 65°C to remove the sacrificial layer. The problem with heated water is that it will cause the whole SU-8 structure to bend and this is against getting a uniform chip that will be bonded to PDMS. Therefore the samples were immersed in water at room temperature overnight for 16 hours. In order for water to be able to access the PVA layer a small scratch in the corner of the mold should be made on the SU-8 structural layer.

4.1.2. Fabrication of the Microfluidic Devices

The first step in fabricating the microfluidic chips that we need for routing liquids in our disposable nozzle is to design the related masks. A microchannel with one input and one output was used. This design is consisting of one input hole for liquid input from the printers pumping system and ink chamber. The output hole will be located on top of the micronozzle when the two layers are bonded together. Different microfluidic devices can be designed for different applications. In this experiment the simplest designs were used in order to focus on the coupling to the nozzle and to study ejection mechanism. The next step after designing the mask is to clean the mask with isopropanol and acetone thoroughly and then spin coat SU-8 2750 after the prebake, the mold should be exposed to UV light. Then the mold is post baked. Finally we

develop the photoresist in the SU-8 developer, after these steps the mold is ready. Appendix B describes the detailed mold fabrication for the microfluidic device.

SU-8 which is a photosensitive epoxy resin that in my project has been used at first as a mold for making the microchannels and second as the structural material for microneedles. SU-8 was chosen since we can get sidewalls with good aspect ratio in the other hand PDMS is chosen for microfluidic channels because it is easy for prototyping and it has good elastomeric properties.

The next step is to mix PDMS with the ratio of 10:1 of PDMS (Sylgrad 184) and the hardener in the automatic PDMS mixer. When completely mixed, PDMS is poured on top of the mold in a thin layer and then degassed in vacuum. Since we are looking for a thin layer of PDMS matching the thickness range of our microneedles (100 μ m-200 μ m) the mold should be spin coated for a long time and high speed. All the details are listed in Appendices C. Then the mold is cured for 2 hours in a convection oven which is set at 70° C. Then this thin layer of PDMS should be released. This step is sensitive, since the layer of PDMS that has been deposited is thin (200 μ m) and adheres to the surface of the silicon wafer. Releasing the thin layer should be started by very carefully lifting up the sides of the PDMS from the silicon wafer and moving to the center of the mold. In this way without tearing the thin layer of PDMS apart the whole layer will be lifted off the surface. Then the PDMS thin layer is carefully placed on a piece of aluminum foil. Then it is cut into small chips with one channel located on each of the parts.

Since the PDMS layer is thin (less than 300 μ m), a piece of a thicker layer is needed to support the input of the liquid from the syringe pump. For this purpose a small cubic block (4mm each side) of PDMS is bonded to the thin PDMS layer using oxygen plasma. First the top surface of each PDMS layer is cleaned by 45 seconds of plasma and then both surfaces are bonded together

by putting the chip in the oven for one hour. And using a punch a 500 μ m wide hole is created in the PDMS cube.

After the PDMS chip is ready, the bottom surface of the chip and also the bottom surface of the SU-8 micronozzles are cleaned by acetone and isopropanol. When completely clean, the two surfaces are dried with a nitrogen gun. The two surfaces are plasma treated in oxygen plasma for 30 seconds. Then the chip is placed on the hotplate on 120°C for 10 minutes to completely bond the SU-8 and PDMS together.

Figure 4.2 shows a fabricated micronozzle which is then bonded to the microfluidic device. In the left side the SU-8 micronozzle is shown. In the right side of this figure the final fabricated polymer micronozzle is depicted.

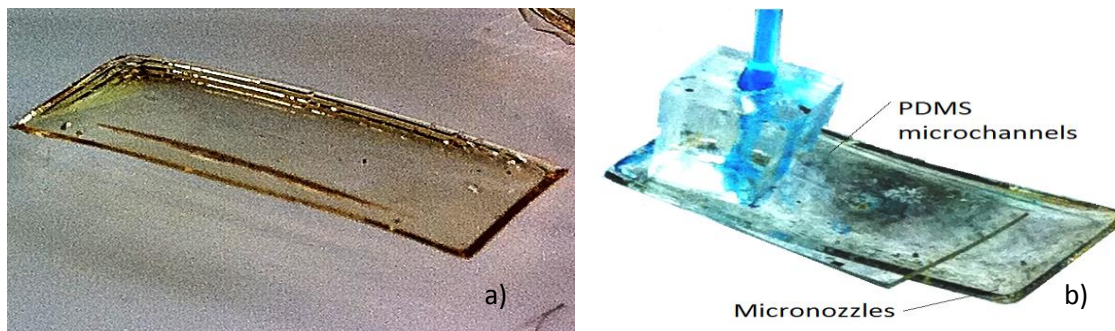


Figure 4.2 a) SU-8 micronozzle chip b) fabricated all-polymer micronozzle cartridge

4.1.3. Actuators

One important point that should be considered for choosing the right actuation method is that the actuation system should not change any physical or chemical characteristics of the ink. Thermal actuation is not a good candidate for droplet ejection, since it heats a region inside the ink and may cause damage to any living cells that are suspended in the ink.

For our intended research objectives and due to the characteristics of our device several actuators can be used. A combination of a magnet and an electromagnet is a suitable option according to the displacements required for the PDMS membrane. However if this combination is used the small magnet needs to be attached to the all-polymer micronozzle and this will add to the price of the chip. This is against our research objectives that intended for fabrication of low cost and disposable micronozzles. The other options that have the similar disadvantage are piezoelectric stack actuators, and piezoelectric bender actuators. The most suitable option is a small electromechanical solenoid actuator. Solenoid City S-68-39 12V tubular actuator has been chosen for this purpose. The advantage of solenoids with respect to the mentioned actuators is that it does not need to be attached to the nozzles. It can be attached to the experiment setup.

4.2. Experiment Setup

The all-polymer micronozzle consists of three different components. The first component is an SU-8 micronozzle chip which is bonded to the second piece a PDMS microfluidic device. A cubic PDMS support is bonded to the device in order to allow for connecting the device to a syringe pump without breaking the thin PDMS layer.

For actuating the new micronozzles these new devices must be connected to a printer to be able to characterize and compare the performance with commercial inkjet nozzles. For this purpose, a custom-made printer in our lab is used. Since the new nozzles are small and fragile, a holder device is needed to support the nozzle as well as the solenoid and also maintain the required distance between these two in such a way that when the solenoid is off it touches the surface of the PDMS membrane without pushing it down. In addition, this device should have the capability to be easily snapped on the custom-made printer and removed each time it is used since as proposed in the objectives of this project these devices are disposable.

A 3D design for the holder device is made using SolidWorks software. Figure 4.3 shows a 3D view. The large circular hole is designed to hold the solenoid in its place. The solenoid will be screwed to this part. The bottom part of the device is designed to be able to connect it to the camera and the strobe system of the custom printer. The other part of the holder device clings to the metal part of the holder device and is secure enough to support the solenoid's movement and the pressure it puts on its bottom layer.

After the design is completed, the device is split using the split option available in SolidWorks in order to be printed by the Maker-Bot 3D printer. No hanging structures should be present in each part that is being printed and also this 3D printer is not able to print structures with an incline bigger than 45° C. The design is transferred to the printer's memory card and each structure is printed separately. Then all of the structures are bonded together using a solution of the same plastic material which is used in the Maker-Bot printer (ABS) diluted in acetone. When acetone is evaporated the whole structure is one strong big piece as if it has been printed all at once.

Figure 4.4 shows a photograph of the front view of the experimental set-up. In this figure the solenoid is mounted on the plastic holder device. The polymer micronozzle is mounted inside the holder. The solenoids plunger is accurately placed on top of the output channel of the PDMS microfluidic device. Figure 4.5 shows a schematic of the experimental setup. A pressure sensor is connected in a feedback loop between the syringe pump and the computer. The micronozzle and the solenoid are connected to the 3D stage of the printer.

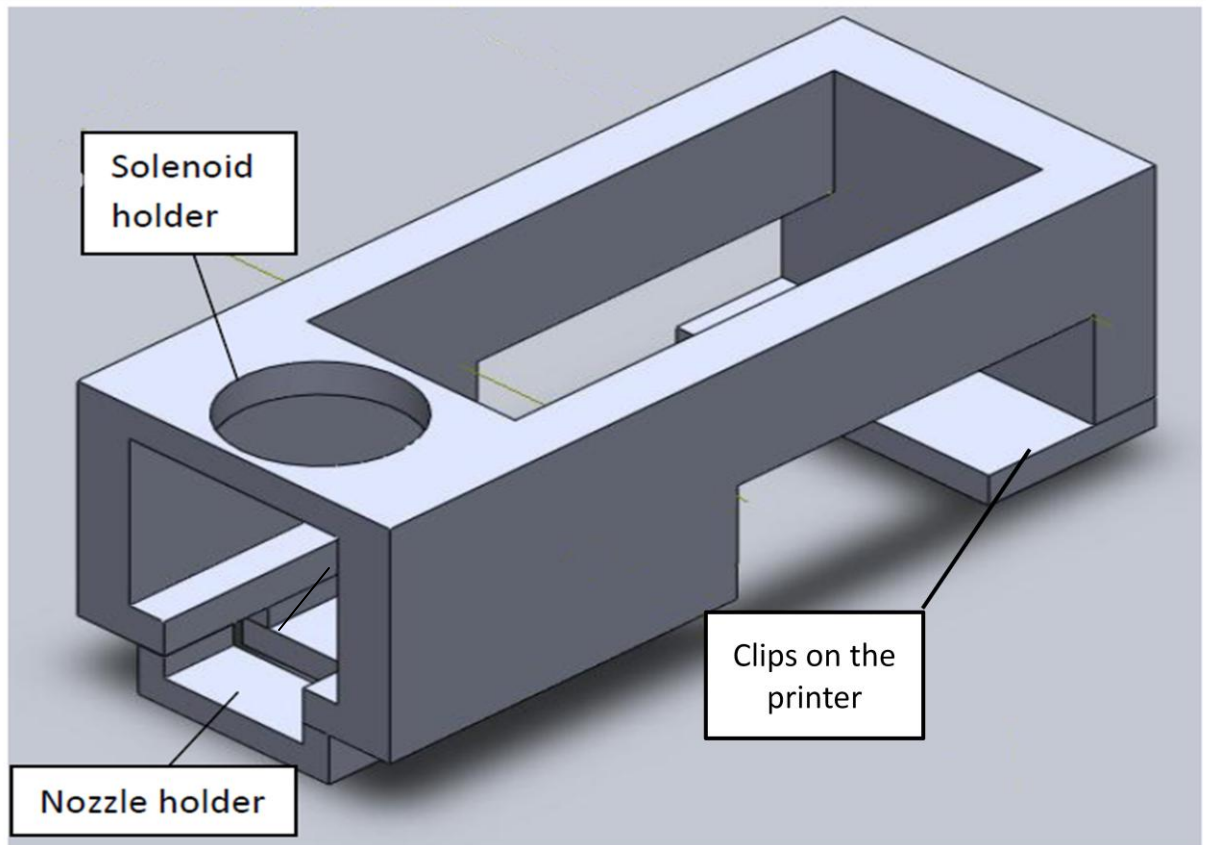


Figure 4.3 Holder Device designed using SolidWorks

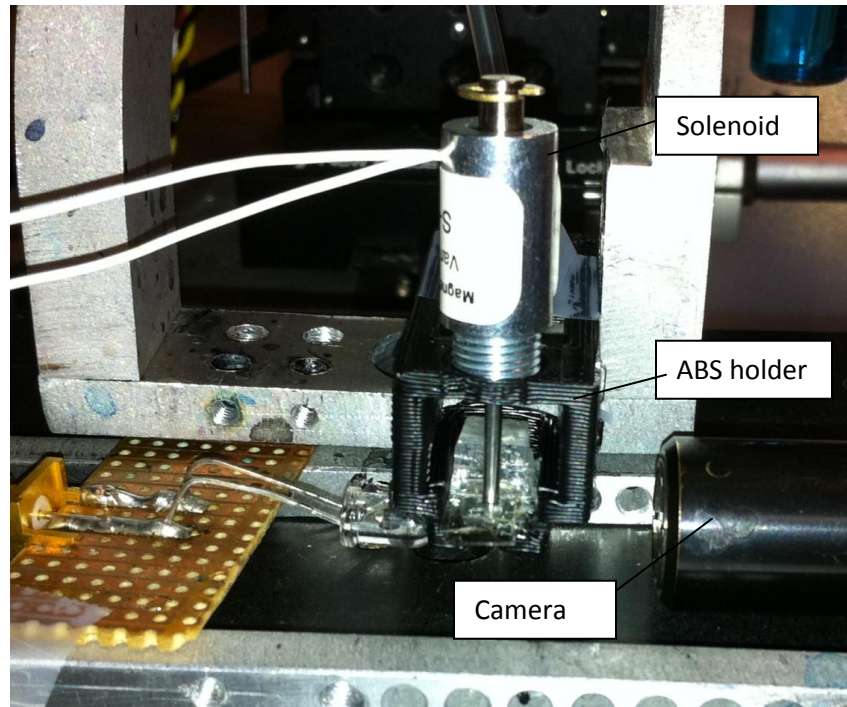


Figure 4.4 Experimental Set-up

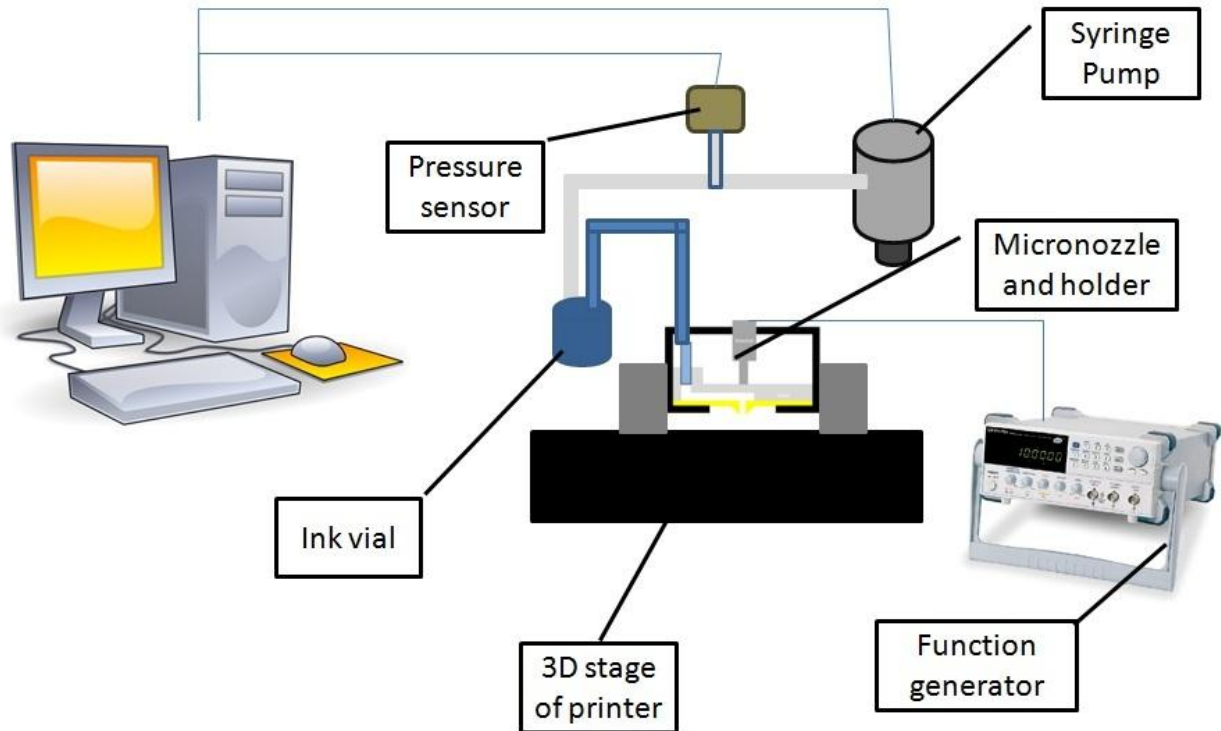


Figure 4.5 Experimental setup

Different experiments have been designed to test the performance of the fabricated micronozzles. The three main variables that can be considered in inkjet printing are the internal pressure, input pulse (operating frequency, the driving backpressure and the pulse width of the input signal) and surface properties. In each of the completed experiments, the corresponding effects on the volume change of the liquid out of the nozzle orifice will be observed by varying one of the above parameters and keeping the rest of the parameters constant.

Here is a brief summary of the experiments which will be explained in more detail in the following chapter:

Experiment 1:

By keeping the frequency and the duty cycle of the actuator constant, the internal pressure which is applied to the input aperture of the nozzle using the syringe pump, will be increased until a constant meniscus out of the nozzle is achieved. The first potential challenge is that if the bonding strength between the SU-8 and PDMS is not sufficient, this applied pressure may break the bond between these two. The backpressure is controlled by a syringe pump connected to the custom made inkjet printer in our lab. It is measured using a pressure sensor connected to the syringe pump.

Experiment 2:

By keeping the driving pressure and the pulse width constant at all times, the effect of actuation frequency on the volume of the droplet or volume forced out of the nozzle is characterized. This experiment is performed for three different nozzle sizes of 30 μ m, 50 μ m and 200 μ m.

Experiment 3:

The effect of adding the surfactant Pluronic F68 to water to reduce its surface tension is observed. In this experiment two different concentrations of Pluronic F68 are added to water. The internal pressure, surface wetting and volume change of the liquid are characterized.

The actuator plunger is cylindrical and has 1.2mm radius which is more than twice the size of PDMS output channel. Since we need to deform the PDMS layer on top, a smaller plunger tip is necessary. After doing several experiments, it seems that a spherical tip is a better option compared to a cylindrical tip. A half sphere made out of ABS plastic is attached to the tip of the solenoids plunger. Figure 4.6 shows the alignment of this half sphere. The diameter of the sphere is 500 μ m the same as the PDMS membrane.

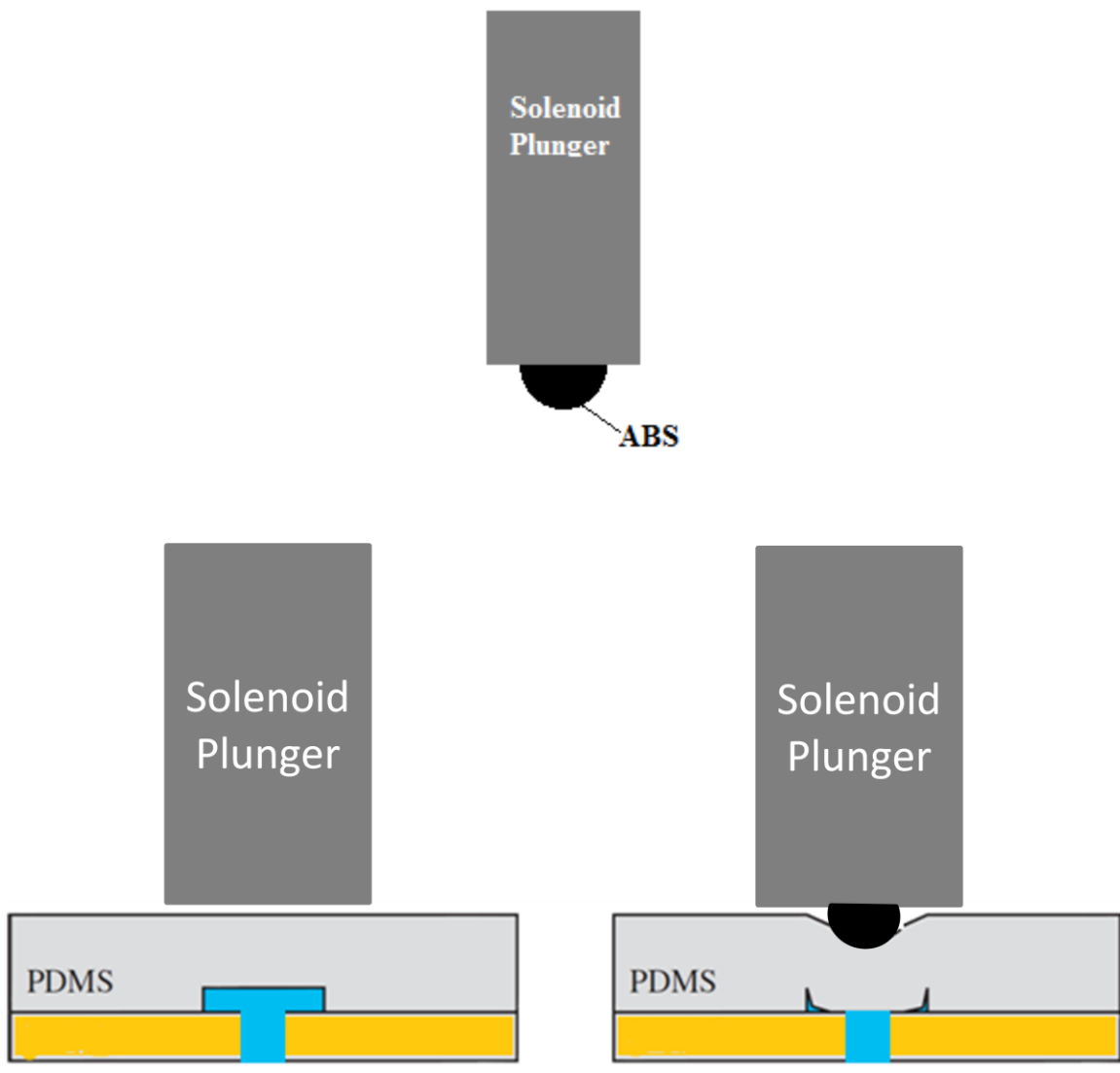


Figure 4.6 ABS attachment on solenoid plunger

5. Results and Discussion

5.1. Internal Pressure

In this chapter the results of the experiments performed on the fabricated all-polymer micronozzles is described. The first experiment performed on the micronozzle chip, was achieving a constant internal pressure for each different diameter of the micronozzles (30 μm , 50 μm , 200 μm). At this pressure the created meniscus is stable and liquid does not tend to flow out of the nozzle or flow back into the nozzle. At this stage the gravity, surface tension and the force from the pressure of the syringe pump are at equilibrium. Two different cases will happen due to fluid characteristics. The first case is a negative meniscus which is created when negative pressure is needed to keep the liquid at equilibrium. Figure 5.1 a, depicts a negative meniscus inside the 200 μm diameter micronozzle. The negative meniscus is caused because the liquid possessed low surface tension (due to using surfactants). Negative pressure was needed to keep the liquid from flowing out of the nozzle. Figure 5.1 b on the other hand depicts a positive meniscus. In this case a high surface tension liquid (Water) is used. The surface tension of water causes a concave meniscus form out of the nozzle. In this case a positive internal pressure is needed to keep the liquid in equilibrium.

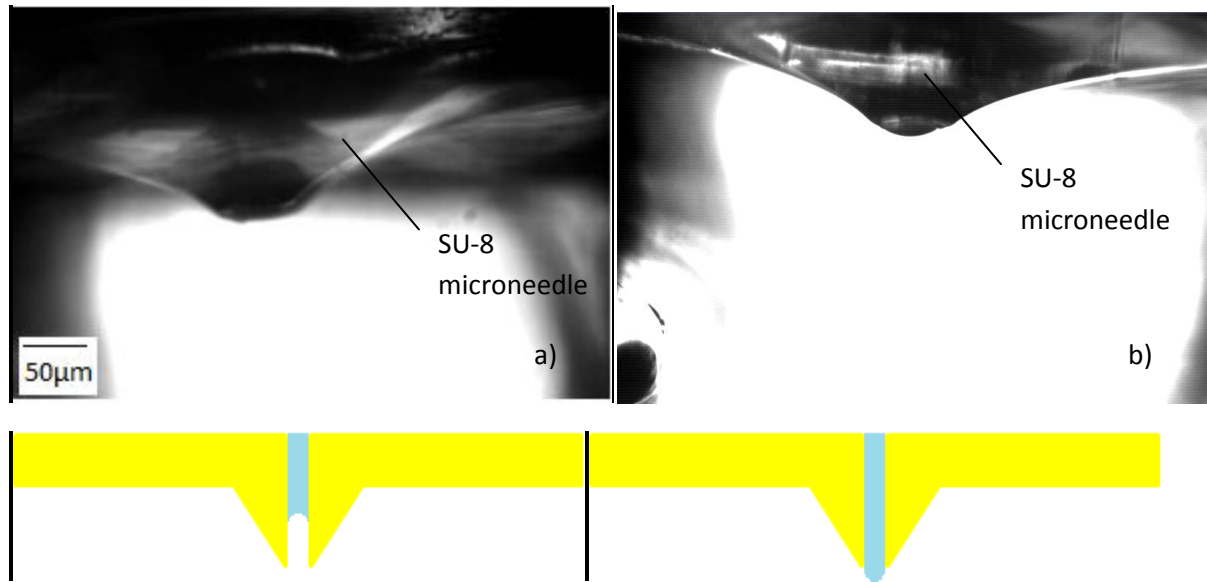


Figure 5.1 Sideview of the micronozzle a) Negative meniscus caused by negative internal pressure b) Positive meniscus caused by positive internal pressure

This experiment at first was performed with water. Water has high surface tension and creates a positive meniscus. Three different nozzle sizes were used to observe the effect of the diameter of the nozzle on the meniscus. With help of the pressure sensor which is connected to the syringe pump the internal pressure of the ink can be measured. This voltage can be converted to pressure using a linear formula which can be derived from the starting point and ending point of the voltage range matched to pressure range. Table 5.1 shows the internal pressure needed for each fabricated diameter of micronozzles. The micronozzles with smaller diameters require higher internal pressure. Figure 5.2 shows a chart showing the resulted internal pressure. The results of Table 5.1 are averaged between three different microchips at each different micronozzle size. For 30 μm nozzle size the standard deviation is 0.39. It is 0.27 and 0.11 respectively for 50 μm and 200 μm micronozzles.

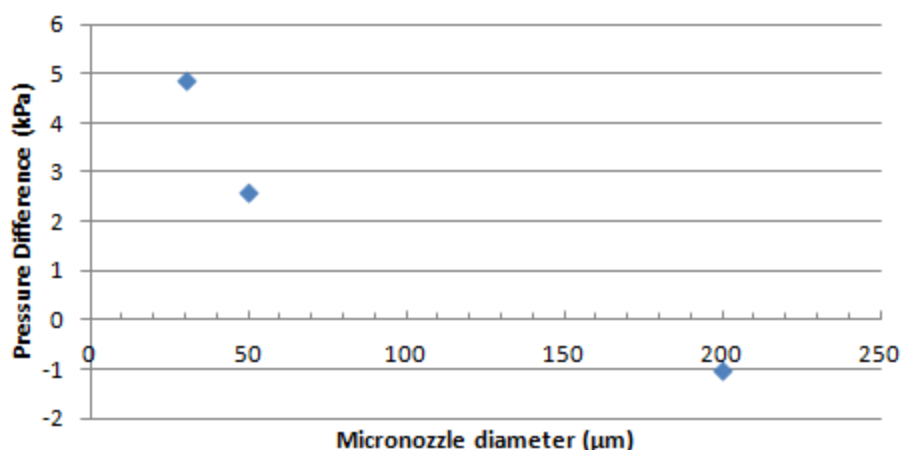


Figure 5.2 Internal pressure of water inside the nozzle (Water)

Table 5.1 Internal pressure of water inside the nozzle (Water)

Nozzle Size (μm)	Internal Pressure (kPa)	Pressure difference (kPa)
30	106.2	4.875
50	103.9	2.575
200	100.3	-1.025

As it is observed in Table 5.1, nozzles with larger diameter need less pressure to control the meniscus. It means that it is harder to control the fluid leakage out of the nozzle. With the larger nozzles a negative internal pressure is needed to keep the liquid from leaking out of the nozzle.

The second part of this experiment was to dilute two different concentrations of Pluronic F68 surfactant into the water to observe how it acts to reduce the surface tension of the fluid. One way to observe this is through the required internal pressure. The lower internal pressure indicates that the surface tension has been reduced. Figure 5.3 shows a chart showing the resulted internal pressure. Table 5.2 depicts the internal pressure at two different concentrations of Pluronic F68/water for three different nozzle sizes. Similar to situation with water, the smaller

diameters of the micronozzle result in higher internal pressures. Adding a surfactant has the advantage of reducing the internal pressure and creating a negative meniscus by reducing the surface tension.

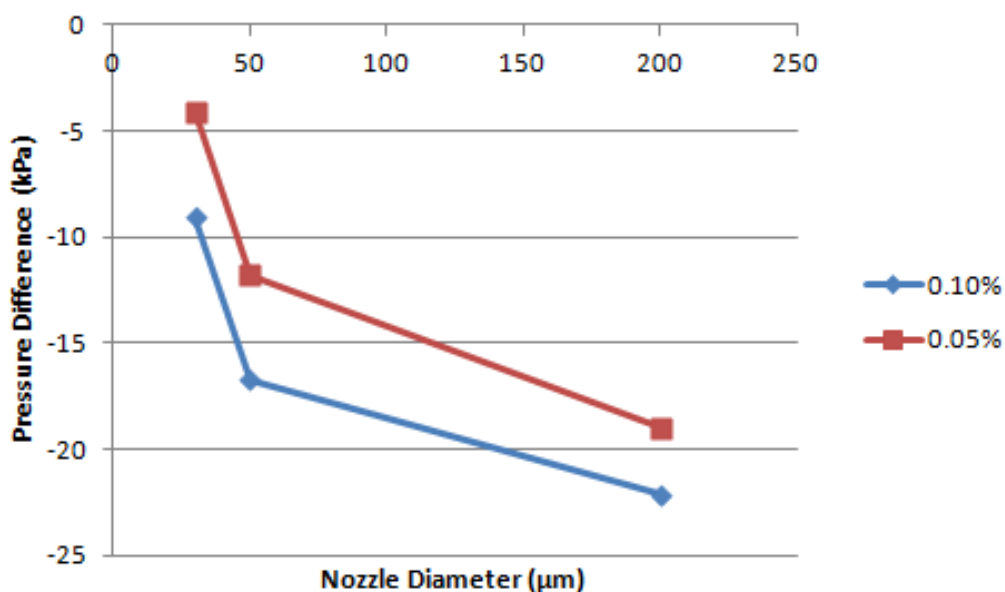


Figure 5.3 Internal pressure at two different concentrations of Pluronic F68

Table 5.2 Internal pressure at two different concentrations of Pluronic F68

Nozzle Size (μm)	Internal Pressure (kPa)		Pressure Difference (kPa)	
	0.1wt %	0.05 wt%	0.1wt %	0.05 wt%
30	92.26	97.22	-9.065	-4.105
50	84.58	89.55	-16.745	-11.775
200	79.17	82.33	-22.155	-18.995

The disadvantage of using a surfactant is that the low surface tension makes the liquid hard to control since it makes the device more prone to leakages. In order to achieve a uniform liquid jet

streaming out of the inkjet nozzle, the nozzles outside surface should act as a hydrophobic surface. In the experiments performed on the nozzle the liquid (water) would totally wet the surface after being ejected out of the nozzle. SU-8 is hydrophobic by nature but since each micronozzle chip has been plasma treated, the SU-8 surface of the nozzle will act hydrophilic which is not desirable. In [108] the authors have demonstrated that SU-8 surfaces that are treated with plasma will remain hydrophilic for several months and after that they will gain back some moderate amount of hydrophobicity. In addition to the characteristics of the fluid, the surface of the nozzle needs to be hydrophobic in order to get micro-droplets out of the nozzle.

Since the surface is hydrophilic adding a surfactant causes an ongoing flow of the liquid on the surface of the micronozzles. This flow will wet the surface and prevent creation of droplets. In general the ink should have a high contact angle (difficult wetting) with the outside surface of the nozzle. The surface free energy and wetting behaviour of the surface has caused the droplets to accumulate on the surface. The surface of SU-8 that was treated with plasma would keep its ultra hydrophilic properties for less than seven days. However it will remain hydrophilic for a long time (several months)[102]. Figure 5.4 depicts the total wetting of the outer surface of SU-8 which occurs as a result of uncontrolled flow of water/Pluronic F68 out of the nozzle and wetting of the hydrophilic surface of the plasma-treated SU-8. Figure 5.5 depicts the same situation when extra internal pressure (110kPa) inside the channel causes the ink (water without surfactant) to wet the hydrophilic surface of SU-8 outside the nozzle.

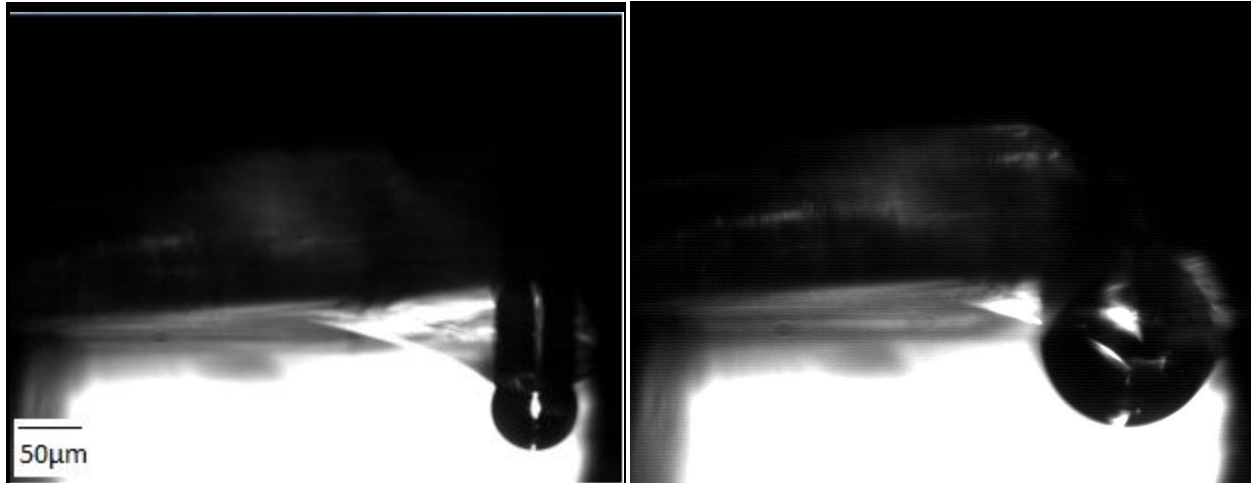


Figure 5.4 Surface wetting of surface caused by extra internal pressure in case water was used without surfactants

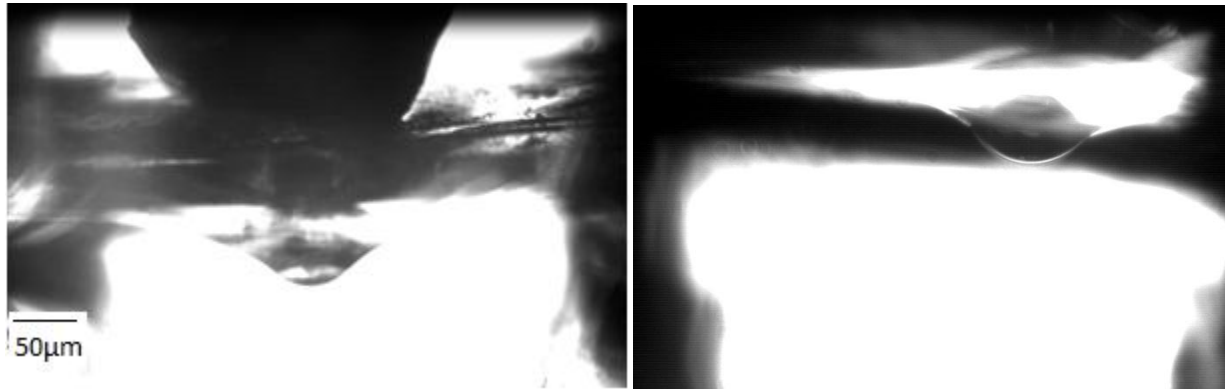


Figure 5.5 Surface wetting caused by out of control flow of water/Surfactant

From these results it can be concluded that the internal pressure required for the equilibrium of the liquid inside the nozzle, is a function of both the diameter of the nozzle and the liquid properties especially surface tension.

5.2. Input Pulse Shape

Now that the proper internal pressure is set for each nozzle, in order to tune the device for droplet ejection, the input pulse to the solenoid needs to be tuned. After the duty cycle tuning is performed and the best duty cycle is chosen, the device is tuned to find a voltage range that will

accommodate the chosen duty cycle. First this experiment starts with constant frequency of 1Hz and constant internal pressure inside each nozzle. Four different duty cycles are tested and the results are compared. The lowest duty cycle that the solenoid (S-68-38 solenoid city) can handle is 10%. The results in Table 5.3 show that the lower the duty cycle causes more displacement in the liquid in each nozzle size. Since frequency is constant, lower duty cycle is equal to lower pulse width. The kinetic energy transferred to the liquid from the solenoids plunger movement needs to be high enough to overcome the surface tension of the water. The results from the previous section depicts that since the surface of the plasma-treated SU-8 is hydrophilic having a low surface tension liquid as the ink is not advantageous in this case. Since the out of control flow of the liquid will wet the outer surface of the SU-8 and prevent formation of droplets, using the Pluronic F68/water solution will cause problems with the pressure control of the system and also droplet ejection. However the volume change for one case of the actuation of the micronozzle with the Pluronic F68/water has been calculated. The operation frequency is 1Hz, the duty cycle is 20% and the micronozzle's diameter is 50 μ m. The resulting volume change is 2.67×10^{-11} L. Figure 5.6 depicts the volume change in the Pluronic F68/ water solution. The left side shows the stable meniscus when the voltage is zero and the right side shows the resulted volume when the voltage is on.

The volume change in each case is evaluated with the help of the images captured at two different states of solenoid actuation by measuring the height and subtracting the volume when the voltage is off from the volume when the voltage is on. The volume of a part of a sphere is calculated using equations 5-1 to 5-3. The height is measured by knowing the diameter of the nozzle and scaling the height to the real size at each different image. Figure 5.7 shows an example of volume measurement. In this case a 50 μ m nozzle is used and the solenoid is

actuated at 50% duty cycle and 2Hz with 10V input voltage. In this figure h is measured and is $22.2\mu\text{m}$. The volume of the part of a sphere resulted from this displacement is $6.54 \times 10^{-11} \text{ L}$.

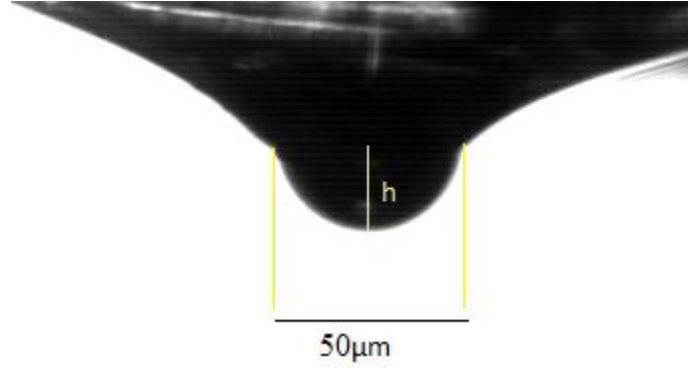
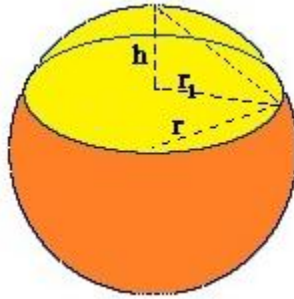


Figure 5.6 Volume measurement



5.7 Volume of a part of a sphere

$$R = \frac{h^2 + r_1^2}{2h} \quad (5-1)$$

$$V = \frac{4}{3}\pi R^3 \quad (5-2)$$

$$V = \frac{\pi}{6}(3r_1^2 + h^2)h \quad (5-3)$$

In these two equations each of the corresponding variables are shown in Figure 5.7.

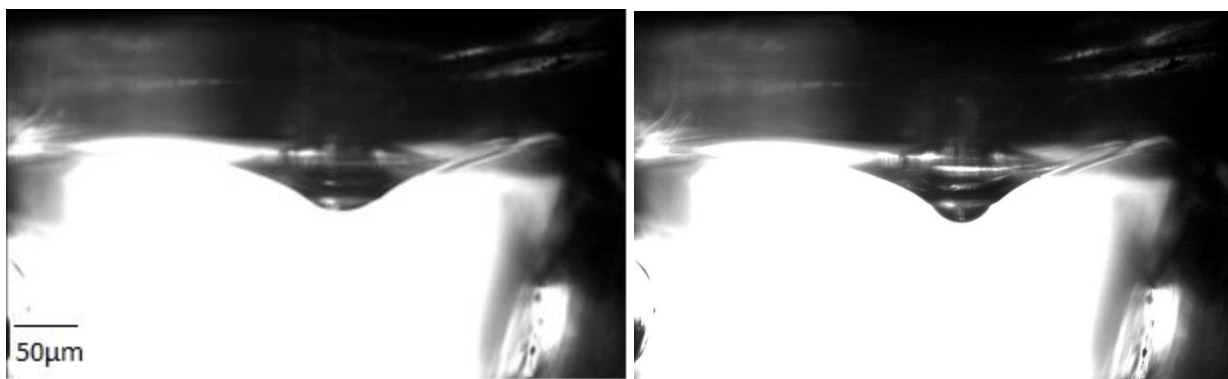


Figure 5.8 Pluronic F68/Water, 1HZ, 20%, 10V

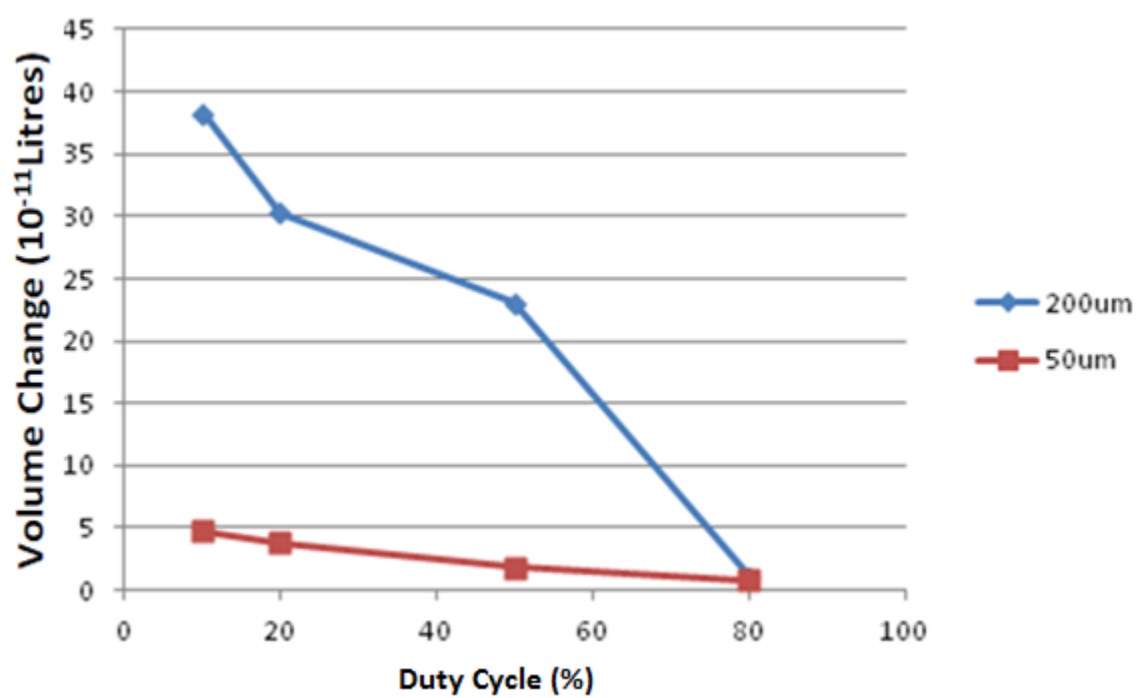


Figure 5.9 Effect of duty cycle on volume change, Water, 1Hz, 12V

Table 5.3 Effect of Duty Cycle on Volume Change, Water, 1Hz, 12V

Duty Cycle (%)	Volume Change (L)	
	Nozzle diameter 50 μ m	Nozzle diameter 200 μ m
10	4.78×10^{-11}	3.83×10^{-10}
20	3.82×10^{-11}	3.03×10^{-10}
50	1.85×10^{-11}	2.31×10^{-10}
80	8.2×10^{-12}	1.14×10^{-10}

After the effect of duty cycle on the volume change in the micronozzle was determined, the effect of frequency is tested on micronozzles with 50 μ m diameter and 200 μ m separately. In this case the duty cycle is kept constant at 50%. Four different frequencies are chosen for this experiment. In a stable system in each stroke of the plunger one droplet should be created out of the nozzle. However in most of the cases that were tested, a high frequency actuation would cause accumulation of the liquid on the meniscus and finally the liquid would wet the hydrophilic surface of the micronozzle. This phenomenon was found with frequencies over 50Hz. To calculate the droplet volume change in these cases that each stroke of the plunger does not cause just one drop forming out of the nozzle, the liquid volume change is calculated as explained here. First a time window is considered (1 Second). The measured volume change is then divided by the frequency to calculate the volume change at each stroke of the plunger. For the low frequencies this incident did not happen. With each stroke of the plunger a constant volume emerged temporarily from the nozzle. Table 5.4 depicts the volume change for four different frequencies and two different nozzle sizes. At the high frequencies the droplet volume change is small. The reason for accumulation of the liquid on the meniscus with each stroke may lie in the fact that the kinetic energy transferred from the solenoids movement to the liquid is not

high enough. In addition to low kinetic energy, the accumulation of liquid with each stroke creates an obstacle in the way of the liquid that wants to be ejected out of the nozzle. This accumulation does not happen in low frequencies since there is enough time for the liquid that could not overcome the surface tension to go back inside the nozzle after the voltage goes back to zero. In case there is no accumulation, there is no obstacle in the way of the liquid and with each stroke the same volume of liquid comes out of the nozzle and goes back in.

Table 5.4 Effect of frequency on volume change, duty cycle 50%, 12V

Frequency (Hz)	Volume change (L)	
	50µm Nozzle	200µm Nozzle
1	2.88×10^{-11}	2.13×10^{-10}
10	1.88×10^{-11}	1.51×10^{-10}
60	1.1×10^{-12}	9.2×10^{-12}
100	6.6×10^{-13}	5.3×10^{-12}

With the results for tuning the volume change resulted from each duty cycle, the device can be tuned to find the voltage amplitude that will create a droplet out of the nozzle. Table 5.5 shows the results for four different settings. Two different duty cycles (10% and 20%) and two different nozzle sizes (50µm and 200µm) are used. The operating frequency is kept constant at 1Hz. The maximum voltage that the solenoid can tolerate is 12 V. In none of the cases the voltage tuning caused droplet ejection. The results indicate that for the experimented duty cycles (10% and 20%) a higher range of voltage is required to create droplets. For some duty cycles there is no

voltage range that will satisfy the droplet formation. Table 5.5 shows the volume change in four different cases.

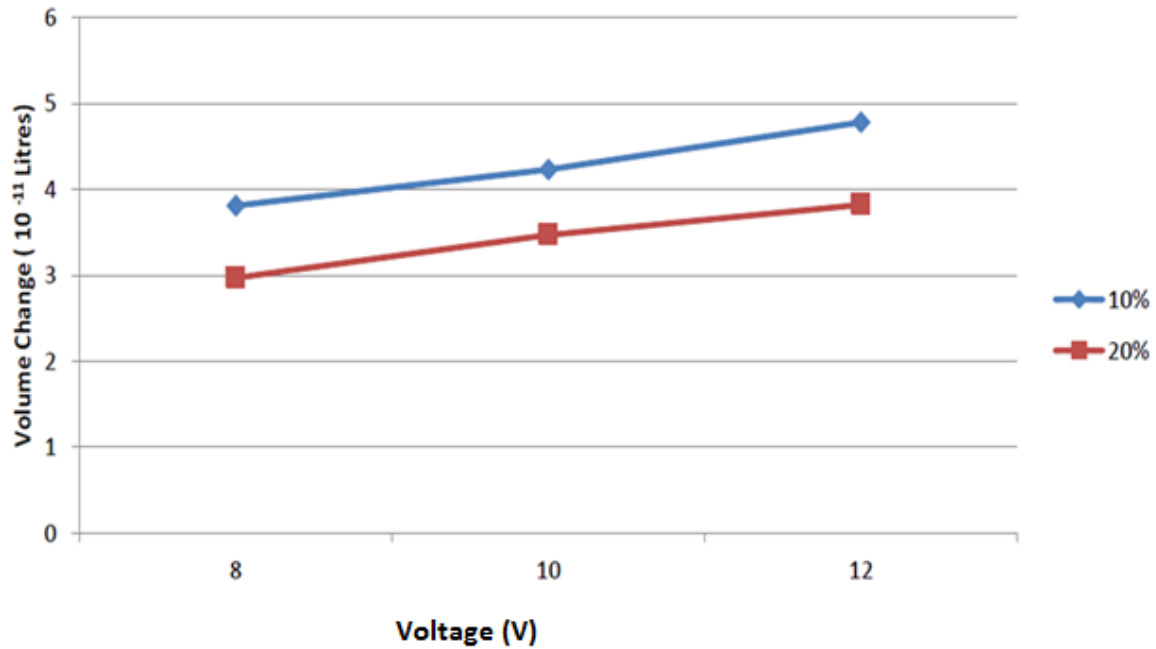


Figure 5.10 Volume change for three different voltages for a 50µm nozzle

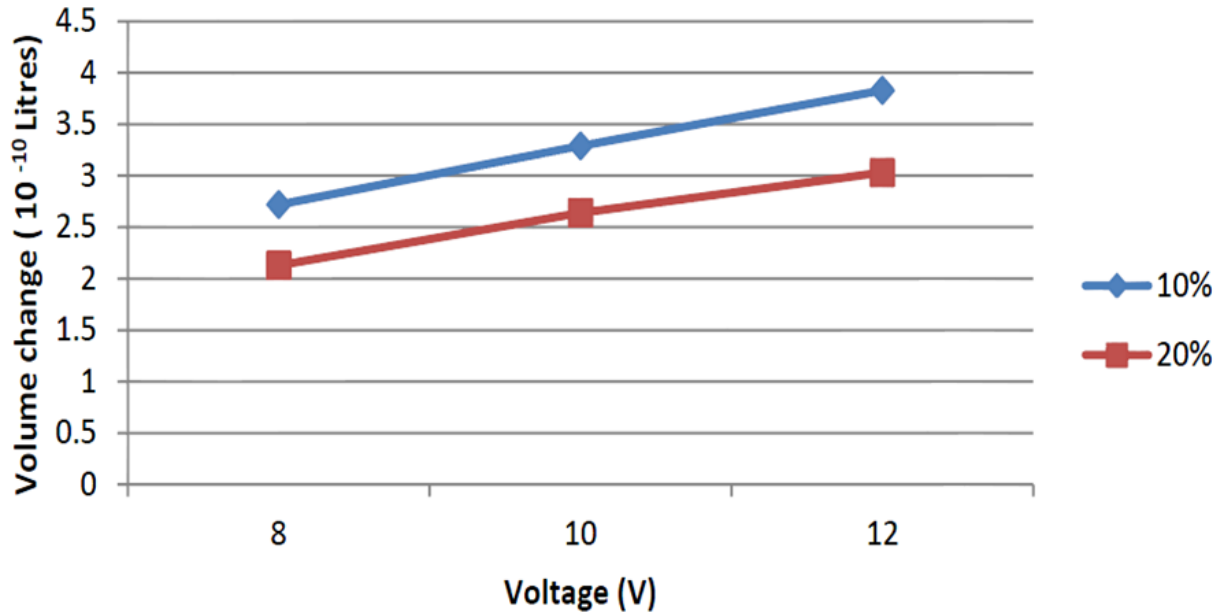


Figure 5.11 Volume change for three different voltages for a 50µm nozzle

Table 5.5 Volume change for three different voltage amplitudes

Input Amplitude (V)	Voltage	Volume change (L)			
		Duty Cycle 10%		Duty Cycle 20%	
		50µm	200 µm	50 µm	200 µm
8		3.81×10^{-11}	2.72×10^{-10}	2.97×10^{-11}	2.13×10^{-10}
10		4.23×10^{-11}	3.29×10^{-10}	3.47×10^{-11}	2.64×10^{-10}
12		4.78×10^{-11}	3.83×10^{-10}	3.82×10^{-11}	3.03×10^{-10}

These results suggest that the voltage change of 2V in each case does not have a large effect on the volume change. In addition, the tables are suggesting that for the experimented duty cycles this voltage range is not high enough to create droplets.

The volume change of the liquid is depicted for several cases to give a visual sense of the effect of changing different parameters on the volume change. Figure 5.12 depicts the volume change in a 50 μ m nozzle resulted from actuation with input pulse of 10V with 20% duty cycle and 1Hz. In figure 5.13 the voltage is increased to 12V.

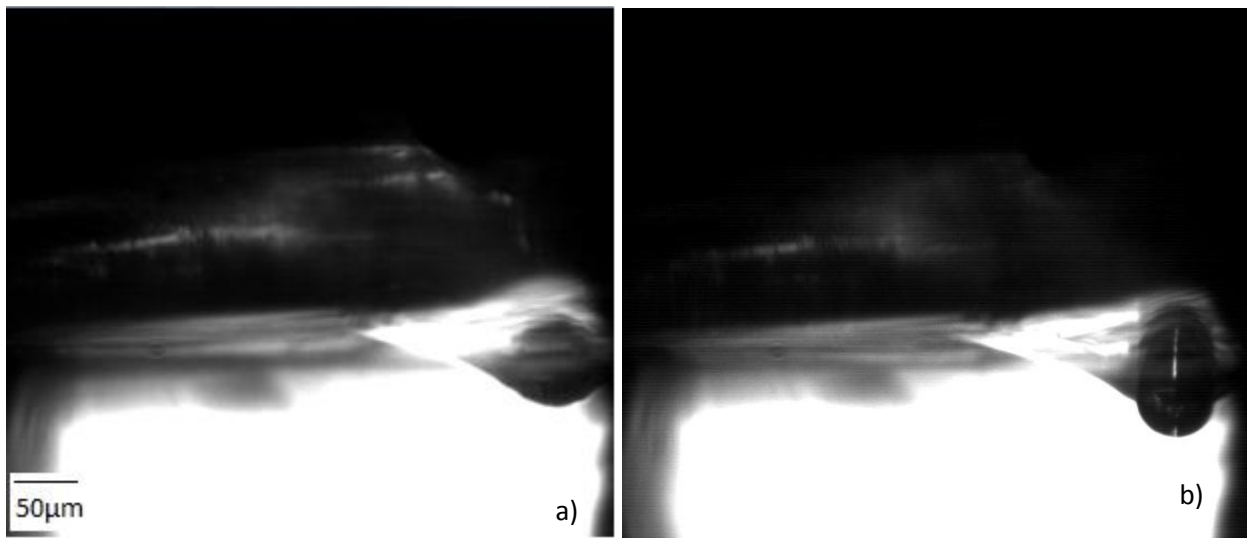


Figure 5.12 50 μ m nozzle ,20% duty cycle,1Hz at a) Zero Voltage b) 10V

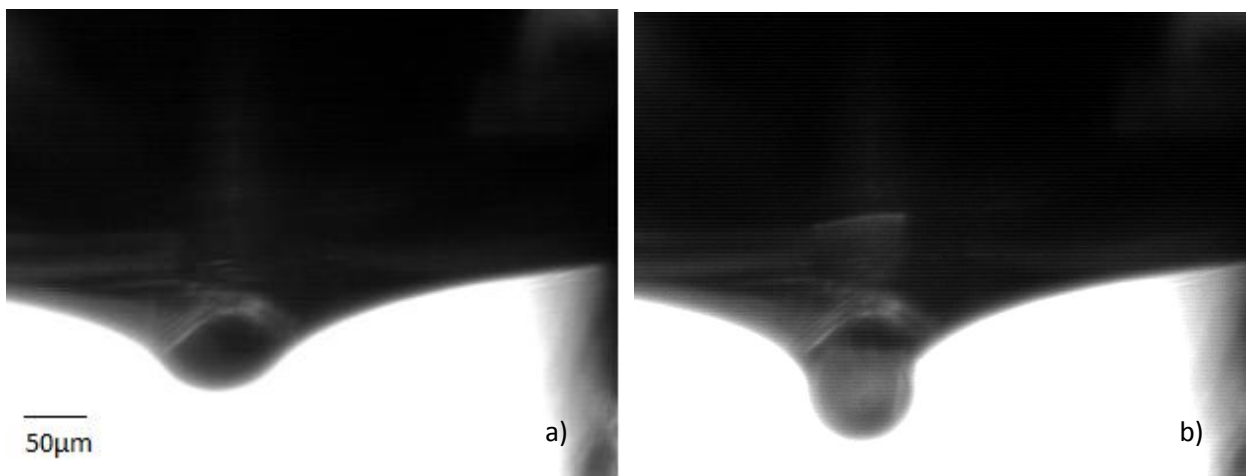


Figure 5.13 50 μ m nozzle, 10% duty cycle, 1Hz at 12 Volts

Figure 5.14 depicts the volume change in a 200 μ m nozzle with a 10V input pulse, 50% duty cycle working at 1Hz.

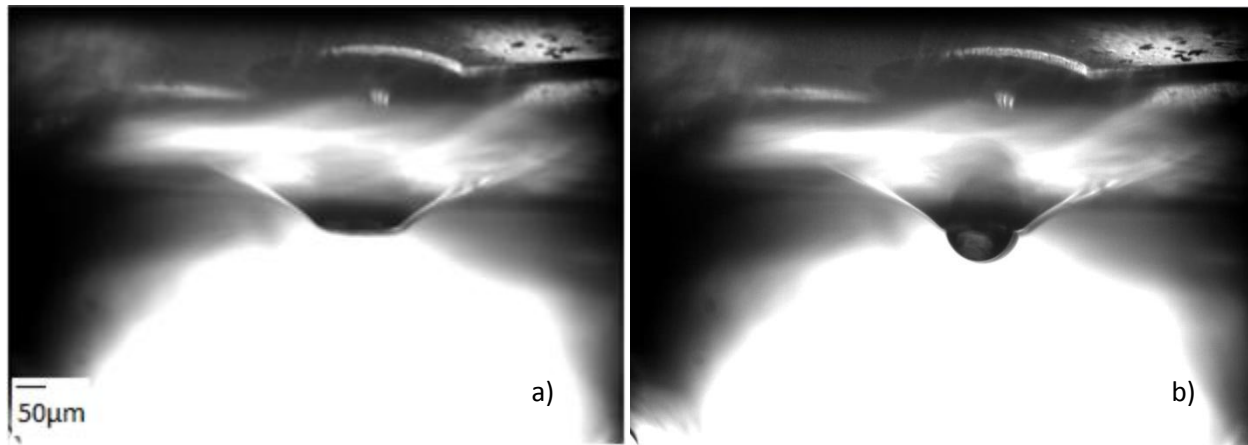


Figure 5.14 200 μ m, 1HZ, duty cycle 50%, 10Volts, 200 μ m

5.3.Other Factors

To make improvement to the volume change resulted from actuation, and to make sure no liquid is flowing back inside the channel a valve was placed before the input of the microfluidic chip as shown in Figure 5.15. Exactly after the proper meniscus was created the valve was closed. Experiment results indicated that the placement of the valve will reduce the displaced volume as opposed to what was predicted. Table 5.9 demonstrates the volume change for a 50 μ m micronozzle with a 10V input pulse operating at 1Hz for three different duty cycles these data are depicted in a chart in Figure 5.16. When there is no pressure force, the capillary forces will dominate and pull back the created meniscus to the inside of the polymer micronozzle. Since PDMS is elastic it may expand and make room for some part of the liquid instead of going through the nozzle orifice. Table 5.9 shows the effect of closing the valve on the volume of the liquid which is pushed outside the nozzle.

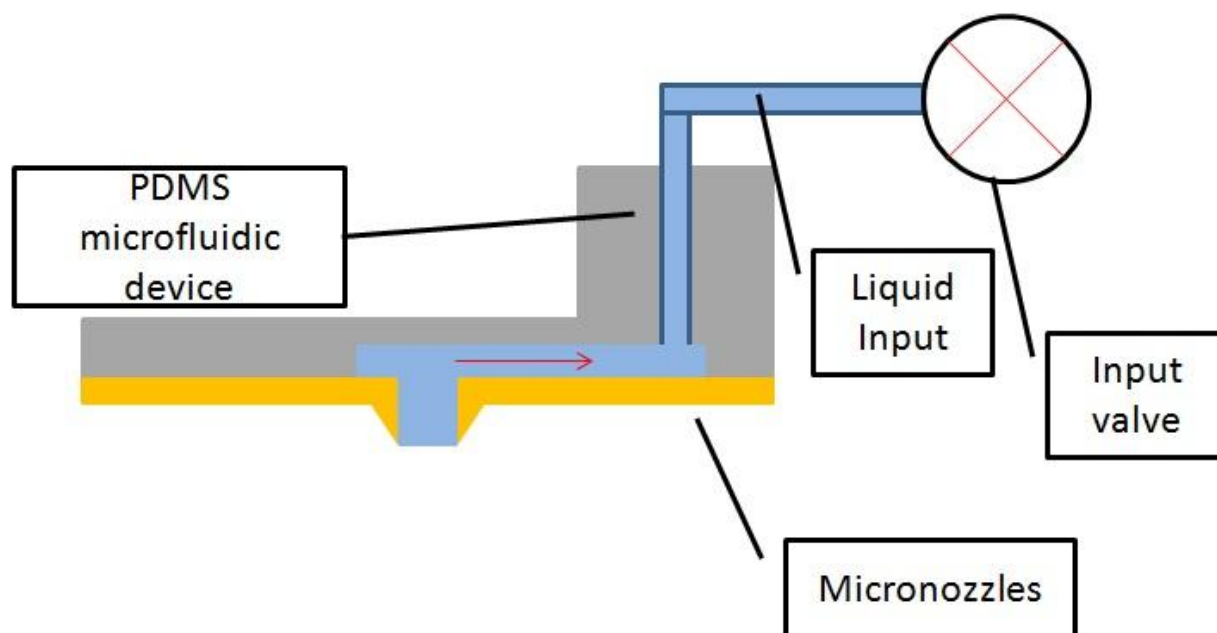


Figure 5.15 Schematic of the input valve

Table 5.6 Effect of input valve on volume change for three different duty cycles at 1Hz, 10V , 50 μ m nozzle diameter

Duty Cycle (%)	Volume Change (L)	
	Input Valve off	Input Valve on
10	4.78×10^{-11}	3.83×10^{-11}
20	3.82×10^{-11}	3.03×10^{-11}
50	1.85×10^{-11}	2.31×10^{-11}

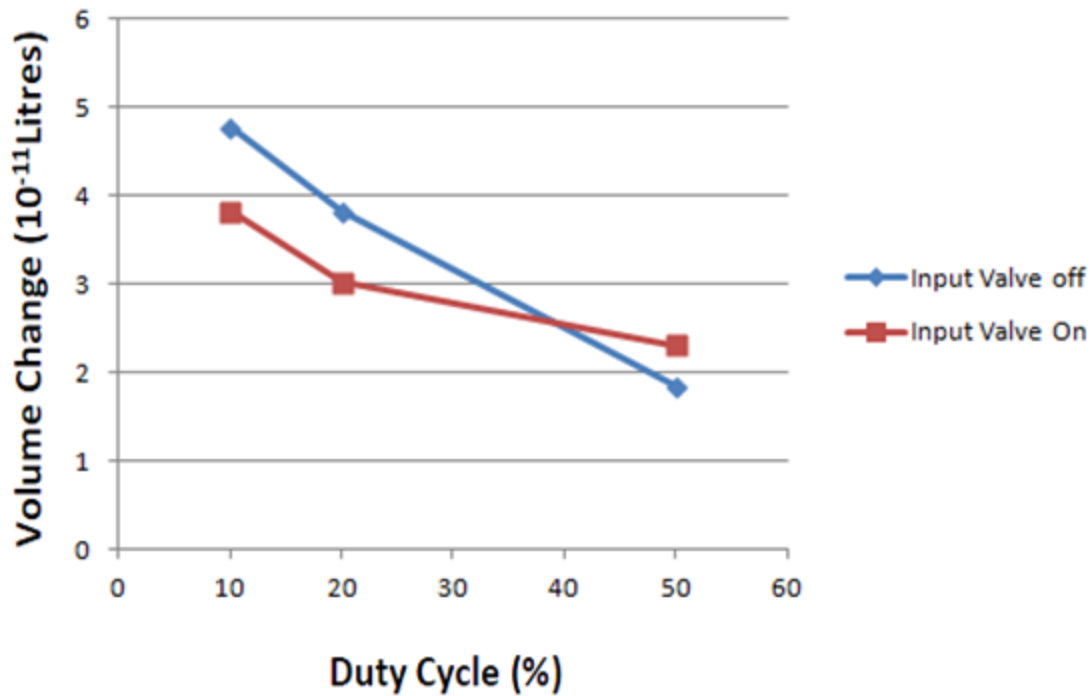


Figure 5.16 Effect of input valve on volume change for three different duty cycles at 1Hz, 10V

As long as the valve is closed (high voltage for solenoids input pulse), the formed droplet is kept out of the nozzle in a controlled manner. As soon as the voltage goes back to zero and the valve is opened, the droplet goes back to the inside of the nozzle. The solenoid is working as an on-off switch. When the switch is on (10V) a droplet with the volume change is created.

If the movement of the liquid jet is not fast enough, internal pressure on the system from the syringe pump will create a large droplet which will break off the surface tension of the ink as a result of its weight. Since the surface of SU-8 is plasma treated, its hydrophobic nature has changed to hydrophilic. The created large droplet will as a result of this hydrophilicity wet the surface instead of falling off the nozzle. This effect is shown in figure 5.17.



Figure 5.17 Total wetting of the surface resulted from low kinetic energy and high internal pressure

Another case is when the speed of the fluid is not high enough but the frequency is increased. The high frequency will act similar to the high pressure in the system and force a large droplet out of the micronozzle. The creation of this large drop will result in total wetting of the hydrophilic surface of the SU-8 as soon as just a small amount of the drop gets in contact with the surface. Figure 5.18 depicts the resulted surface wetting in this situation. In the left picture there is the accumulation of the liquid out of the nozzle resulted from high frequency and in the right the image shows the instant that this large droplet wets the surface.

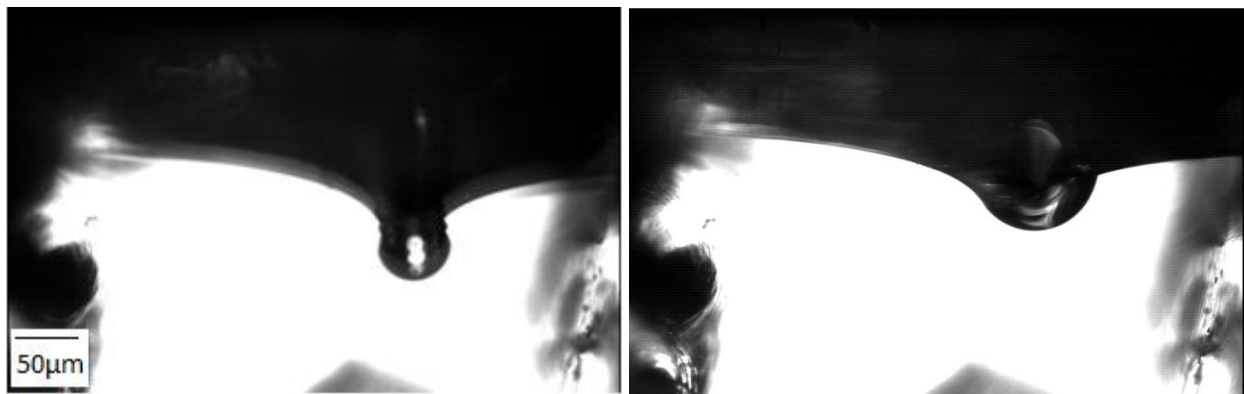


Figure 5.18 Surface wetting caused by high frequency and low kinetic energy

6. Conclusions and Future Work

6.1.General Discussion

Low cost disposable all-polymer micronozzles were fabricated. These nozzles consist of two different parts. First part is the polymer nozzle-shaped structures. The second part is the microfluidic microchannels. These devices were first fabricated and then bonded together using oxygen plasma. Transdermal drug delivery microneedles were used as micronozzles. One of the most important factors to fabricate these polymer micronozzles was to choose the polymer structure material of the micronozzles. Initially polyimide was chosen for the micronozzles. Due to several disadvantages of this type of micronozzles the structure material was then changed to SU-8. SU-8 micronozzles resulted in several advantages over polyimide. The most important advantage is the strong bonding between SU-8 and PDMS with simple plasma treatment as opposed to required adhesive layer in the polyimide system.

A simple microfluidic device consisting of one input channel and one output channel each with a 500 μ m diameter, and connected with a channel 100 μ m in width, was fabricated using soft lithography process and PDMS casting. To achieve a thin PDMS membrane, spin casting of PDMS over the mold was required. The fabricated PDMS microfluidic thin layers were bonded to polymer nozzles using oxygen plasma treatment and precisely aligning the two layers together.

The resulting micronozzles were connected to the pressure system of a custom made inkjet printer, by the means of a plastic holder device. The holder device was designed in SolidWorks and printed using a 3D printer. Finally a solenoid actuator was attached to the setup. A different plunger tip was designed to maximize the deformation of the PDMS membrane which causes the liquid ejection. The effect of frequency and duty cycle of the solenoids input pulse on the created

pending droplet volume was experimented. Effect of adding a surfactant to the ink to reduce the surface tension of the liquid was also tested.

6.2. Conclusions

The combination of fluid characteristics and micronozzles diameter resulted in either a negative or positive meniscus caused by negative or positive internal pressure. Water created a positive meniscus with positive internal pressure inside a 30 μ m and 50 μ m nozzle. Negative internal pressure was created inside a 200 μ m nozzle in case water was used as ink. To reduce the high surface tension of water, Pluronic F68 was used with two different concentrations of Pluronic F68/ water. For all the three different nozzle sizes at two concentrations of 0.1wt% and 0.05wt% negative internal pressure was needed to create a stable meniscus. Although creation of negative meniscus is an advantage in drop-on-demand devices, in this case ongoing flow of Pluronic F68/ water resulted in total wetting of hydrophilic surface of plasma-treated SU-8.

Input pulse tuning was performed after proper internal pressure was set on each nozzle. Duty cycle of 10% is the minimum duty cycle achievable using the solenoid. After the effect of duty cycle was experimented, the frequency was changed to observe its effect on the droplet volume change. Since each stroke of the solenoids plunger does not transfer high enough kinetic energy to the ink, for frequencies higher than 50 Hz, in each stroke of the plunger the ink would accumulate on the nozzle's aperture preventing the liquid from being ejected out of the nozzle. This would result in accumulation of a large drop out of the nozzle and finally the drop would wet the hydrophilic surface of the micronozzle. At 1Hz with 50% duty cycle and 12V the volume change is 2.88×10^{-11} L for a 50 μ m nozzle and 2.13×10^{-10} L for a 200 μ m micronozzle. The volume of the ink chamber is 75×10^{-9} L this shows that just a small deformation in the PDMS membrane has been resulted.

Voltage tuning was performed for two different duty cycles of 10% and 20% and two nozzle sizes of 50 μ m and 200 μ m. Three different voltages of 8V, 10V and 12V were experimented which resulted in maximum volume change of 3.83×10^{-10} L. For the experimented duty cycles there was no voltage range which would transfer high enough kinetic energy to overcome the surface tension of water and create droplets. The highest voltage that the solenoid can withstand is 12V.

Another experiment that was performed was placing an on-off valve before the input of the micronozzle to prevent any flow back inside the microchannel when the voltage is on. As opposed to what was predicted, closing the valve reduced the volume change of the liquid outside the nozzle. For a 50 μ m nozzle at 10% duty, 10V and frequency of 1Hz the volume change is reduced from 4.78×10^{-11} L to 3.83×10^{-11} L.

Pulse width of the input pulse is the most important factor in tuning a drop-on-demand device. Another important factor in drop-on-demand droplet creation is that the outside surface of the aperture needs to be hydrophobic to create a stable liquid jet outside the nozzle that can be disturbed with the actuation.

In the experiments performed on the micro-nozzles, the liquid is pushed out of the nozzle with positive voltage and drawn back to the nozzle when the voltage goes back to zero. The maximum input voltage of the solenoid is 12 V.

6.3.Recommendations for Future Work

For achieving drop-on-demand ejection it is important to change the lower surface of plasma-treated SU-8 back to its hydrophobic nature. Teflon coating is a widely used technique to make different surfaces hydrophobic. One should be careful that the Teflon coating does not enter the

inside the nozzle's walls. The inside needs to be hydrophilic. If the inside walls are coated, then extra energy is needed to force the liquid outside the nozzle. Covering a surface with fluorine atoms is one way of making low surface energy surfaces but this process requires high temperatures which are not suitable for polymers such as SU-8 [111]. One idea is to cover the unwanted treated areas of SU-8 with another layer of SU-8 as an example using dip coating.

One technique of producing droplets is using a double pulse. The first pulse will eject the fluid, and the second one will break the surface tension. Solenoid actuators do not possess a fast enough response to produce double pulses with the required pulse width. A piezoactuator is suggested. The pulse widths in this case range between 1-3 microseconds with less than 30 microseconds separation between them. In the case of using a double pulse for actuation, finding the right separation between the two pulses has the major effect on creation of the droplet.

Drop on demand inkjet nozzles can result from this device with further developments on this kind of nozzles especially the actuators and improvements in their performance. Piezoelectric actuation should be used in order to achieve small pulse widths in the sub-millisecond range. Piezoelectric stack actuators are a good option since fast actuation is needed. The suggested piezoactuators work with low currents of about $10\mu\text{A}$.

The results are suggesting that either a lower duty cycle should be experimented or a higher voltage is required. With the current small solenoid actuator which is used in these experiments the lowest duty cycle is 10% and the highest voltage is 12 V. As a result the small solenoid actuators do not provide high enough kinetic energy inside the liquid to create droplets. There are solenoids which take advantage of input pulses with higher voltage amplitudes (24V). However these solenoids are not suitable for these all-polymer micronozzles. The large size of these high-

voltage solenoids does not accommodate the fabricated small-sized micronozzle chip. High-speed actuators such as piezoelectric actuators may be required to achieve a duty cycle and amplitude which will result in droplet creation.

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Appendices

Appendix A. Detailed fabrication of molds for microneedles

Standard MEMS photolithography process was used to fabricate the molds required for both types of microneedles listed in this thesis (polyimide and SU-8). The fabrication process was performed in UBC AMPEL Cleanroom (class 10). Pyrex wafers of two different thicknesses of 500 μ m and 300 μ m were used. The negative thick photoresist SU-8 2150 is used as the structure for the mold.

First the Pyrex wafer is washed with acetone for 2 minutes followed by isopropanol for 2 minutes. The process should be repeated until the wafer is completely clean. Followed by the cleaning process, the wafer is rinsed with di-ionized water and dried with nitrogen gun. To completely dry the surface the wafer is heated up to 120°C on a hotplate for 5 minutes. When the wafer is completely clean and dry, the photoresist is spin coated on the wafer. The wafer should be placed on the center of the chuck then the vacuum pump should be turned on to fix the wafer in place. As mentioned before SU-8 2150 is a thick and sticky photoresist. It is challenging to pour the thick photoresist on the wafer. SU-8 is poured on the center of the wafer until half of the wafer is filled with SU-8. Depending on the required thickness of the mold, different spinning speeds and times are required. After spinning process is finished, the wafer is soft baked on a hotplate. A flat hotplate surface is necessary in order to get uniform distribution of SU-8 on the mold. The next step is the photolithography of the sample. The masks that have been used throughout this project are listed in Appendices C.

SU-8 as mentioned is a negative photoresist. In a negative photoresist the exposed parts will stay after the exposure and the unexposed areas are washed away using the developer. The exposure

technique which is used for this mold is to expose the UV light from the backside of the Pyrex wafer. Before exposure the backside of the glass wafer is wiped with acetone. During the exposure the photoresist side of the wafer should be facing down with the mask on top of this layer. The exposure energy needed is dependent on the thickness of the SU-8. The exposed masks are then placed on a hot plate for a post bake of 2 hours. Then the samples are placed in a beaker of SU-8 developer. After the development step, the unexposed areas of SU-8 are washed away. To ensure that the development process is complete the wafer is removed from the developer and washed with isopropanol for 30 S. If a white milky liquid was released the wafer is under developed and the development step needs to be repeated for a few minutes until the white liquid does not appear again.

Table A.1 Mold Fabrication parameters for microneedles

Spinning	500 rpm @ 110 rpm/s for 10 seconds 2000 rpm @ 330 rpm/s for 30 seconds
Soft Bake	10 min @ 65°C 100 min @ 95°C
Exposure	11 minutes and 40 seconds 6.9 mw/cm ² exposure power
Post Exposure Bake	5 min @ 65°C 30 min @ 95°C
Photoresist Development	45 min in SU-8 2000 developer

Appendix B. Mold fabrication for microfluidic device

The fabrication process of master molds for PDMS is similar to fabrication procedure of molds for microneedles. SU-8 2025 or SU-8 2075 (negative photoresist) are used as the structure of this mold. The substrate is a silicon wafer. First the silicon wafer is cleaned with acetone and isopropanol until completely clean. For a better cleaning process piranha wet etch (H_2SO_4 and H_2SO_2) can be used. Then the wafer is washed with DI water and dried with nitrogen gun. For evaporation of all the water droplets the wafer is placed on hotplate in 120°C for 5 minutes. There is poor adhesion between SU-8 and a silicon wafer. For producing molds that last longer and the structure material does not come off the surface after usage, HMDS is used as an adhesion promoter. The next step is the spinning process. We fix the wafer in the center chuck of the spinner with the vacuum on. SU-8 2025 is poured on the wafer starting from the center until 50% of the wafer is filled with SU-8. Avoiding bubbles is necessary when pouring the SU-8. Then the wafer is spanned at the speeds given in Table B.1. After the spinning process is complete, the wafer needs to be soft baked on a uniform and level hotplate. After the soft bake first we allow the wafer to cool down and then expose it using the photolithography mask aligner. After the exposure the wafer is baked and cooled down. SU-8 2000 Developer is used to remove the unexposed areas of photoresist. The wafer is then rinsed with fresh developer and isopropanol. If no white milky liquid appears the sample is developed. If not, another few minutes of rinsing in the developer are needed until no white liquid appears. Finally an optional step of hard bake at 30 minutes in 150°C can improve the final mold structure. Table B.1 shows the fabrication parameters of the master mold for microfluidic device.

Table B.1 Master Mold Fabrication properties

Spinning	500 rpm @ 110 rpm/s for 10 seconds 2000 rpm @ 330 rpm/s for 30 seconds
Soft Bake	5 min @ 65°C 20 min @ 95°C
Exposure	3 minutes 6.9 mw/cm ² exposure power
Post Exposure Bake	5 min @ 65°C 10 min @ 95°C
Photoresist Development	45 min in SU-8 2000 developer

Appendix C. Masks

The following figure shows the darkfield mask used for fabrication of microneedle molds. The mask contains many arrays of circular dots enclosed by transparent square or circular regions. The mask was designed using CleWin Layout Editor v. 4.0.2 and printed by CAD/Art Services (Bandon, OR).

Figure C.1 Mask for microfluidic channels of width 100μm and length 400μm

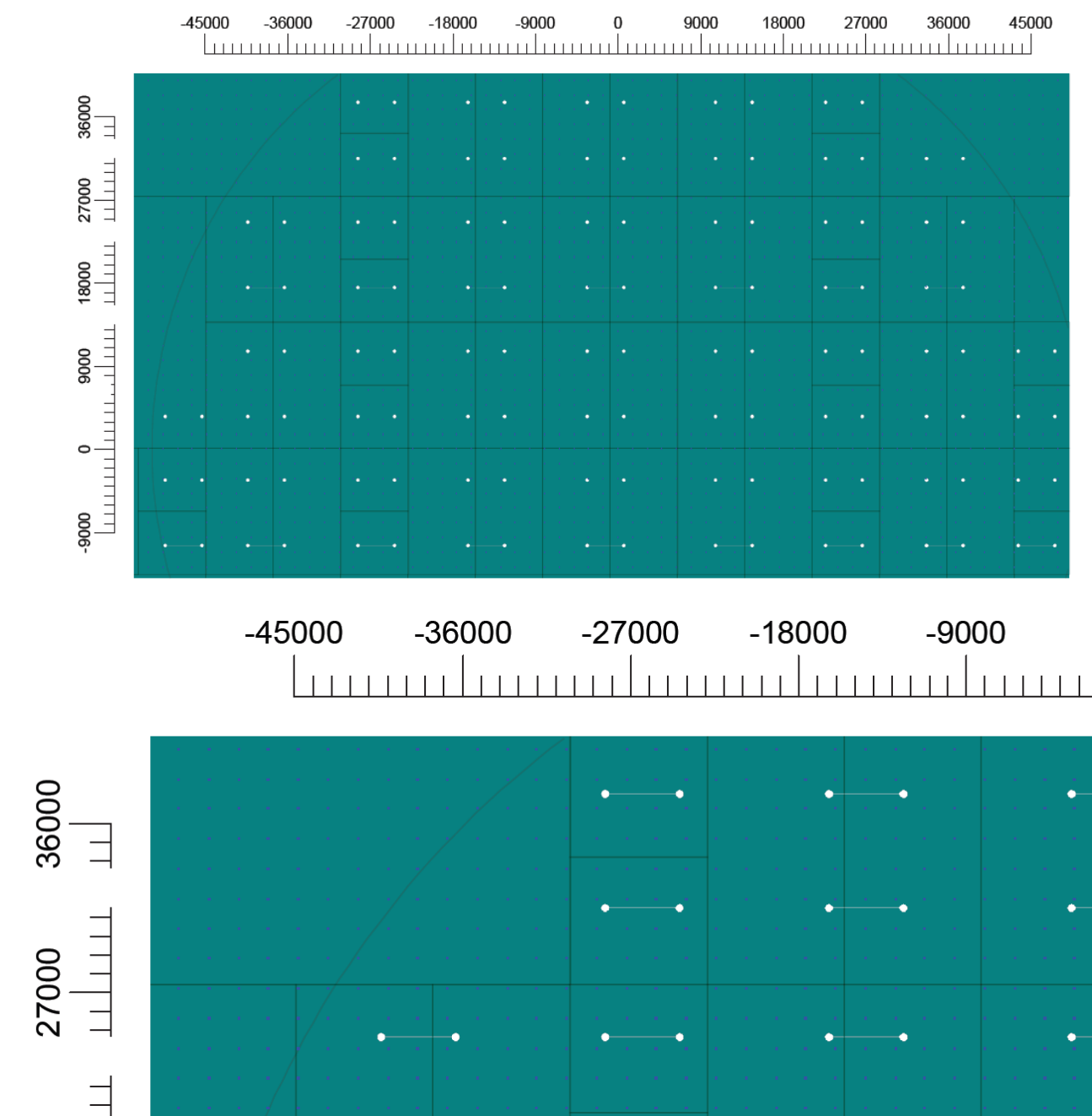


Figure C.2 Mask for microneedles

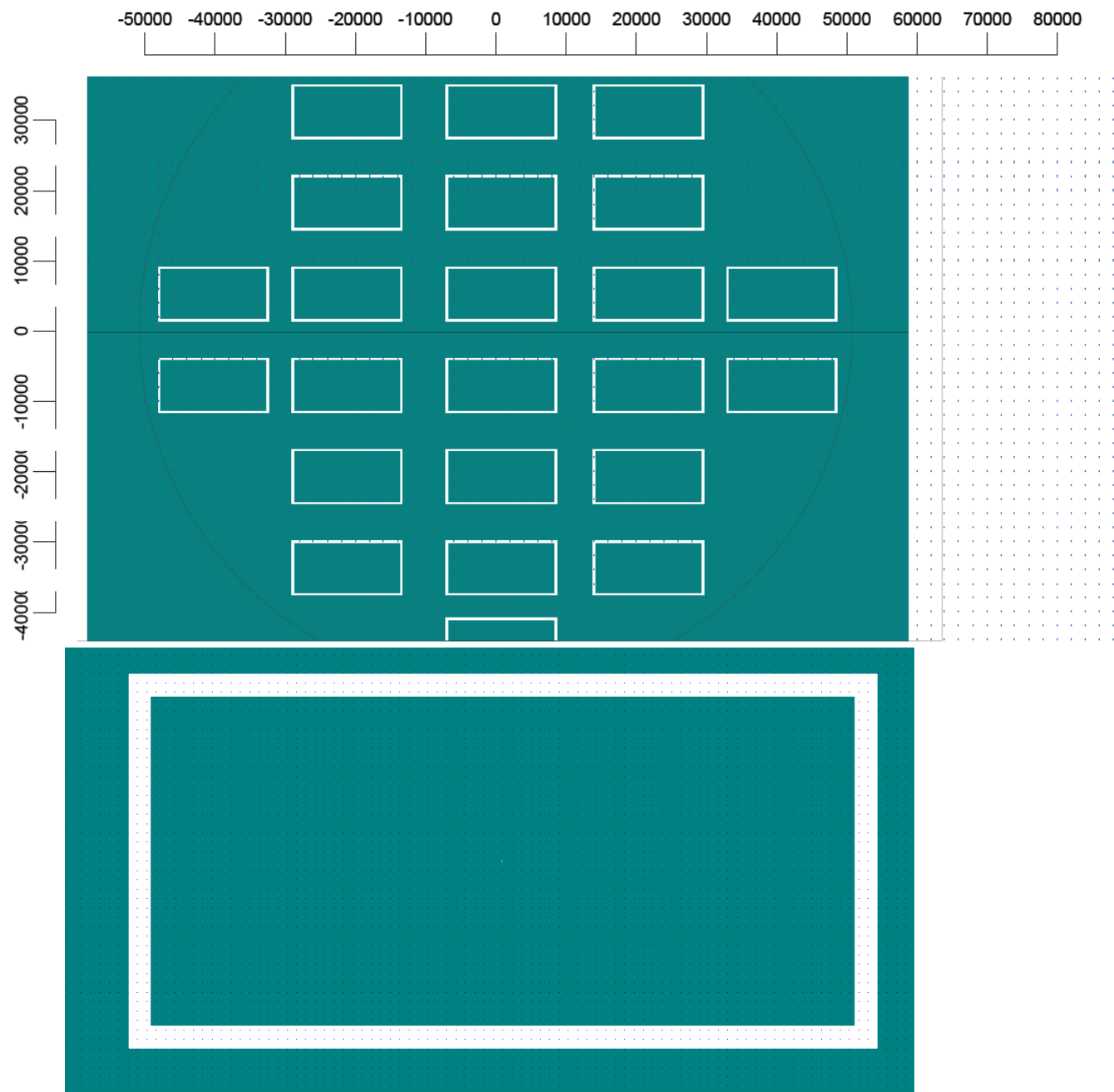


Figure C.3 Another mask for microfluidic device

