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> Central Florida

A CUSTOMER PROGRAMMABLE MICROFLUIDIC SYSTEM

by

MIAO LIU B.S. Tsinghua University, 2000 M.S. Tsinghua University, 2002

A dissertation submitted in partial fulfillment of the requirements for the degree of Doctor Philosophy in the Department of Mechanical, Materials, and Aerospace Engineering in the College of Engineering and Computer Science at the University of Central Florida Orlando, Florida

Fall Term 2008

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ABSTRACT

Microfluidics is both a science and a technology offering great and perhaps even revolutionary capabilities to impact the society in the future. However, due to the scaling effects there are unknown phenomena and technology barriers about fluidics in microchannel, material properties in microscale and interactions with fluids are still missing. A systematic investigation has been performed aiming to develop "A Customer Programmable Microfluidic System". This innovative Polydimethylsiloxane (PDMS)-based microfluidic system provides a bio-compatible platform for bio-analysis systems such as Lab-on-achip, micro-total-analysis system and biosensors as well as the applications such as micromirrors. The system consists of an array of microfluidic devices and each device containing a multilayer microvalve. The microvalve uses a thermal pneumatic actuation method to switch and/or control the fluid flow in the integrated microchannels. It provides a means to isolate samples of interest and channel them from one location of the system to another based on needs of realizing the customers' desired functions.

Along with the fluid flow control properties, the system was developed and tested as an array of micromirrors. An aluminum layer is embedded into the PDMS membrane. The metal was patterned as a network to increase the reflectivity of the membrane, which inherits the deformation of the membrane as a mirror. The deformable mirror is a key element in the adaptive optics. The proposed system utilizes the extraordinary flexibility of PDMS and the addressable control to manipulate the phase of a propagating optical wave front, which in turn can increase the performance of the adaptive optics.

Polydimethylsiloxane (PDMS) has been widely used in microfabrication for microfluidic systems. However, few attentions were paid in the past to mechanical properties of PDMS. Importantly there is no report on influences of microfabrication processes which normally involve chemical reactors and biologically reaction processes. A comprehensive study was made in this work to study fundamental issues such as scaling law effects on PDMS properties, chemical emersion and temperature effects on mechanical properties of PDMS, PDMS compositions and resultant properties, as well as bonding strength, etc. Results achieved from this work will provide foundation of future developments of microfluidics utilizing PDMS. This dissertation is dedicated to my parents, Weiping Liu and Yuqi Zhu, for always loving me, believing in me, inspiring me and encouraging me to be a strong woman in the engineering field and reach higher to achieve my dream.

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CHAPTER 1 INTRODUCTION

1.1 Background for the Study

Since the first research in microfluidics was carried out in $1977^{[1]}$, the research in this area has undergone an enormous growth and many different microfluidic devices have been developed. Microfluidic devices assure the manipulation of liquids and gases in channels having cross-sectional dimensions on the order of $10 \sim 100 \mu m$. Microfluidic systems comprising of micronozzles, microchannels, micropumps, micromixers, and microvalves, etc., can be used for a variety of applications (Table 1) involving general manipulation of liquid, gases and their mixtures. As such, microfluidics is an important technology in a number of miniaturized systems that are being developed for chemical, biological and medical applications, and even systems for fundamental research.

Compared to conventional macro-scale fluidic systems, microfluidic devices and systems have a number of significant advantages such as: enhanced performances in reaction/detection, low cost, compact size, disposability, smaller volumes of reagents or samples and low energy consumption. For example, microfluidic channels can approximate the size and flow condition found in invivo capillaries around 10 μ m with 0.1 cm/s. The use of research and diagnostic devices of with similar sizes and elasticity as those found in biology could lead to more accurate information and greater understanding of physiology.

1

Area	Application
Miniaturized analytical system.	Rapid, high density sequencing, DNA fingerprinting, combinatorial analysis,
Genomics and proteomics	forensics, gene expression assays, integration of fluidic with DNA array
Chemical/biological warfare	Early detection and identification of pathogens
defense	and toxins; early diagnosis triage
Clinical analysis	Rapid analysis of blood and bodily fluids, point of care diagnostics based on immunological or enzymatic assays, electrochemical detection, cell counting
High throughput screening	Combinatorial synthesis and assaying for drugs, toxicological assays
Environmental testing	In situ analysis of environmental contamination
Biomedical/Implantable	Devices for in vivo drug delivery, in vivo
devices	monitoring for disease and conditions
Tools for chemistry and biochemistry	Rapid reactor
Small-scale organic synthesis	Combinatorial synthesis
Sample preparation	Purification of biological samples for analysis
Amplification of nucleic acids/sequences	PCR, RT-PCR
System for fundamental research	Microscale plasma
Systems with which to study	Studies on EOF and laminar flow in small
the flow of fluids	channels and diffusion
Studies of chemical reactions	Enzyme-substrate
Biomimetic systems	Development of machines at mimic biological functions
Systems to study small amounts of sample	Detection of single molecules

Table 1 Potential applications of microfluidic systems

In addition, smaller channels increase resolution while decreasing the overall size of the device, but small channels also make detection a more demanding process. At the same time they are more susceptible to the blockages from particles and are more sensitive to the adsorption of species on the surfaces. The proper handling of small amount of liquids with volumes in the microliter and the nanoliter range has become extremely important with the development of μ TAS (micro-total-analysis-system) and Lab-on-chip. These technologies have been applied in a variety of areas including separation science, chemical synthesis and immunoassays, DNA amplification and sequencing, protein separations and single-cell analysis. Microfluidic systems have been developed to conduct a wide range of DNA analyses. Polymerase chain reactions (PCR) have been successfully carried out within integrated microfluidic systems. DNA sequencing and genotyping are achieved using microchannels and capillary array electrophoresis. Microfluidics technology has also illustrated a potential to be allied with the detection of very low numbers of DNA molecules. Single molecule detection has been achieved in a Polydimethylsiloxane (PDMS) channel using near field optics^[2].

Miniaturized microfluidic systems require the ability to control precisely and efficiently the transport of reagents and samples throughout the different parts of the system. Most of these systems are developed with bulk and surface micromachining processes using microfabrication techniques on silicon, glass or polymer substrates.

A great variety of microchip fabrications and materials are used for producing highly sophisticated two- and three-dimensional microstructures with integrated modules. The earliest microfluidic systems were fabricated in silicon and glass to take advantages of the technology already existing in microelectronics and microelectromechanical system (MEMS). Although Silicon and glass are attractive materials for fabricating microfluidic devices, polymers also have unique characteristics. Polymers are less expensive than silicon and glass and involve simpler and less expensive manufacturing processes. Lower production costs also mean that single-use devices, which eliminate contamination between analyses, are more feasible in applications. Examples of polymers used to fabricate microfluidic systems include: polyurethane ^[3], polycarbonate ^[4, 5], polymethylmethacrylate (PMMA) ^[6-8], polystyrene^[9], polyethyleneterephthalate glycol (PETG) ^[10], polyethylene ^[11] and Polydimethylsiloxane (PDMS) ^[12-15].

Soft polymers (elastomers) have additional advantages for microfluidic device fabrication. Particularly, PDMS is a soft polymer that is being actively used in both academic and industrial research groups for applications in microfluidics. PDMS devices are fabricated by soft lithography, a technique that allows rapid prototyping of microfluidic devices. Soft lithography is particularly useful as a non-photolithographic technique for pattern replication to enable rapid prototyping of devices. Devices made of PDMS can be easily integrated with outside components because the polymer conforms to most materials and both reversible and irreversible sealing are possible. This polymer is attractive for applications requiring temperature gradients since it is stable at temperatures necessary for processing biological substrates. PDMS is also bio-compatible with many optical detection methods because it is transparent in the visible/UV region. PDMS channels are appropriate for cellular studies because PDMS is nontoxic to proteins and cells. These good characteristics have made it the key material for exploratory research and research engineering at the early stages in the development of microfluidic systems. Two particularly important achievements have been the development of soft lithography in PDMS as a method for fabricating prototype devices and the development of a simple method of fabricating pneumatically activated valves, mixers and pumps on the basis of softlithographic procedures. These methods have made it possible to fabricate devices that test new ideas with a faster pace using silicon technology. Quake's pneumatic valves^[16] are particularly important as components that have enabled the design and examination of complicated devices and these have opened up a number of areas of applications. "Quake valves" use the restriction of a fluidic channel by an adjacent channel under pressure; their operation depends on the fact that PDMS is an elastomer and no corresponding devices exist in rigid materials such as silicon and glass. And also, microfluidic systems are studied by Janasek et al to exploit the scaling law that relate macroscopic and microfluidic systems with special emphasis on lab-on-a-chip devices^[17].

While microelectronics emphasis component size reduction, microfluidics research is focused on making more complex system with better fluid-handling capabilities using macro system components such as pumps valves mixers, filters and separators at microscale. With this possibility of integration, the ultimate goal for microfluidic system is to be able to detect biological molecules, transport, mix and characterize a raw sample, all with one device. The domain of this kind of integrated analysis systems has been designated as μ TAS or also lab-on-chip systems. In 1994, S. Jacobson ^[18] first fabricated a chip integrating three different functions: mixing, reaction and separation. This achievement was just the

beginning of integrated microfluidic system. Four years later, Science published a single device^[19] capable of titrating aqueous solutes and then performing the mixing, amplification, enzymatic digestion, electrophoretic separation, and detection. During the last few years researchers have come up with all sorts of solutions to improve and simplify the manipulation of fluids on-chip. In the meantime, lab-on-chip devices accomplishing a small number of functions have already been commercialized. For example, the Biosite has commercialized systems (as in

Figure 1) that can diagnose whether the patient had suffered a heart attack in just 15 minutes.



Figure 1 Biosite Triage System

Though this system is not completely integrated because a computer is required to analyze the data produced, it successfully demonstrates the excellence of the microfluidic system's portability. It is clear that there is still an enormous amount of progress to be made in this domain, which is precisely one of the tasks allocated to microfluidics. The development of microfluidic systems has just begun. The extension of microfluidic systems into nanofludics, in which the dimensions of the channels and thickness of the layer of structured fluid at the walls of the device become comparable, will make possible the exploration of the properties of near-surface water and of ion and electrolyte transport at this interface. The biocompatibility of PDMS suggests that it might ultimately be possible to embed microfluidic devices in vivo for certain types of biomedically relevant analysis.

Microfluidics is both a science and a technology. It offers great, perhaps even revolutionary, new capabilities for the future. However the fundamentals of the field are very strong: much of the world's technology requires the manipulation of fluids, and extending those manipulations to small volumes, with precise dynamic control over concentrations is a huge step. Discovering and exploiting new phenomena occurring in fluids at the microscale level is vital to the advancement of this field

Microelectromechanical systems (MEMS) have enabled a wide range of sensors and actuators to be realized by allowing non-electrical devices onto microchips. In the early years of MEMS development, fluidic components were among the first devices which were realized in microscale using silicon technology. The most common components were flow sensors, microvalves and micropumps. Research on the systems consisting of a combination of these devices and methods has progressed rapidly in recent decades and form the nature of microfluidic systems. In this combination, each element of the system is miniaturized by micromachining and is integrated on a substrate. This process reduces the system size, cost, and dead volume. The sample volumes and the amount of reagents needed for measurements are also significantly reduced so that the measurement time may be reduced and the power consumption for the entire system can be decreased. With the growing importance of genomics, proteomics, and the discovery of new drugs, microfluidics expanded to the development of numerous micro devices: filters, mixers, reactors, separators. New effects such as electro-kinetic effects, acoustic streaming, magnetohydrodynamic effects, electrochemical, and more, which were previously neglected in macroscopic applications, have now gained new importance in microscale systems.

1.2 Microfluidic System

1.2.1 Recent Development of Microfluidic System

A market study of microfluidics and MEMS devices was conducted by BCC Research^[20]. New developments in fluidics, microelectronics and detection systems have enabled microfluidics to move from theory to commercial reality in only a few years and the worldwide market for microfluidic-based systems and devices is expected to climb at an average annual growth rate (AAGR) of 15.5% to nearly \$2 billion in 2008, from \$950 million in 2003. Remarkable growth in the areas of genomics, high-throughput screening of drug targets, and lead compounds has sparked interest in microfluidics technology among healthcare businesses. Its broad range of solutions supports numerous genomics-based drug discoveries. Other significant applications for microfluidics include clinical diagnostics, proteomics, glycomics, cell manipulation, tissue engineering, structural biology, combinatorial chemistry, environmental monitoring, and drug delivery. The popularity of miniaturization is expected to fuel the market. The extraordinary ability to also shrink sample volumes down to the microscale is expected to make microfluidic systems attractive to the biomedical industry.



Figure 2 Market for Microfluidic Technologies, 1997-2008 (\$ Millions)

Microfluidic systems have the tremendous potential both in fundamental research fields and everyday applications such as inkjet printers, blood-cell-separation equipment, biochemical assays, chemical synthesis, genetic analysis and drug screening. With the development in fabrication and MEMS materials, microfluidic systems are now rapidly evolving towards their ultimate goal: fast, portable, robust, flexible, inexpensive and easy-to-use all-add-on systems which is one system to provide all of the possible required analyses for a given type problem. And this leads to the concept of "lab-on-chip" or μ TAS. Synthesis, purification, analysis and diagnostics would be performed by such a system rapidly, economically and with minute volumes of samples. The microfluidic systems

connected to liquid reservoirs by tubing systems in turn linked to syringes. The channel measures in the range of a few micrometers, which greatly facilitates the handling of volumes much smaller than a microliter. Appropriate channel design and integrated modules such as electrodes or a specific pattern are now facilitating the incorporation of many operational steps. As microfluidic systems continue to evolve, it will be very important to have portable application codes that can run without modification on successive generations of chips. Since the first μ TAS, the micro gas chromatograph (GC) created by a group from Stanford University in the late 1970s, the development of μ TAS lead researchers to the realization of a number of successive operations such as blood gas analysis, glucose/lactate analysis, DNA analysis, proteomics analysis, combinatorial synthesis analysis, immunoassays, toxicity monitoring and even forensic analysis applications.

1.2.2 Components of Microfluidic System

A microfluidic system must have a series of generic components: a method of introducing reagents and samples (probably as fluids or gas or even particles), methods for moving these fluids around on the chip and for combining and mixing them and various other devices such as detectors for systems used in synthesis. Microfluidics has been noticed of the rapid development of new methods of fabrication, and of the components – the microchannels that serve as pipes, and other structures that form valves, mixers and pumps – that are essential elements of microfluidic systems.

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1.2.2.1 Micropumps and Microvalves

Among the most important fluid-handling elements are micropumps. Micropumps are devices that set micro scale fluids into motion while microvalves are designed to control this motion and define a preferred direction for the motion of the fluid. Micropumps and microvalves are always integrated together for full function and this research is one of the biggest fields in microfluidic systems.

Micropumps can generally be categorized into two groups (as in

Figure 3): mechanical and non-mechanical (without moving part). At least there are three kinds of mechanical micropumps developed: peristaltic, reciprocating and rotary pumps. On the other side, the non-mechanical micropumps have electro hydrodynamic, electro osmotic and ultrasonic micropumps. A breakdown of some of the micropumps developed so far and their performance is shown in Figure 4.



Figure 3 Micropumps categories by their pumping principles



Figure 4 Review of micropump technologies Green represents mechanical micropumps, and pink non-mechanical

1

Actuation method	Pressure	Displacement	Response time
Disk type PZT	Small	Medium	Fast
Stack type PZT	Very large	Very small	Fast
Pneumatic	Small	Large	Slow
Shape Memory Alloy	Large	Large	Slow
Electrostatics	Small	Very small	Very fast
Thermo-pneumatic	Large	Medium	Medium

To date, the reciprocating displacement micropump has been the most popular choice for researchers. This type of micropump generally comprises of a pressure chamber bounded by a flexible diaphragm driven by an actuator and passive or dynamic microvalves. The check valves and the actuator play very important roles in the flow rate and the maximum output pressure. Many types of actuators such as piezoelectric, pneumatic, electrostatic and thermo-pneumatic have been used. The performance of active valves and micropumps depend strongly on the features of the actuator. And the maximum pressure head realizable by the micropump depends directly on the available force of the actuator used (Table 2). Performance of micropump and microvalves are described as follows.

1.2.2.1.1 Piezoelectric Micropumps and Microvalves

After Smits first toyed with the concept of a silicon micropump, Van Lintel et al published their work on a silicon based micropump in 1989 ^[21]. In the paper, two pumps based on the micromaching of silicon were described. The pumps which were of the reciprocating displacement type comprise one or two pump chambers, a thick glass pump membrane actuated by piezoelectric disc and passive silicon check valves to direct the flow, as in Figure 5. The deflection of the pump chamber will cause the pump chamber pressure to either increase or decrease depending on the direction of deflection and this in turn creates a differential pressure across the valve that either opens or closes them. The sequential upwards and downwards deflection of the membrane results in a pumping effect. And this work can hold up to 2 m H₂O pressure while maintaining a maximum flow rate around 10 μ L/min.



Figure 5 Schematic of the first reciprocating displacement micropump

Soon after this, another silicon micropump was fabricated by M. Esashi in 1989 ^[22]. The micromachined device contains a normally closed microvalve and a diaphragm-type pump, which contains two polysilicon one-way valves and a diaphragm driven by a small piezoelectric actuator. The maximum pumping flow rate was 20 μ L/min and pressure 0.78m H₂O/cm².

Despite the major disadvantage that these piezoelectrically driven pumps need more than 100 V to drive, reciprocating micropumps, especially those based on piezoelectric actuation still have grown to be the dominant type of micropump and tremendous improvements have been made ^[23-26]. Among them was the first commercial micropump developed by IMT of Germany ^[27], a polycarbonate micropump fabricated using microfabrication and conventional thermoplastic replication technologies.

1.2.2.1.2 Thermopneumatic Micropumps and Microvalves

Before 1990, all the micropump prototypes developed usd piezoelectric bimorph or monomorph discs for the actuation until F.C.M. Van De Pol et al

reported thermopneumatic micropumps and valves ^[28] by adopting the idea of using a gas/liquid system and resistive heating to power a microvalve by Zdelblick and Angell ^[29]. The micropump has a sealed pressure chamber and a movable diaphragm together with a built-in aluminum microheater resistor (Figure 6). When an electrical voltage is applied to the heater resistor, the temperature inside of the chamber will rise and a related pressure will increase and introduce a downward deflection of the pump membrane. When the membrane deflects down, the pressure in the chamber will decrease. This causes a pressure difference across two valves which results in the open/close action of the valves. The pressure generated in the chamber can be estimated using Equation 1:

$$P = P_0 \exp(\frac{-L_0}{RT}) \tag{1}$$

where P_0 is the initial pressure, L_0 is the latent heat of vaporization, R is the gas constant and T is the temperature. A maximum yield and built-up pressure of 34 ml/min and 0.05 bars at 6V were reported.



Figure 6 Schematic of the first thermopneumatic micropump^[29]

In recent research, S. Zimmermann et al developed a planar micropump using a wafer-level, four-mask process, making it attractive for integration into the micro-total-analysis system ^[30]. It employs thermopneumatic actuation and two inplane flap valves (Figure 7 and Figure 8) with fluidic-resistance ratio greater than 1300 to reach maximum pressure of 16KPa and maximum flow rate of 9 μ L/min at an average power assumption of 180mW with a frequency of 10 Hz and 20% duty cycle.



Figure 7 Photo of the planar micropump utilizing two platinum hearers for thermopneumatic actuation and two in-plane flaps valves



Figure 8 Cross-sectional view of the valve seat showing that all leakage paths around the valve seat are sealed

1.2.2.1.3 Electrostatic Micropumps and Microvalves

The first electrostatic actuated micropump was developed in 1991 ^[31]. The micropump consists of two identical microvalves joined to opposite sides of a micro mechanical membrane by microchannels. The fabricated diagnostic chip consists of various test arrays. One is composed of varying valve length and microchannel width combinations, while another small test area consists of the three sizes of membranes. The operation of this pump utilizes electrostatic forces between movable parallel plate polysilicon conductors. The electrostatic forces deform thin polysilicon cantilever valves and membranes to control fluids or gases through narrow channels. Both the upper and lower electrodes are encapsulated by LPCVD Si₃Nr so that the electrodes will not electrically short when pumping conductive fluids.

Zengerle et al published another micropump which was based on electrostatic actuation ^[23]. The pump was made of four silicon layers which formed two passive cantilever valves, a pump membrane and a counter-electrode for electrostatic actuation (as in Figure 9).



Figure 9 Schematic view of an electrostatically driven diaphragm

In the case of an electrostatically driven displacement unit the pump is actuated by a square wave with an amplitude of 150 V - 200 V and frequencies of 0.1 Hz up to several hundred Hz. If the supply voltage is switched on, the pump diaphragm is driven by electrostatic forces and bends towards the counterelectrode. Thereby fluid will be sucked in through the inlet valve. When the supply voltage is turned off, the relaxation of the diaphragm pushes the fluid through the outlet valve. Two passive check valves direct the flow from inlet to outlet and so, miniaturized diaphragm pumps are known as unidirectional pumps. The outer dimensions of the electrostatically actuated diaphragm pump are 7 x 7 x 2 m³. The pump has achieved maximum pump rates of 850 μ L/min and a maximum hydrostatic back pressure of 310 hPa (310 cm H₂O) at a supply voltage of 200 V. The total power consumption of the pump element is less than 1 mW and the strong electrostatic field is completely separated from the fluid.

1.2.2.1.4 Pneumatic Micropumps and Microvalves

Pneumatic actuation is not very popular due to its relative complexity in terms of design and fabrication. However, Rapp et al's research does need to be mention here ^[32]. By combining the LIGA technique with processes used in the manufacture of micromembranes, micromembrane pumps have been built with pump chambers 100 μ m in height and with pump membrane diameters between 2 and 5 mm. The pump case is made of gold, whereas the 2.7 μ m thin pump membrane consists of titanium. The passive microvalves of the pump consist of patterned micromembranes arranged one over the other and made of titanium and

polyimide by use of thin-film techniques. The micropump has been tested so far with an external pneumatic drive. Both air and water have been pumped. A maximum flow rate of 82μ L/min and a maximum pressure of 0.47 m H₂O were obtained for pumping of air at a driving frequency of 5 Hz. The remarkable achievement of Rapp's micropumps is the utilization of thermoplastic microreplication. It embarks the way of producing microstructure components in high volume at low cost. It greatly increases the potential use of microstructures in industry.

Most of the research about pneumatic micropumps followed after Rapp's work began to take the advantage of microreplication. Though the substrates vary from silicon, glass to elastomer, most of diaphragms of the micropump design are using polymeric materials to get flexible membranes. Grosjean et al ^[33] chose Parylene and silicone rubber, Meng ^[34] et al use silicone rubber on their thermoplastic construction, and lately in Grover's design PDMS has been chosen to make the diaphragm ^[35]. Unger et al even proposed a multi-layered elastomeric microstructure which is also pneumatic driven ^[16] and this design shows the great potential of microreplication and popularize the use of elastomeric microstructures in microfluidic systems.

Other than the above micropumps, shape-memory alloys have been used to develop a flap structure in Benard's mechanical micropumps^[36] and electromagnetic actuation was involved in both Dario^[37] and Bohm's^[38] research on micropumps.
As the research about microfluidic system moves on, more and more attention is gathered on the applications in the biological and chemical analysis, which gave a hand to the development of non-mechanical micropumps. In contrast to mechanical micropumps, non-mechanical micropumps normally don't have either moving parts or valve. This simplifies the design and fabrication and brings long life cycles and minimal risks of blockage. However, this kind of pumps usually feasible only at microscale because the generated forces are generally too small to move large amount of fluids unless increasing the input to 10 kV. In addition, such pumps are restricted to certain types of pumping fluids. Examples of such pumps include electrohydrodynamic (EHD), electroosmotic (EO) and magnetohydrodynamic (MHD) pumps.

1.2.2.1.5 Electrohydrodynamic (EHD) Micropumps

Electrohydrodynamic micropumps are based on the interaction of electrostatic forces with ions in dielectric fluids. In EHD pumping, the fluid must be of low conductivity and dielectric. The electric body force density F_e that results from an applied electric field E is given by Equation 2:

$$F_e = qE + P \cdot \nabla E - \frac{1}{2}E^2 \nabla \varepsilon + \frac{1}{2}\nabla [E^2(\frac{\partial \varepsilon}{\partial p})_T \rho]$$
(2)

Where q is the charge density, ϵ the fluid permittivity, ρ the fluid density, T the fluid temperature and P is the polarization vector ^[39]. In *induction* EHD pumps, charge is induced in an inhomogeneous working fluid through the application of a potential difference across the fluid. The waves of electric fields travel perpendicular to the temperature and conductivity gradient, and induce charges in the liquid bulk. Figure 10 shows the structure Fuhr et al proposed in 1990 ^[40]. Instead of inducing charges at the interface and relying on Coulomb forces, Fuhr's device uses the dielectric force that results from the application of an electric field to a fluid that contains a permittivity gradient. The electrode array is formed on the substrate and the flow channel is formed across the electrodes. Traveling waves are produced by 90 °-phase shifted rectangular voltages of high frequency and low voltage applied along the channel direction. Flow in the range of 0.05 to 5 μ L/min was obtained.



Figure 10 Induction EHD pumps

In *conduction* EHD pumps, Coulomb forces are acting on the ions produced during the dissociation of ionizable groups at the electrode/fluid interface under a weak electric field. These Coulomb forces give rise to conduction through the bulk liquid and EHD pumps rely on this bipolar conduction. Few micropumps based on conductive EHD have been reported. EHD micropumps based on the injection of ions into the working fluid at electrodes have also been reported. For specific electrode/liquid interfaces, application of a very high electric field, greater than 100kV/cm, injects ions to into the bulk fluid. The Coulomb force acts on the injected charges; viscous interaction generates bulk flow. Richter et al reported a micromachined EHD micropump based on injection ^[41]. Driven by a 600V electric field, this micropump pumps ethyl alcohol with a maximum flow rate of 14mL/min and maximum pressure of 2.5kPa.

1.2.2.1.6 Electroosmotic (EO) Micropumps

Electroosmosis was discovered in 1809 by the Russian physicist Reuss. It is an electrokinetic effect, which can be used for pumping in small channels when the surface to volume ratio is large (as in Figure 11). Ions in the liquid form a thin electric double layer at the walls of the channel due to chemical interactions. An electric potential drop ΔV applied along the channel will exert a force on the charged double layers and the force will accelerate the ions and hence the liquid (as in Figure 12).



Figure 11 Electroosmotic flow in a channel



Figure 12 Electric double layer

EO flow is used in a wide range of applications. A number of important techniques and processes used for μ TAS incorporate EO flow including electroosmosis-based microchannel flow injection analysis ^[42], on-chip electrophoretic separation ^[43-45] and on-chip liquid chromatography ^[46]. The most basic EO pumps are simply capillaries or microchannel sections with electrodes submerged within end-channel reservoirs and a flow resistance in series with the channel. As an example, Ramsey et al applied a 350V/cm electric field to a portion of a microchannel network to produce roughly 90 nL/min flow ^[47]. An EO micropump containing a 75 µm inner diameter fused silica capillary packed with silica beads was reported by Paul et al ^[48]. This pump produces a 200 nL/min flow rate with an exceptionally high pressure around 20MPa under a 6.75kV applied voltage.

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Operation voltages and geometries of EO pumps vary widely. A detailed description of the history and development of EO pumps is presented by Yao and Santiago^[49].

1.2.2.1.7 Magnetohydrodynamic (MHD) Micropumps

Magnetohydrodynamic refers to the flow of electrically conducting liquids in electric and magnetic fields. The typical structure of a MHD pump has been drawn in Figure 13^[50]. It is comprised of microchannels with two walls bounded by electrodes to generate the electric field while the other two walls are bound by permanent magnets of opposite polarity to generate the magnetic field. Lorentz force is the pumping source in the conductive, aqueous solution in MHD micropumps in the direction perpendicular to both magnetic and electric fields. This actuation is similar to EHD micropumps.



Figure 13 Schematic diagram of the actuation principle of the MHD micropump where driving force is Lorentz force

Jang and Lee reported an MHD micropump with a 40 nm long microchannel with a hydraulic diameter around 1mm. With permanent magnets producing a magnetic flux density 0.44 T and a total current 1~100 µA, this pump can reach a maximum flow rate of 62 μ L/min with a pressure of 170Pa^[50]. Lemoff and Lee used a miniature electromagnetic coil operation at 1kHz to avoid electrolysis associated with DC citation ^[51]. A 1 M NaCl solution was pumped using this pump at a flow rate of 18 μ L/min.

Compared with other types of non-mechanical micropumps, the MHD micropump has several advantages, such as simple fabrication process, bidirectional pumping ability, and its suitability in pumping of conductive fluids which EHD and EO micropumps have limits on.

In summary, flow rate, pressure generation and overall size are important figures of merits for micropumps. The EO micropumps and PZT-driven reciprocating displacement micropumps perform well in terms of absolute flow rate and pressure generation and exhibit comparable performance. Though thermopneumatic micropumps tend to produce low flow rate, the application of microreplication make these kinds of micropumps still very hot among researchers. Given the comparatively high self-pumping frequency and small size of Zengerle's electrostatically driven micropump, further research on electrostatic actuation for micropumps is needed. Since the first micropump was introduced in the early 1980s, progress and development have been moving rapidly. As micropumps continue to improve, we can expect that micropumps will be increasingly used in a wide variety of microfluidic systems in fields including life science, semiconductors and even space exploration.

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1.2.2.2 Channels in Microfluidic System

Another important component in microfluidic systems is the microchannel. In any microfluidic system, microchannels serve as pipes in the form of either a single channel or a network. Different cross sections of microchannels (rectangular, circular, trapezoidal, triangular and elliptical) have been studied by several researchers to help understand the flow behavior inside the channels. Among them, rectangular and trapezoidal cross-sections have been extensively studied for a wide variety of applications due to practical considerations such as fabrication techniques, cost and ease of manufacturing, etc. In most of the research about different physical phenomena at the micro level, friction and pressure drop ^[52], electroviscous effect ^[53], velocity measurement ^[54], flow control^[55] and flow visualization ^[56], rectangular cross-section microchannels are employed. Some new design using trapezoidal cross-sections proposed during this microchannel study. Ross et al ^[57] choose trapezoidal shape microchannels to study sample dispersion in electroosmotic flows in plastic microchannels. The frictional and pressure drop characteristics in trapezoidal microchannels were studied by several researchers ^[58-60]. Circular cross-sections are mainly limited to heat transfer equipment such as microheatexchanger. In the mean time, halfcircular cross-section microchannels proposed in Quake's paper demonstrate a better seal for the membrane valve, which is a very critical requirement for a microfluidic system.

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A diversity of fluid properties and microfluidic device applications is translated into a wide variety of requirements in microscale flow control in microchannels. For example, the position streams within the cross-section of the microchannel for precise delivery of reagents, the mixing of solutions for chemical reactions and the transportation of small amounts of solution for high throughput synthesis and analysis are all need the support of flow control in the channels. With the technology of patterned surface charges and electrodes used for manipulating electroosmotic flow, patterned wettibility and peristalsis for control of pressure driven and patterned topography for both of these two flows, the various mechanisms implied by these technologies have been used to control dispersion, mixing and pumping, etc.

There are so many possible areas of research in the field of microfluidic. Microfluidics has emerged as a distinct new field and has attracted attention recently past due to the many applications in biotechnology. As microfluidic continue to evolve, it will be important to have portable applications that can run on successive generations of chips. And that also is the reason why the progressive miniaturization of chemical and biological instrumentation towards creating highly integrated "lab-on-chip" system or µTAS systems has always been the most exciting scientific developments of recent years.

1.3 Introduction of Deformable Micromirrors for Adaptive Optics

Adaptive optics is the control of optical wavefront phase in a real-time, closed-loop fashion. It has been mainly used in astronomical applications to

correct aberrations in the atmosphere. A typical AO system is a combination of a deformable mirror, a wavefront sensor and a real-time controller, which is used to modulate the spatial phase of the optical wavefront (as in Figure 14). The deformable mirror is a key element in a high-resolution retinal imaging system.



Figure 14 Elements of a simple adaptive optical imaging system

Although currently available macro technology, such as the mirror from Xinetics, Inc., Devans, Mass., yieled impressive results, there is still a need for a lower-cost, larger-stroke mirror. The challenge lies in producing a mirror that can exhibit up to 10-µm actuation in each of approximately 200 regions populating a mirror roughly 7-mm-dia, all at a cost of about \$1,000. Existing macro-mirrors can cost more than \$100,000, making it far too expensive to include adaptive optics in clinical instruments. Low-cost deformable mirrors are seen as essential to widespread deployment of adaptive optics-enabled instrumentations. MEMS technology would seem ideally suited to the application. From a MEMS point of view, adaptive optics is an important and challenging application that takes full advantage of the unique features of micromachined technology such as the ability to fabricate thousands of microscopic mechanical actuators and optical elements (including lenses, lasers and sensors) on a single silicon chip and the potential integration of this micromachined optical bench with control circuits and imaging sensors.

Dr. Larry J. Hornbeck at Texas Instruments first invented a micromirror which is a reflective spatial light modulator monolithically fabricated over an address circuit consisting of conventional CMOS SRAM cells ^[61]. The micromirror is actuated by electrostatic force which is generated by applying a voltage difference between the mirror plate and the electrode. Light from a projection source illuminates the micro mirror array at an angle of $\pm 2\theta L$ from the normal to the plane of mirrors in their flat state. The angle $2\theta L$ is the rotation angle of the mirror when the yoke is touching its mechanical stops, or landed. The mirror in its flat state reflects the incident light to an angle of $-2\theta L$. The projection lens is designed so that flat state light misses the pupil of the projection lens, allowing very little light to be projected through the lens. But the mirrors are only briefly at the flat state as they make a transition from one landed state to the other. When the mirror is in its off state, the reflected light is further removed from the pupil of the projection lens and even less light is collected by the projection lens. When the mirror is in its on state, the reflected light is directed into the pupil of the projection lens, and nearly all the light is collected by the projection lens and imaged to the projection screen. Because of the large rotation angles of the mirror, the off-state light and on-state light are widely separated, allowing fast projection optics to be used. The result is efficient light collection while maintaining a high contrast ratio.

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Three types of deformable micromirrors are currently being investigated: continuous face sheet mirrors backed by individual actuating elements, microfabricated membranes mirrors and segmented mirrors.

Continuous face sheet mirror backed by individual actuating elements have been demonstrated to be particularly well suited for high power laser application ^[62].



Figure 15 Schematic of Stanford continuous membrane mirror

The shape of the face sheet is controlled by a number of discrete actuators that are fixed to its back ^[62]. The shape of the mirror depends on the combination of forces applied to the face sheet, boundary conditions (the way the face sheet is fixed to the mirror) and the geometry and the material of the face sheet. These mirrors are considered to be the best, as they allow smooth wavefront control with very large - up to several thousand - degrees of freedom. The good surface quality leads to good reflectivity, which is very important for high power applications.

Microfabricated membrane mirrors have high optical efficiency and a very good potential for aberrations correction, while keeping small dimensions. The membrane can be deformed electrostatically by applying control voltages to

the electrostatic electrode actuators that can be positioned under the membrane and also over the membrane. If there are any electrodes positioned over the membrane, they should be transparent. It is possible to operate the mirror with only one group of electrodes positioned under the mirror. In this case a bias voltage should be applied to all electrodes, to make the membrane initially spherical. The membrane can move back and forth with respect to the reference sphere. These properties make them very suitable for optical fiber switching applications. A microfabricated membrane mirror has been used to optimize the coupling efficiency for an optical fiber switch for more than 3000 interconnects.

Segmented deformable mirrors are formed by independent flat mirror segments. Each segment can move a small distance back and forward (a pure piston motion) to approximate the average value of the wavefront over the patch area. Normally these mirrors have little or zero cross-talk between actuators. Stepwise approximation works poorly for smooth continuous wavefront. Sharp edges of the segments and gaps between the segments contribute to the light scattering, limiting the applications to those non-sensitive to scattered light. Considerable improvement in the approximation performance of the segmented mirror can be achieved by introducing three degrees of freedom per segment: piston tip and tilt. These mirrors require three times more actuators than piston segmented mirrors and they suffer from diffraction on the segment edges. This concept was used in the fabrication of large segmented primary mirrors of Keck telescopes. Segmented mirrors are fast, due their small size. Typically, a single pixel is 100µm aside. Their reliability, small size, light weight and ease of fabrication make them particularly well suited for free space optical communication.

Deformable micromirror was first proposed in 1986^[63] with a layer of deformable viscoelastic reflective material with electrostatically driven light valves. The principle of operation is shown in Figure 16.



Figure 16 Principle of operation of deformable viscoelastic mirror

On top of the substrate carrying the pixel electrodes there is a viscoelastic control layer covered with a mirror electrode. A bias voltage, U_0 , of typically 250V, is applied between the pixel electrodes and the mirror electrode whereby the reflective viscoelastic layer behaves like a plane mirror. Upon applying a signal voltage, U_s , of about $\pm 15V$ to neighboring pixel electrodes the viscoelastic mirror is deformed sinusoidally forming a phase grating corresponding to the active pixels. Kuck H et al fabricated deformable micromirror devices using PDMS as its viscoelastic layer ^[64].

Later research from Boston University reported their micromachined micromirror ^[65], which consists of 10 x 10 arrays of electrostatic actuators supporting a thin-film continuous or semicontinuous silicon mirror membrane via attachment post. The actuators consist of a 300 µm x 300 µm x 3 µm polysilicon membrane anchored to a substrate on two opposing sides. The schematic of the mirrors is shown in Figure 17. The deflection of each actuator is a monotonically increasing function of applied voltage and is used to control the shape of the continuous and stress-relieved mirrors. Each actuator membrane is supported over an isolated address electrode to realize addressing.



Figure 17 Schematic of micromirror with a continuous membrane

Another study about deformable micromirror is brought by Huang Chen, et al ^[66]. As shown in Figure 18, they proposed a deformable micromirror using an electroactive polymer. The complete device basically consists of 2D array of interdigitated electrode structures. The polymer is metalized with a thin reflecting layer that serves as both mirror and a counter-electrode of the addressed pixel to produce a deformation pattern. The polymer is squeezed by electrostatic force induced by the electric field between the energized address electrodes and counter-electrode. If the polymer is incompressible it will protrude into the spaces between the energized electrodes. The result is a diffraction grating effect on the energized pixel. The polymer surface of the non-energized pixel remains flat. Gray scale is achieved by varying the voltage on the address electrodes.



Figure 18 Electroactive polymer deformable micromirror

Divoux, C developed an alternative approach to make deformable mirrors for adaptive optics using magnetically driven^[67]. The mirror was a thin and flexible reflective membrane coated with a magnetic layer as shown in Figure 19. The microcoils were deposited at the opposite side of the membrane to generate local magnetic fields and deformed the membrane for a displacement of ± 5 µm.



Figure 19 Principle of the magnetic mirror

Through the efforts in the past several years, several types of high performance deformable micromirror have been developed. Yet the limitation of small displacement or deformation in current achievements restricts the growth of adaptive optics' applications. A good choice of soft material to provide larger deformation for mirrors would great benefit this demand. The flexibility of fabrication and integration of polymer materials such as PDMS reduced significantly the cost of the mirrors and combining with the advantage brought by MEMS technology, it is easy to expect that micromirrors would be as popular and in such demand as car airbag MEMS sensors.

1.4 PDMS and Soft Lithography

PDMS has been widely used in fabricating lab-on-a-chip or Micro Total Analysis Systems (μ TAS), due to its unique properties, like optically transparent, easy in fabrication, and cheap. Most important of all, its biocompatibility is critical for biological assays. PDMS is optically transparent to wavelengths close to 256nm ^[68]. This transparent nature is well suited for biological analysis using visual techniques such as fluorescence, and facilitates on- and off-chip detection. PDMS's elastic and soft nature makes it easy for reversible deformations ^[69]. μ TAS ^[70-72] system made of PDMS can be realized by molding, casting or soft lithography with high fidelity ^[73-76]. PDMS can seal to itself and other flat surface with a good watertight capability. Meanwhile, the fabrication of μ TAS or other micro-devices are cost effective, which suits the disposable nature of most biomedical applications ^[77, 78]. Based on these characteristics, PDMS appears to be highly interesting as a bio-MEMS material and the potential to utilize PDMS for a number of biochips increases^[79].

1.3.1 Introduction of PDMS

PDMS (polydimethylsiloxane) normally contains a base polymer (part A), siloxane oligomer and a curing agent (part B), siloxane cross-linker. Once mixed at a weight ratio of 10A:1B, poured over the master, and heated to elevated temperatures, the liquid mixture of these two parts at specific weight ratios will become a solid, cross-linked elastomer in a few hours. When mixing patios are controlled during fabrication, two different PDMS parts can be bonded tightly as one part without any material properties change or using conventional plasma treating method ^[77]. This PDMS specialty makes multiple layers soft lithography possible to make 3-Dimentional MEMS devices.

The chemical formula for PDMS is $(CH_3)_3SiO [SiO(CH_3)_2]_nSi(CH_3)_3$, where n is the number of repeating monomer $[SiO(CH_3)_2]$ units, as shown in Figure 20. During the curing process, the hydrosilylation reaction happens between vinyl (SiCH=CH₂) groups and hydrosilane (SiH) groups. The surface of PDMS in its natural state is hydrophobic.



Figure 20 Chemical formulas of PDMS

Table 3 gives out the major properties of GE RTV 615, a commercial PDMS used often in current research^[80].

Hardness, shore A durometer		44
Elongation	%	120
Shrinkage	%	0.2
Refractive Index		1.406
Dielectric Strength	KV/mm (V/mil)	19.7(500)
Dielectric Constant @ 1000 Hz		2.7
Dissipation Factor @ 1000 Hz		0.0006
Volume Resistivity	ohm-cm	$1.8 \ge 10^{15}$
Thermal Conductivity	°C/cm	0.00045
Coefficient of Expansion	cm/cm, °C	27 x 10 ⁻⁵
Specific Heat	cal/gm, °C	0.3

Table 3 Cured RTV 615 properties

Many PDMS-based micro-devices involve manipulating a PDMS membrane by pneumatic ^[76], thermomechanical ^[81] or hydraulic ^[78] actuation. Acting as a moving part in devices, PDMS's mechanical properties are critical regarding these micro-devices design and reliability. One of the extraordinarily interesting properties of PDMS is that two different parts of PDMS can be bonded together to form a single-material device, which is very important in bio-medical applications.

There are several ways to bond two parts of PDMS together. The easiest way to realize bonding is to apply an extremely thin layer of PDMS to one of the bonding surfaces and bring the two pieces in contact tightly. After contact, the bonded PDMS parts are put into oven or onto hotplate and cured. This method is easy to operate but is not suitable for bonding two PDMS parts with complex structures. The applied "glue" PDMS will also spread out into the structures instead of just the bonding surface. The bonding layer will also change the original thickness of the device structures, which may be a very critical requirement. Another disadvantage of this method is that, it is easy to trap air bubbles when bringing the two surfaces into contact. High pressure is required to remove the bubble between the two pieces when contacting. Oxygen plasma treatment can overcome the bubble trapping problem of the previous method. PDMS is a hydrophobic polymer for which exposure to oxygen plasma leads to oxidation and chain scission as well as cross-linking and the formation of a silicalike surface ^[77]. Oxygen plasma treatment can bond PDMS with not only itself but also glass, silicon and other flat surfaces. The downside to this method is that PDMS cannot maintain this state for more than 10 minutes and will reveal back to its natural state. This problem is made worse by the fact that the equipment cost of oxygen plasma is high. Quake et al developed another method to bond PDMS parts ^[16]. They utilized the feature that different mixing ratios can result in different properties of PDMS to realize bonding between two parts with different mixing ratios. By re-curing the two parts together under heat, two PDMS parts can be bonded into one part. This method is cost-effective and no expensive equipment is required. Also because the chemical components in the two parts are the same, the whole device will be made of a single-material, which is great for bioMEMS design.

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1.3.2 Soft Lithography

Soft lithography refers to a suite of techniques for replicating patterns of organic molecules or other materials (for example, ceramics or metals) on both planar and non-planar curved substrates. It is an alternative non-photolithographic set of micro fabrication. For most applications, soft lithography uses mechanical processes to transfer organic material by physical contact between a topographically patterned stamp or mold and a substrate. The mechanisms for pattern transfer (molding, embossing, and printing) more closely resemble the methods used for bulk manufacturing than they are to those used commonly in fabricating microelectronic devices (for example, photolithography or electronbeam lithography, where beams of light or beams of electrons write patterns in polymeric materials). The term "soft" originally came from the field of physics where organic materials are known as soft matter. The process can be carried out in ambient laboratory conditions. Therefore expensive cleanroom facilities are not a must to fabricate in the size range of $20 \sim 100 \,\mu\text{m}$ which is the most relevant to microfluidic systems used in µTAS. In most studies, PDMS "stamps" are used with patterned structures on the surface to generate features. The stamps can be prepared by casting prepolymers against masters patterned by conventional lithographic techniques, then cured and peeled off.

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Figure 21 PDMS in soft lithography

There are six methods developed that are regarded as soft lithography methods: microcontact printing (μ CP), microtransfer molding (μ TM), micromolding in capillaries (MIMIC), replica molding (REM), solvent-assisted micromolding (SAMIM) and phase-shift photolithography. Cast molding, embossing and injection molding are also developed as part of this technology.

 μ CP is similar to the mass printing of newspaper. An "ink" of alkanethiols is spread on a patterned PDMS stamp. The stamp is then brought into contact with the substrate. The substrate can range from coinage metals to oxide layers. The thiol ink is transferred to the substrate where it forms a self-assembled monolayer that can act as a resist against etching. μ CP is oftern used to transfer selfassembled surfactant monolayers SAMs from an elastomeric mold to a substrate ^[82].



Figure 22 Microcontact printing

 μ TM is able to generate multilayer structures. In μ TM, a PDMS stamp is filled with a prepolymers or ceramic precursor and placed on a substrate. The material is cured and the stamp is removed. In 1996, Zhao et al successfully generate features as small as 250 nm using μ TM technology^[83].



Figure 23 Microtransfer molding

As shown in Figure 24, micromolding in capillary (MIMIC) is a single step process. Continuous channels are formed when a PDMS stamp is brought into conformal contact with a solid substrate. Capillary force spontaneously fills the channels with a polymer precursor and then once cured, the PDMS stamp is removed. Micromolding was performed on a variety of substrates, including (100) oriented silicon wafers, 100 nm thick Pt-coated (100) silicon wafers, stainless steel plates, and silver and platinum foils. In applications requiring submicron scale features, MIMIC provides a clear advantage over the μ CP and channelstamping techniques. Further, since the capillaries can be used as microreactors, this method has also been used to impose a hierarchy of structures on thin solid films by combining self-assembly surfactant for templating nanoscale channels with silica. MIMIC has recently been used to generate patterned microstructures of ZrO₂ and SnO₂ from their polymeric precursors^[84].



Figure 24 Micromolding in capillaries

Replica molding (REM) uses polyurethane (PU) together with a PDMS stamp to realize patterning. A PDMS stamp is made by cast on a conventionally patterned master. PU is then molded again with this secondary PDMS stamp master. After removal of the PDMS master, a PU copy is easily obtained. REM's advantage relies on the fact that it can make multiple copies without damaging the original master mold. The minimum feature size fabricate using this technique is as small as 30 nm ^[85].



Figure 25 Replica molding

In solvent-assisted micromolding (SAMIM), a polymer film is coated onto a substrate and baked, similar to photoresists. An elastomeric mold with relief patterns, usually made of PDMS, is wetted with a good solvent for the polymer, and brought into contact with the polymer surface. The solvent on the mold dissolves the polymer yielding the channels or cavities formed between the mold and the substrate. Figure 26 shows the pattern developed by Luan et al using SAMIM technique ^[86].A 60nm feature size has been reported by Kim et al ^[87].



Figure 26 Solvent-assisted micromoldings

In phase-shift photolithography, a transparent PDMS phase mask with relief structures on its surface is placed in conformal contact with a layer of photoresist. Light passing through the stamps is modulated in the near-field. If the relief on the surface of the stamp shifts the phase of light by an odd multiple, a node in the intensity is produced. Features with dimensions between 40 and 100 nm are produced in the photoresist at each phase edge ^[88]. This technique is best suited for the fabrication of narrow electrode gap SAW devices that are designed for the efficient SAW excitation at higher harmonics. The combination of near-field phase shift lithography with narrow-gap SAW designs thus opens up a way for simple and low-cost SAW devices operating above 5 GHz ^[89].



Figure 27 Phase-shift photolithography

Soft lithography appears to be a promising route to micro- or even nanostructures in µTAS. Combining advanced microfabrication techniques, soft lithography can easily transfer structures into organic polymers or other materials. It provides researchers a convenient, effective and inexpensive way to make µTAS with good biocompatibility. Soft lithography's advantages, patterning on non-polar surfaces, patterning of solid materials other than photoresists, patterning over large area and three dimensional fabrications, will make this technique become extremely competitive and widely used.

Considering PDMS's many advantages, including low costs, short fabrication time, biocompatibility, transparency, and combining the feasibility of making devices using soft lithography, this technology is currently moving forward rapidly and PDMS-based microfluidic systems are indeed a growing area of interests.

CHAPTER 2 PROGRAMMABLE MICROFLUIDIC SYSTEM

2.1 Introduction

Advances in microfluidics technology are revolutionizing molecular biology procedures for enzymatic analysis, DNA analysis (e.g., Polymerase chain reaction and sequencing), and proteomics. The basic idea of microfluidic biochips is to integrate assay operations such as detection, as well as sample pre-treatment and preparation on one chip. The microfluidic systems on the "chip" perform as small platforms comprising channel systems connected to liquid reservoirs by tubing systems linked to syringes. The channel measures in the range of a few micrometers, which greatly facilitates the handling of volumes much smaller than a microliter. As microfluidic systems continue to evolve, it will be important to have portable application codes that can run without modification on successive generations of chips.

Though restricted by the traditional IC fabrication technology, siliconbased microfluidic systems reveal their disadvantages like high cost and nonbiocompatibility to those bio-applications with the rising demand of bio-analysis. Microfluidic research is focusing on the fabrication of a low cost and biocompatible microfluidic system. PDMS stepped on this stage in the 1990's. Its great elastomeric properties and simplicity in fabrication enable the expanding of microfluidic systems' application field and bring more and more multidisciplinary integrations as a total analysis system. Integrated with polymeric soft lithography, the potential advances of PDMS-based microfluidic systems are huge: mass produced disposable microfluidic chips that make anything from drug in vitro delivery to biosensor detection possible.

Quake et al developed a multilayer soft lithography (MSL) that enabled facile and inexpensive large scale integration of valves on a chip^[16]. It brought the possibility of integrating a thin flexible elastomeric membrane to a monolithic PDMS chip. This method greatly impacted by introducing a mechanical microvalve with a structure that can provide function as a microvalve and act as the foundation for the integrated micropumps and micromixers. A wide variety of microfluidic systems have been developed based on a similar idea. Micropumps, microvalves, micromixers, sensors and detectors are all integrated on a chip.

2.2 Design and Simulation

The proposed design develops a PDMS-based microfluidic system which can provide a universal platform for different application of microfluidic systems where microflow control is needed, no matter it is used in chemical or biological analysis. The proposed microfluidic system uses both thermal pneumatic and electrostatic actuation to realize the action and control of the microvalve and flow in the microchannel. At the same time, the proposed design can be integrated into a deformable micromirror array. The system will be made mainly of PDMS, therefore, both photolithography and soft lithography will be used as fabrication methods.

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2.2.1 Design

The proposed design contains an array of single unit device; the structure is shown in Figure 28.



Figure 28 Single unit device in proposed system

The microchannel is integrated with a microvalve that contains a thin PDMS membrane and a microchamber. A microheater is fabricated on glass substrate and placed in the center of the microchamber. At the beginning, the microheater is off and the microflow can flow through the microchannel under either external applied pressure or surface tension. When the microheater is turned on by applying voltage to it, the heat generated by the microheater will boil the water trapped inside of the microchamber above. The bubble generated by this boiling effect can deform the flexible PDMS membrane integrated on the chamber. The deflection of PDMS fully blocks the microheater is cut off and another voltage is applied to the membrane and inner surface of the microchannel, where both have been treated to be electric conductive and a dielectric materials is coated to avoid a short. This newly applied voltage creates an electrostatic force which holds the PDMS membrane in its deformed state until the releasing signal is sent. Microchamber is connected to its feeding microchannel by an opening on the sidewall. After the heater is off, the pressure of the chamber drops quickly. The pressure difference between the feeding channel and the microchamber will open the flapper structure at the microchamber entrance and push the liquid into the feeding channel into microchamber and make up the loss during the heating process. Figure 29 shows the actuation process of single unit device as proposed. This design avoids the high voltage which is needed to use thermal pneumatic actuation to maintain the deflection and provides the feasibility of selective control.





An array of this proposed single unit is integrated together with multicomplex control bus to realize programmable control function, which can direct microflow in the microchannel as desired (Figure 30).



Figure 30 Integration of array and control bus

2.2.2 Basic Theory

In the proposed design, both thermal pneumatic and electrostatic actuation are used.

2.2.2.1 Thermal Pneumatic Actuation

Thermal pneumatic actuation is utilized to generate bubbles in the microchamber. This is a process which involves multi-phase change of water. The microheater provides the heat under the supply of a pulse voltage signal and the water trapped in the microchamber gains this heat and turns boiling. Water bubbles are generated during this process.

As mentioned in Christopher Earls Bernnen's *Cavitations and Bubble Dynamics* ^[90], the governing equation for transient pressure surrounding the bubble in the liquid (r>R) can be written as Equation 3:

$$P(r,t) = \rho_l \left[\left(\frac{2R}{r} - \frac{1}{2} \frac{R^4}{r^4}\right) \left(\frac{dR}{dt}\right)^2 + \frac{R^2}{r} \frac{d^2R}{dt^2} \right] + P_{\infty}$$
(3)

Where ρ_1 is the constant liquid density, *R* is the radius of the bubble at time *t*, *r* is the position vector and P_{∞} is the initial pressure of the liquid (Figure 31).



Figure 31 Semi-sphere bubble diagram

At the surface of the bubble, where the pressure is applied to the membrane, the pressure is described as Equation 4:

$$P_{b} = \rho \left[R \frac{d^{2}R}{dt^{2}} + \frac{3}{2} \left(\frac{dR}{dt} \right)^{2} \right] + \frac{2S}{R} + P_{\infty}$$
(4)

At different time, the radius of the bubble follows Equation 5:

$$R = b \frac{2\Delta T c \rho_l}{\pi L \rho_v} \sqrt{\pi \alpha t} \left[1 - \frac{q_b \sqrt{\pi \alpha t}}{2k\Delta T}\right]$$
(5)

Where *b* is a constant, α is the thermal diffusivity of the liquid and is a function of liquid thermal conductivity *k* and liquid density ρ_1 and specific heat of the liquid *c* as in Equation 6:

$$\alpha = \frac{k}{\rho_l \cdot c} \tag{6}$$

Choosing water as the simulation liquid, we have the properties as shown

in Table 4:

Critical temperature: 647.3 K Chemical formula: H_2O Critical pressure: 22,129 kPa Critical density: 351 kg/m ³ Heat flux: $10^8W/m^2$										
No.	1	2	3	4	5	6	7	8	9	
$T_{sat}(K)$	373.15	400	430	460	490	520	550	580	610	
$P_{sat}(kPa)$	101.3	247	571	1172	2185	3773	6124	9460	14044	
$\rho_1 (kg/m^3)$	958.3	937.5	910.3	879.4	844.3	803.8	756.1	697.2	619.5	
$\rho_v (kg/m^3)$	0.597	1.370	3.020	5.975	10.95	18.90	31.52	51.85	87.5	
L (kJ/kg)	2256.7	2183	2092.8	1990.4	1871.5	1731.0	1562.6	1350.3	1064.2	
c _{pl} (kJ/kg K)	4.22	4.24	4.28	4.45	4.60	4.84	5.07	5.70	8.12	
$k_l (mW/m K)$	679.0	685.7	683.3	671.3	646.0	618.3	580.9	536.6	464.0	
<i>S</i> (mN/m)	58.91	53.5	47.16	40.66	33.90	26.96	19.66	12.71	6.26	

Table 4 Properties of water vapor

The simulations of the pressure and radius of the bubble have the

following results (Figure 32):



Figure 32 Bubble radius and pressure vs. time with 10^8 W/m^2 heat flux

When the heat flux is decreased down to $2x10^7$ W/m², though the diameter of the bubble can reach approximately 150 µm theoretically, the pressure inside of the bubble decreased dramatically to 0.2MPa. It is clear that when the heat flux is smaller, the pressure get will be lower. However the time to reach the point where the pressure is relatively high and the bubble is relatively large is longer.

When the bubble is formed, the pressure in the bubble is the vapor pressure which can be simulated as the following method. The temperature T_v and pressure P_v in the bubble are assumed to be uniform and are related by an integral form of the equilibrium Calpeyron-Clausius equation

$$P_{v} = P_{atm} \exp(\frac{wQ_{vap}}{R} (\frac{1}{T_{b}} - \frac{1}{T_{v}})$$
(7)

And the equation of state for ideal gas:

$$P_{v} = \frac{\rho_{v}}{w} RT_{v}$$
(8)

where the initial condition is,

$$P_{atm} = 101.3 KPa$$
 $R_c = 8.3148 \frac{J}{mol \cdot K}$ $T_b = 373 K$

$$w = 1 \times 10^{-3} \frac{kg}{mol}$$
 $Q_{vap} = 2.26 \times 10^{6} \frac{J}{kg}$

The vapor inside of the bubbles is based on the law of ideal gas and when the temperature is below the critical temperature, the higher the vapor temperature, the higher pressure the vapor. So under the assumption that there is no mass transport between the liquid and gas interface (where the bubble surface is), once the bubble is deformed, if the temperature continues rising, the pressure will exceed the original bubble pressure.

Figure 33 shows the change of the pressure with the temperature under 10^8 W/m² heat flux.



Figure 33 Temperatures and pressures of Vapor in Bubbles

2.2.2.2 Electrostatic Actuation

Electrostatic actuation provides the force needed to maintain the deflection of the PDMS membrane to close the microchannel. As designed, both the membrane and inner surface of the microchannel have been treated to be electric conductive and a dielectric material is coated to avoid short contact. The two surfaces will act as a capacitor. To simplify the simulation, these two surfaces are treated as two parallel plates since the dielectric layer is uniform along the surface, and a parallel plate capacitor is used as simulation model. When a bias voltage is applied, an electrostatic force will be produced. Since the lower metal plate is fixed onto the substrate, the electrostatic force will act on the upper plate and cause the deformation of the upper plate. We can deem the electrostatic force like a spring force and the whole device can be modeled as shown in Figure 34.



Figure 34 Simplified Model of Parallel Metallic Electrodes

Electrostatic force in a parallel plate capacitor obeys the following basic equations (Equation 7 and 8):

$$Q = CV \tag{9}$$

$$C = \frac{\varepsilon_0 A}{g} \tag{10}$$

Where g is the gap distance between the two plates and A is the overlap area A = wx (w is the width of the plate and x is the length). The energy stored between these two plates under the voltage V is displayed in Equation 9.
$$W = \frac{1}{2}CV^{2} = \frac{1}{2}\frac{\varepsilon_{0}A}{g}V^{2}$$
(11)

Therefore the force is:
$$F = \frac{\partial W}{\partial x} = \frac{1}{2} \frac{\varepsilon_0 A}{g^2} V^2$$
 (12)

At equilibrium, electrostatic force is balanced by elastic force:

$$F_{electrostatic} = F_{elastic}$$
 $\frac{1}{2} \frac{\varepsilon_0 A}{g^2} V^2 = kz$

Where z is the biggest displacement (at equilibrium) of the upper plate and the k is the spring constant of the membrane, which leads to Equation 11:

$$z = \frac{\varepsilon_0 A}{2kg^2} V^2 \tag{13}$$

At the same time, when electrostatic force is applied to the upper metal plate, which is covered by a layer of PDMS, the deformation caused by the electrostatic force is expressed as Equation 12:

$$\Delta t = \frac{Ft}{AE} \tag{14}$$

If the PDMS membrane with electrodes is around 20 μ m thick and the dielectric constant layer uses 2 μ m thick parylene, the voltage needed between the two electrodes will satisfy Equation 13:

$$V_{need} = \sqrt{\frac{2 \cdot w_{\text{max}} \cdot E \cdot h^3 \cdot d^2}{3.15 \cdot \varepsilon_0 \cdot 0.0138 \cdot a^4}}$$
(15)

Where w_{max} is the maximum deflection of the PDMS membrane and $w_{max}=50 \ \mu m$ and equals to the radius of the proposed microchannel, *E* is Young's modulus of PDMS membrane, *h* is the thickness of the membrane, *d* is the gap caused by the dielectric layer which is the thickness of parylene layer and *a* is the side length of the square membrane which is 200 μ m. Using these values in, the voltage needed applied to the two electrodes to hold a 50 μ m deflection of a PDMS membrane is 50V.

2.2.2.3 Conclusion

As mentioned, the governing equation for transient pressure surrounding the bubble in the liquid (*r*>*R*) is $P(r,t) = \rho_l [(\frac{2R}{r} - \frac{1}{2}\frac{R^4}{r^4})(\frac{dR}{dt})^2 + \frac{R^2}{r}\frac{d^2R}{dt^2}] + P_{\infty}$. Simultaneously, the radius change of the bubble versus time follows (Equation 5) $R = b \frac{2\Delta T c \rho_l}{\pi L \rho_v} \sqrt{\pi \alpha t} [1 - \frac{q_b \sqrt{\pi \alpha t}}{2k\Delta T}]$. The simulation result shows that when the heat

flux is smaller, the pressure get will be smaller but the time to reach the point where the pressure is relatively high and the bubble is relatively large is longer. The heat flux can be located at a value of $5 \sim 8 \times 10^7 \text{ W/m}^2$ to get a better actuation. Figure 32 shows that the pressure drops down to normal in about 4 µsec, which means that if the pressure is used as an actuation force, only the early time of the bubble growth period can be used and the response frequency can be around 1 MHz and the driven parts should be as close as possible to get a better actuation.

Based on simulation result, optimized parameters of each components in the system are chosen. To generate a heat flux of $5 \sim 8 \times 10^7 \text{ W/m}^2$, the resistance of the microheaters are set at 200~300 Ω . The length of each microchamber in the device is 200 µm. Therefore the turn on voltage for the microheaters to generate the desired heat flux is approximately 25V. A 20 µ thick PDMS membrane can be achieved by spinning. A 2 µm thick parylene layer is used as the dielectric layer between the PDMS membrane and the channel electrode. A 50V voltage is needed to hold the deformation of the PDMS membrane and close the microchannel.

2.3 Fabrication

The fabrication of single unit device contains three parts: fabrication of microheater and microchamber, fabrication of microchannel and bonding of the two parts fabricated.

2.3.1 Fabrication of Microheater and Microchamber

As shown in Figure 35, the fabrication involves seven steps. Lift off is used to fabricate microheaters on a glass substrate. Thickness of the metal is controlled to guarantee the resistance of the pattern around $200~300\Omega$. A thin sacrificial layer is spun on and patterned in order to release flapper structure. Double spin method is used to make the opening on the SU-8 wall to open the chamber to the feeding channel. Thick positive photoresist AZ 4620 then covers the hold surface and later is patterned with the mask of the chamber shape and flapper shape. Well-mixed PDMS is spun on at 1500 rpm to generate the membrane and the main chamber body. After heat-curing, AZ 4620 and sacrificial layer are removed in Acetone bath and this action will release the chamber and flapper as designed.



Figure 35 Fabrication process of microheater and microchamber

Before the integration of this unit and the microchannel, a layer of conductive material is needed to deposit on the PDMS membrane, which either conductive PDMS or a spiral shape electrode is fabricated onto PDMS membrane. Due to the fact that conductive PDMS is not transparent and hard to fabricate on, the latter one is more preferred. The reason for the spiral shape is that this structure can add more flexibility to hard material, like metal, while under deformation, as shown in Figure 36. This spiral shape structure can be made by deposit metal or ITO onto O_2 plasma etched membrane.







2.3.2 Fabrication of Microchannel

Figure 37 Fabrication of microchannel

The fabrication of the microchannel uses the technology developed by Quake et al. Thick photoresist AZ 4620 is patterned and then reflowed at 130 °C. Reflowed photoresist will hold its round shape due to the surface tension effect of liquid and after cooling down. The round shape can then be transferred to the PDMS as shown in Figure 37. In order to make the inner surface of the channel to be conductive, a thin layer of ITO is deposited on to it. A 2 μ m thick parylene film is then used to cover this conductive area in order to avoid short contact with the conductive material on top of the membrane.

2.3.3 Bonding between Microchamber and Microchannel

Because the bonding surfaces of two parts are all PDMS, two methods are performed to bond them together.

The first method is used in conventional fabrication of PDMS. Both PDMS surfaces are treated under O_2 Plasma for about 20~30 seconds and then forcibly pressed together for about 2 hours for full bonding. The oxygen plasma introduces hydroxyl groups on the surface of the materials that are used to make them bond irreversibly to each other.

The second method is developed by Quake's group. They utilized the feature that different mixing ratio can result in different properties of PDMS to realize bonding between two parts that contain different exceeding components. The two parts can be bonded together into one part under heat. By choosing the microchannel to be base-over part and the PDMS membrane to be crosslinker-over part and re-curing them together, the proposed structure can be easily realized.

2.4 Test Result and Discussion

2.4.1 Test Method Setup

In order to observe the working status of the fabricated device, a real-time monitoring system is set up as shown in Figure 38.



Figure 38 Monitoring system setup

The computer controlled power supply provides a duty ratio adjustable square wave signal to drive the microheater. The deformation of the PDMS membrane is then captured by a digital camera connected to a microscope and a computer. LabView ® is used to record the video and save it on the computer.

A second monitoring system can be setup to measure the deflection of PDMS membrane. As shown in Figure 39, Keyence LC-C1A Laser Position Sensor is connected to a computer through RS 232 port. The deflection of the membrane can be detected by the laser and the change will be digitally transferred to the computer.



Figure 39 Measuring system setup

2.4.2 Test Results and Discussion

Since the majority of the device is made of PDMS, soft lithography is used as the major fabrication method.

Figure 40 shows the fabricated microheater and microchamber.



Figure 40 Microheater and microchamber

The microheater is made of Au by a lift off process with a resistance of 320Ω . A 50% duty ratio square wave with a magnitude of 5V is applied to the two pads through needle contact and Figure 41 shows the reaction process recorded.



Figure 41 Testing result of deformed membrane under 5V actuation

Figure 41 (a) shows the initial status of the device. The PDMS remained flat when there was no power added. When a 5V voltage was applied, the bubble began to form on the "turned on" microheater and grew larger as the heating continued (Figure 41 (b) and (c)). After the bubble grew large enough and filled the whole chamber, the pressure of the bubble began to push the PDMS membrane up, as shown in Figure 41 (d). This deformation is confirmed by the simulation in I-DEAS using the same parameters (Figure 42).



Figure 42 Comparison of experimental and simulated deformation of PDMS membrane

The flapper design in the preliminary result is not working as desired. The flapper is too small and becomes very stiff. The pressure inside of the chamber is not strong enough to push it enough to close tightly. We experienced leaking due to this problem. New design is then proposed to avoid this problem by releasing more degrees of freedoms of the flappers. Only one degree of freedom will be kept by the flapper. The structure is connected to the PDMS membrane as a "portiere" close to the microchamber entrance. Except this connection, all the other edges of the flapper will be set free.

Plasma etching is employed as the fabrication method for the electrodes on the membrane. In Figure 43, a photoresist pattern is fabricated with the spiral shape. And before this shape is etched on the PDMS, a basic PDMS plasma etching with SF₆ atmosphere is conducted at University of Florida. The plasma etching lasts 15 minutes at 600W with an SF₆ flow of 3sccm and O₂ 50sccm.As discovered in the SEM picture (Figure 44), the slope surface suggests that the etching speed of the PDMS and the photoresist (AZ 4620) has a ratio of 1:3. This means that in order to get a 2 μ m etching depth on PDMS, the thickness of the photoresist covering the spaces between the circles inside the spiral shape as well as any other areas that must be protected from the plasma etching process should be at least 6 μ m.



Figure 43 Spiral shape photoresist pattern



Figure 44 SEM of O_2 plasma etched PDMS surfaces

Except for the fabrication and test above, microchannels were also made as proposed. As shown in Figure 45, PDMS microchannels are made by re-flow method developed by Quake et al.



Figure 45 PDMS microchannel fabricated using re-flow method

Though bonding between the microchannel and the microchamber hasn't yet to be performed, the bonding experiment between two PDMS parts with different mixing ratio has been done and groups with different mixing ratios have been tested. Details can be referred to in chapter 4. Figure 46 demonstrates an example of the bonding boundary. The lower part contains 3 parts of base and 1

part of agent (3A:1B). It is bonded to the upper part whose mixing ratio is 30A:1B. This bonding has a bonding strength around 130 KPa. And from Figure 46 we can see that the two surfaces are bonded pretty well into one whole piece of PDMS.



Figure 46 Bonding effect between PDMS parts with different mixing ratios

Though the final device has yet to be fully assembled, the basic fabrication steps have been detailed established. The simplified prototype of the microfluidic system also revealed the problems existing in the current design and a new revised design has been made based on the analysis of these results.

2.5 Conclusion and Outlook

Many researchers have described various kinds of microfluidic systems. Recently, more and more interests are rising in the development of microfluidic systems for applications in medical or biological analysis. A microsystem that can be used in these applications often requires good bio-compatibility which cannot be easily satisfied with traditional silicon-based microfluidic system. Also, the latter systems restricted by its traditional fabrication methods reveal a disadvantage of high cost. The demand is high to fabricate a low cost and biocompatible microfluidic system for microfluidic research. PDMS distinguishes itself from other MEMS materials by its great elastomeric properties and simplicity in fabrication, low-cost and good biocompatibility. Soft lithography enables the expanding of PDMS-based microfluidic systems' application field and brings more and more multidisciplinary integrations as a total analysis system.

In the work of this chapter, a PDMS-based customer programmable microfluidic system has been developed. A prototype of the system is tested with a 5V voltage for actuation. Soft lithography is employed in the fabrication process for this microfluidic system. Thick positive photoresist AZ 4620 was used as a sacrificial layer to form the reaction chamber. Different ratio PDMS bonding was tested with the consideration of final integration of the system.

This microfluidic system provides a universal platform for different application of microfluidic systems where microflow control is needed, whether it is used in chemical or biological analysis. The major components of the system are made from PDMS which assures the good biocompatibility of the whole system. The applications for this microfluidic system are not restricted to microfluidics only. The good deformability of the PDMS membrane in the system provides even more possibility for it to be adopted into other applications such as micromirror arrays.

The developed systems showed a noticeable deformation of the PDMS membrane. Yet the characterization of this deformability of PDMS needs further study into the mechanical behavior at a microscale which is discussed in the following chapter.

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CHAPTER 3 MICROMIRROR ARRAYS

3.1 Introduction

The ideal of deformable micromirror was first proposed in 1986 ^[63] with a layer of deformable viscoelastic reflective material with electrostatically driven. Through the efforts of the past few years, several types of high performance deformable micromirror have been developed. Most of the micromirrors usually are voltage-driven and provide the potential of integration with independent pixel control through different control circuits. However, with the use of traditional microfabrication, these families of micromirror are limited from most applications because of their prohibitively high cost introduced by the fabrication of complicated structures. At the same time, even though these actuation methods are well developed; limitations in performance, size and complexity in their manufacturing restrain their practical applications. The flexibility of fabrication and integration of polymer materials especially PDMS which is optically transparent at a wide range of wavelengths has significantly reduced the cost of the mirrors and combined with the advantage brought by MEMS technology. It has been used to form lenses^[91, 92] and waveguides^[93, 94].

Deformable micromirrors provide a low-cost adaptive optics solution which holds the promise of a low power compact high performance. With large mirror deflection achieved, it can be used in applications from sophisticated optical cross-connects^[95] to human retina imagining^[66], extending the field from astronomical imaging systems^[96] to laser-based communication^[97] and biomedical imaging^[66, 98, 99].

3.2 Design and Simulation

Inspired by the design of the programmable microfluidic system discussed in the previous chapter, we inherit the system's design and adjust it for fabricating micromirror arrays for adaptive optics. A thin layer of metal is patterned into micromirror arrays to reflect the incoming light. The mirror arrays are then transferred to the PDMS membrane. This membrane is deformed by the microheaters, which are integrated in the PDMS chamber under the membrane. The arrays embedded in the PDMS membrane inherit the deformation of the membrane and form a deformed reflection surface. The deformability is precisely controlled by adjusting the voltage applied on the microheaters. Since the microheaters are addressed with multi-complex control bus, the deformation of the whole mirror surface can be adjusted based on needs. Based on the discussion in Chapter 2, this thermal-pneumatically driven scheme provides a significant benefit in achieving large stroke deformations. State-of-the-art electrostatically driven mirror can provide only 8 µm of stroke with very high voltage while the proposed micromirrors can easily reach up to 10 µm with less voltage.

Multiple arrays were designed with different lengths (16 μ m, 32 μ m, 64 μ m, 128 μ m and 256 μ m). Each different length design is arrayed into several array sizes. Demonstrated in

Figure 47 is a 14×14 array with a dimension of $64 \mu m \times 64 \mu m$.



Figure 47 Micromirror array design (single array)

A simulation was performed to verify how the metal layer would conform to the deformation of the PDMS membrane. A simplified model of PDMS membrane with a 5×5 array was generated in I-deas \circledast . Both the PDMS membrane and the metal layer were meshed using "mapped" method.

Figure 48 illustrates the results when the membrane was deformed under a pressure applied at the bottom of the membrane. The metal layer was set to be aluminum. As it shows in

Figure 48, the aluminum layer deformed together with the membrane and conformed to the deformed shape of the membrane.



Figure 48 Simulation of micromirror array

3.3 Fabrication

Fabricating a metal layer on a soft substrate is always a challenge in microfabrication. Many micromirror arrays studied for the human retina^[100] requires the structure to sustain large mechanical strain or deformation while retaining electro conductivity and surface roughness. More challenges lie in the fabrication process while trying to keep the surface roughness of the exploited metal layer. Aluminum and gold are two commonly used metals for mirror design due to their good reflectance. When thermal evaporating or sputtering either of these two metals onto the PDMS substrate, a compressive stress is produced due to a thermal expansion mismatch. Aluminum has a thermal expansion coefficient (CTE) of 24×10^{-6} /K , gold's CTE is 14×10^{-6} /K while PDMS has a CTE as large as 960×10^{-6} /K. When a thin metal film is deposited directly onto PDMS substrate, the mismatch exploits random wrinkles on the metal surface which results in a

significant surface roughness change of the reflective metal layer^[101].



Figure 49 Evaporated gold layer on PDMS substrate^[101]

To avoid this "wrinkle" effect, we present a fabrication process to transfer a thermal evaporation deposited aluminum pattern into a PDMS membrane without the "wrinkle" effect.

Figure 50 illustrates the fabrication process of the micromirror on the PDMS membrane. Negative photoresist NR 9 - 1500 P is spun onto a clean silicon wafer and flood exposed before a hard bake in a 150 $^{\circ}$ oven. An evaporation of 0.5 µm aluminum is performed on Cyro system before Shipley S1813 is spun at 3000 rpm for 30 seconds. Then, the S 1813 layer is exposed under UV light for 10 seconds and developed afterwards. Aluminum etch is then followed before S 1813 is flood exposed and removed by remover.

After the patterning process, well mixed PDMS is poured on to the substrate and spun at 3000 rpm to trap the aluminum mirror in the PDMS membrane.



Figure 50 Fabrication of Aluminum Micromirrors Arrays

Figure 51 is a microscopic image of an array of aluminum mirrors taken after the aluminum mirror array was released from the substrate with an immersion in acetone. The sacrificial photoresist layer was dissolved in acetone and a PDMS membrane with patterned aluminum micromirrors was achieved. Since Acetone can significantly reduce the adhesion between the aluminum layer and PDMS membrane, this releasing immersion bath should be performed quickly with extreme cautions. As noticed during fabrication, transferred micromirrors with a length smaller than 64 µm have difficulty adhering to PDMS membrane due to this effect.



Figure 51 Aluminum micromirror array on PDMS substrate

Surface roughness measurement of the transferred micromirrors was performed on a Veeco® NT 3300 Optical Profiler system to examine the aluminum surface. Figure 52 exhibits the roughness of a $64 \,\mu\text{m} \times 64 \,\mu\text{m}$ mirror. The aluminum mirror surface reflected the measurement light and generates interference fringes. The fringes simultaneously changed with the roughness of the mirror surface. Veeco captured this change and analyzes the surface condition measured. Approximately 40 nm roughness is reported for the measured mirror surface.



Figure 52 Veeco measurement of surface condition of aluminum micromirror on PDMS substrate

3.4 Test Results and Discussion

In order to test the deformations of the fabricated micromirror arrays, a prototype of the proposed device was made by assembling the mirrors membrane to a PDMS test substrate with test chambers.

The PDMS test substrate with chambers was built with the help of rapid prototyping. A 3-D structure was first created in pro-E as illustrated in

Figure 53. A plastic mold was then fabricated using 3-D System SLA-250 with UV-light sensitive resin RenShape FL-5170.



Figure 53 Test Chamber for micromirror arrays

Well mixed PDMS is then poured on to the mold and cured at 65 $^{\circ}$ C for 48 hours before it is peeled off from the mold. The previously fabricated PDMS membrane with micromirrors was bonded irreversibly to this test substrate in such a way that the aluminum micromirrors are facing up (

Figure 54).



Figure 54 Aluminum Micromirror arrays on PDMS substrate

A syringe was then used to push air into the chamber under the mirrors. The pressure provided by this compressed air deformed the membrane and the deformed surface was investigated using the Veeco profiler system. The surface curvature of a deformed micromirror array with a $25\,\mu m$ deformation was presented in

Figure 55. Distribution of the curvature along x and y axis demonstrated the deformation of the mirror arrays.



Figure 55 Veeco measurement of deformed micromirror array

Images of a simulated and actual deformed mirror array are compared in Figure 56. The center of the array holds the most deformation and the mirrors deformed as well yet retained a good mirror surface. As noticed, the center mirror maintained a good curvature without breakage or peeling off. The actual deformations conform to the simulation results well.



Figure 56 Comparison of simulated and actual deformed mirror array

3.5 Conclusion and Outlook

Micromirror arrays are always constrained by the small deformation of the mirrors or small tilt angle. The use of a soft substrate such as PDMS membrane can provide large deformation with low power actuation. But to fabricate a good reflective metal on a soft substrate can be challenging.

We modified our proposed microfluidic system and presented a sacrificial layer assisted process to transfer the reflective aluminum micromirrors to the PDMS membrane. The whole device inherits the major structures and the actuation method. A simplified test was performed to verify that the deformation of the micro-mirror arrays on PDMS membrane matched the simulation results. The surface profile was studied with the aid of a Veeco profile system. The deformed micromirror arrays maintained a good curvature without breakage or peeling off.

Further characterization of this deformable micromirror array will be able to reveal the detailed relationship between the driven power and the reflection ability. Also, an improved structure or a primer coating process to improve and secure the adhesion of reflective surfaces to PDMS membrane in large deformation scenario is foreseeable by the current result.

The developed micromirror system provides a cost-effective solution to those applications from sophisticated optical cross-connects to human retina imagining with its unique deformability.

CHAPTER 4 MECHANICAL PROPERTIES OF PDMS

4.1 Introduction

Recent developments in MEMS technology are generating new opportunities for applications in biological and medical analysis. The diversity of these applications has encouraged new fabrication technologies and new materials to be revealed. The practical nature of polymers along with rapid prototyping and mass production as well as lower cost compared with silicon make them uniquely qualified for the development of BioMEMS.

Much like other MEMS materials used in a wide variety of applications, polymer structures also suffer from degradation or failure mechanisms. In fact, the properties of all the materials in thin film technology are often different from the macro scale bulk behavior. Nowadays, the macro-scale bulk material properties of MEMS materials are often used as micro-scale properties by assumption, which produces unreliable results in MEMS design and simulation. As a result of scaling laws, materials behave stiffer or stronger as the sample size decreases. This means a material which is soft in bulk volume might act like a hard material when its application size is scaled down. Therefore, micro mechanical test on MEMS materials is necessary when studying the mechanical behaviors of materials on the micro-scale.

Among the polymers used to fabricate microfluidic systems, like polyurethane, polyimide, polycarbonate, polymethylmethacrylate (PMMA),

polystyrene, polyethyleneterephthalate glycol (PETG) and polyethylene, Polydimethylsiloxane (PDMS) is the most widely used polymer in both academic and industrial research groups for applications in microfluidics. With desirable properties such as low cost, ease of mass production, microfabrication compatible, high deformability and transparency, PDMS is a formidable and promising material in MEMS. There is a significant need to examine this material's mechanical properties at both macro and micro scales with its application in MEMS.

PDMS is a heat curable polymer which always comes in a two-part kit consisting of a pre-polymer (also called base or part A) component and a crosslinker (also called curing agent or part B) component. Although PDMS is commonly mixed with a weight ratio 10A:1B, other ratios are also being used to fabricate 3-D MEMS structures by bonding PDMS parts with different mixing ratio. The mechanical properties of the cured PDMS vary with different mixing ratios and may affect the function of designed devices. At the same time, the bonding strength between these bonded PDMS parts is neither well-known nor studied.

The use of PDMS in BioMEMS applications is promoted by the development of Soft Lithography techniques including micromolding, micro contact printing, replica molding, microtransfer molding, and solvent-assisted micromolding. All these techniques usually involve photolithography to create a mold before transferring micron or even submicron features to PDMS. During the process, PDMS is either exposed or immersed into different chemicals such as photoresist developers, organic solutions and acids. When PDMS is fabricated into embedded structures in a microfluidic system, this polymer material may sustain more than one kind of chemical liquid during the microfabrication process. In addition to this, microfluidic systems which are made out of PDMS are often used in bio-MEMS and serve as platforms for bio-reactions, such as in a Lab-onchip or in a PCR process ^[16, 77, 79, 102-104], long periods of contact with different biochemicals are required.

Though PDMS stands out with its inner properties when used as a bulk material, small changes can be observed after experiencing the chemical immersion ^[105]. The study of the mechanical property change of PDMS after chemical immersion will benefit those researchers who utilize PDMS as a MEMS material and make micro-scale PDMS devices. With the microfluidic systems being used widely in bioMEMS, it is important to know how the PDMS will react in these systems at a micro-scale in order to provide correct or precise information in the results, both from a chemical aspect or mechanical aspect. Therefore, a thorough mechanical property study of PDMS will be important for improving the performance and durability of the MEMS devices.

Several studies have been conducted to reveal PDMS properties when it is being used in a MEMS structures. Hwa Seng Khoo1, et al performed a mechanical characterization of microscale elastomeric membrane by applying a central point load ^[106]. Some of the effects involved with chemical immersions were studied by Alvaro Mata, et al. 1cm² patterned PDMS samples were immersed in different chemicals and changes in weight, surface hydrophilicity and degradation of patterns were studied ^[105]. PDMS can hold up well in most chemical immersions with few or even no pattern degradations except corrosive acids and a few active organic solvents. As they reported, contacting with most organic solvents will result in weight loss while at the same time there was no pattern degradation. How this weight change will influence micro-devices' behaviors has yet to be studied.

Another challenge PDMS faces in its MEMS applications is temperature effect. There are many occasions that require structures made of PDMS can sustain high temperature for a long time, such as used in thermal pneumatic driven systems. This brings out the consideration of the effect the high temperature could take on PDMS.

Detailed study of these issues will be able to prompt the wide applications of PDMS in the development of bioMEMS.

4.2 Standards for Mechanical Properties Tests

The American Society for Testing Materials (ASTM) has defined a set of standard test procedures to identify mechanical properties of bulk materials, such as Young's modulus, Poisson's ratio, fracture strength and etc. These standards are strictly followed when materials are studies on the macro-scale. However even they are not perfect appropriate, guidance and references can still be obtained by following and adapting these rules. There are two major categories for materials properties testing: direct methods and inverse methods. Direct methods include the most commonly used tensile test, which is also adapted by ASTM standards. Membranes test, resonant test, bending test and other similar tests are grouped as inverse methods which a model is constructed for the test structure. Both groups of tests are used to study materials on the microscale by researchers.

4.2.1 Tensile Test

Many of the mechanical properties of a material can be extracted from a tensile test. In a tensile test, a sample is strained at a constant rate and the stress needed to maintain this strain rate is measured. The stress and strain can either be measured in terms of engineering stress and strain or true stress and strain. The elastic modulus, the ultimate tensile stress, the fracture stress, the modulus of toughness, and the modulus of resilience can all be determined from a tensile test.

Figure 57 shows the test configuration. The specimen is fixed to a grip at one end and held firmly while the other end is fixed to another grip which is moving at a constant rate. Attached to the fixed grip, a force/displacement transducer is glued using adhesive or connected with a pin and ring mechanism to sense the elongation of the attached specimen.



Figure 57 Tensile test configurations

The stress of the specimen can be obtained using Equation 15

$$\sigma = \frac{F}{A} \tag{16}$$

And the strain of the specimen is defined as Equation 16

$$\varepsilon = \frac{\Delta L}{L_0} \tag{17}$$

The Young's modulus is then deduced from General Hook's Law as Equation 17

$$E = \frac{\sigma}{\varepsilon} = \frac{F}{A} \frac{L_0}{\Delta L}$$
(18)

A tensile test stands out from other tests because of its simplicity. Yet the test requires extra attention when handling and mounting the specimen, especially in the case of brittle materials. And microscale materials can be more difficult than bulk materials.

4.2.2 Bending Test

Bending test can be used to avoiding the difficulty of mounting a microscale specimen in a tensile test. In the bending test, a cantilever beam is bent by a small force and the lateral deformation is measured under microscopy. For a beam with a length of L, width b, thickness h, Poisson's ratio v the Young's modulus is exacted by the expression in Equation 18:

$$E = \frac{4(1-\upsilon^2)L^3}{bh^3} \frac{P}{\delta}$$
(19)

Chasiotis, I. and Knauss, W.G. demonstrated such a micro tester performing bending test with the aid of Atomic Force Microscope (AFM)^[107]. Figure 58 shows a bending test where the silicon micro cantilever is pushed down by a stylus.



Figure 58 Micro cantilever bending

4.2.3 Resonant Test

For an isotropic elastic cantilever beam, the following differential equation (Equation 19) describes its free bending vibration with no damping:

$$EI\frac{\partial^4 y}{\partial x^4} + \rho A\frac{\partial^2 y}{\partial x^2} = 0$$
(20)

Where y is the deflection of the beam, E is the Young's modulus, I is the inertia, ρ is the density of the material and A is the area of cross-section of the beam.

Based on the boundary condition of the cantilever beam, we can derive from this equation that the 1st order frequency of the beam's vibration with no damping has the following relation with Young's modulus (Equation 20):

$$f_i = 0.162h/I^2 \sqrt{E/\rho}$$
 (21)

When the damping is not zero, the resonant frequency is associated with f_i as in Equation 21:

$$f_x = f_i (1 - \frac{I}{4Q^2})^{1/2}$$
(22)

Where Q is the quality factor and can be derived from the peak width of the resonant curve. So, by measuring the 1st order of resonant frequency, we can measure the Young's modulus of the materials.

In resonant tests, a cantilever beam is fixed above the electrode. A sine wave voltage is applied between the beam and the electrode to generate electrostatic force and vibrate the beam. A laser beam is focused on the tip of the cantilever beam and an aperture detector is positioned near the edge of the deflected beam. When the cantilever beam is vibrating, the aperture's size change with the amplitude of the vibration, and the voltage generated by the detector follows the change. The bigger the amplitude of the vibration, the higher the output voltage from the detector. When the amplitude reaches the largest value, the frequency is recorded as the 1st order resonant frequency.

4.2.4 Membrane Test

A bulge or membrane test uses a model of the behavior of the test structure and, in the case of a linear material can provide Young's modulus. The bulge test is really a specialized form of deflection technique, where the substrate is removed to leave a thin membrane of the film, surrounded by the film still supported on the substrate. If the membrane is pressurized differentially and the bow is measured using interferometric techniques, a stress strain curve can be obtained, the slope of which gives the modulus of the film, and the intercept gives the initial residual stress.

There are many variations of mechanical properties test. Each of these methods has its own advantages and weaknesses with respect to test specimen preparation and experimental result analysis. Among all the mentioned techniques, microtensile test is highly preferred because of its simple boundary conditions and a direct capture of force-displacement relation and is trusted in characterizing mechanical properties of materials.

4.3 Methodology

The tensile test of PDMS under different conditions is performed with the aid of an MTS Tytron 250 micro force tester (Figure 59). The tester has a resolution in force of 10 μ N and an accuracy of 0.1 μ m in displacement measurement. After the installation, a tensile force was applied to the specimen. The elongation and the corresponding force during the test were recorded automatically by the Basic TestWare®.



Figure 59 MTS Tytron 250 Micro Force Tester with PDMS samples mounted on through two leveled clamps and control software interface

The stress and strain are calculated according to the geometric information of the specimen. The strain-stress curve is drawn and analyzed to extract the mechanical properties of the tested PDMS.

4.3.1 Experiment Method

Silicone RTV 615 (General Electric) and PDMS Sylgard® 184 (Dow Corning Corporation) were tested. The manufacturers recommend that the prepolymer and cross-linker be mixed at a 10:1 weight ratio, respectively. In this research, PDMS specimens with various mixing ratios of 2:1, 5:1, 7.5:1 10:1, 12.5:1 20:1 and 50:1 were tensile tested. Bonding strength between different mixing ratio groups was studied. Different lengths and thicknesses were chosen for PDMS specimens with 10:1 ratio to characterize the effect of scaling law on PDMS. Tests of PDMS specimens after chemical immersion were also performed to quantify the effect of various chemical used in microfabrication and biological analysis.

All prepared samples are mounted onto the micro force tester and a tensile force is applied until failure. Experimental data is gathered by the computer software and processed according to Hook's Law.

4.3.2 Specimen Design

The tensile test specimens are prepared following the ASTM standard *ASTM D 412 test standard for vulcanized rubber and thermoplastic elastomers - tension*^[108]. The dog-bone shaped test samples are made one-fifth of the standard
size as shown in Figure 60. Samples fabricated obey this standard and have a gauge length of 6.6mm.



Figure 60 Specimen design geometry

To study the mechanical properties changes of PDMS with different mixing ratio, several mixing ratios (A: B=2, 5, 7.5, 10, 12.5 and 50) were assigned to a series of specimen with same gauge length, width and thickness. Bonding strength was studied with several groups of PDMS pairs as in Table 5. Each pair has two different mixing ratios and the bonding processes were operated by heating at 80C for 1.5 hours.

Pair #	1	2	3	4	
Ratio 1 (A: B)	20:1	30:1	40:1	50:1	
Ratio 2 (A: B)	2:1	3:1	4:1	5:1	

Table 5 Bonding ratio pair

In order to study the scaling law effect, some samples were categorized into two groups. One group was for the study of scaling law effect with different sample lengths. A group of length (2.2mm, 3.3mm, 4.4mm, 5.5mm, 6.6mm 7.7mm and 8.8mm) were chosen. Another group of PDMS samples have controlled thickness as 50 μ m, 100 μ m, 150 μ m, 200 μ m and 300 μ m. The widths and lengths of the samples remain identical.

Samples in the studies for temperature effect and chemical immersion effect were all based on the design shown in Figure 60. Samples for temperature effect study were heated on 100 \mathbb{C} , 200 \mathbb{C} and 300 \mathbb{C} hotplates with different time periods. Samples for chemical immersion effect were processed in different solutions for different time length (1/2 hour, 1 hour, 2 hour, 10 hours and 24 hours). All of the samples have a same gauge length of 6.6mm, a same thickness of 50 µm and a same width of 1.1mm.

4.3.3 Specimen Fabrication

PDMS itself is not a photo-curable material and is difficult to form patterns as photoresists or other photo-sensitive materials doing UV light exposure. When making testing specimens, researchers always use doctoral scalpels to cut bulk PDMS into desired shape. When the samples thickness goes up to millimeter scale, the material can be treated as bulk material. Patterning method using scalpel will be then appropriate in fabrications. This technique could bring non-ignorable errors into the test if considered at a microscale level. In this research, other methods are used to prepare PDMS specimens. The choice of fabrication method is based on the desired thickness.

For thin thickness samples ($30 \sim 50 \mu m$), a simple molding method is employed. The fabrication process is shown in Figure 61. Photolithography is used to make the pattern on thick positive photoresist AZ 4620. The patterned photoresist forms the mold and PDMS is spun onto the substrate at a low speed on a spinner. After that, excess PDMS is removed mechanically using a rubber blade. After curing, PDMS samples are pulled off the silicon substrate with care. Figure 62 gives out the fabricated samples before and after the positive photoresist is removed.



Figure 61 Molding method for specimen preparation



Figure 62 Specimen prepared with molding method

For thick samples (>50 μ m), a hot embossing method is utilized to form patterned PDMS. Figure 63 demonstrates the fabrication process. Thick negative photoresist SU-8 is UV-patterned on a Cr/Cu coated silicon wafer before electroplating.



Figure 63 Hot embossing method for specimen preparation

The electroplating experiment is set up as shown in Figure 64. A power supply from Dynatronix is connected to the sample and copper electrode in a

copper plating bath. The desired thickness of electroplated copper is controlled by current.



Figure 64 Electroplating experiment setup

After electroplating, SU-8 and pre-coated Cr/Cu layer are removed and the electroplated Cu is released from silicon substrate (as shown in Figure 65). This electroplated Cu acts as a mold in the following hot embossing step.



Figure 65 SU-8 mold on Cr/Cu coated wafer (left) and electroplated Cu after release (right)

After the mold is placed on a silicon wafer, PDMS is poured onto the surface. Another silicon wafer is then placed on top of the mold and put into Nanonex NX2500 nanoimprinter. A 300 psi pressure is applied on the wafers for 5 min to squeeze the excess PDMS out of the wafers and then the sample is heated up to 150 °C to cure the PDMS. After the sample cures and cools down, the patterned PDMS specimens are removed from the Cu mold and ready for test after allowing the PDMS to relax for 24 hours.

The reason why two fabrication methods are involved is due to the need for precision. For thin samples, the spacer mold will be hard to handle with and the deformation or residue stress of the spacer could lead to huge errors into sample sizes. Photolithography can keep the exact shape and thickness of the mold making. So the first method is used to fabricate specimens thinner than 50 μ m. However this method is not ideal for thick samples since it is hard for positive photoresist to reach a thickness above 50 μ m. Negative photoresists such as SU-8 can be fabricated with large thickness; however, the removal of patterned photoresists involves long time immersion in remover at high temperature. This removal process could introduce unnecessary effects on PDMS properties. So, electroplating and hot embossing are chosen to fabricate those thick specimens.

A set of PDMS specimens for chemical immersion effect study with the same geometric sizes (50 μ m thick, 6.6 mm long, 1.1 mm wide) and same mixing ratio (10:1) were prepared using fabrication process showed in Figure 66. The specimens were then put into different chemical solutions for a certain amount of

time (30 minutes, 1 hour, 2 hours, 10 hours and 24 hours). After immersion, the samples were cleaned using DI water and dried before tensile tests.



Figure 66 Fabrication process of samples for chemical immersion test

4.3.4 Testing Setup

When performing test, the specimen is mounted onto MTS Tytron® 250 micro force tester using a special designed grippers as shown in Figure 67.



Figure 67 Customerized gripper

Two axial alignment guiders on the grippers guarantee the alignment and reduce the error caused by misalignment along the axial direction. This design is suitable to a great variety of materials such as metal, plastics and composites. After mounting, the specimen is stretched by an axial force. The loading controlling model can be chosen from either force mode or displacement. The displacement and forces are recorded automatically.



Figure 68 Tensile test procedure

4.4 Results and Discussion

As mentioned in Section 3.2.1, the strain expressed by $\sigma = \frac{F}{A}$ and the

strain defined as $\varepsilon = \frac{\Delta L}{L_0}$ obey the General Hook's Law where Young's modulus

can be calculated as Equation 17:

$$E = \frac{\sigma}{\varepsilon} = \frac{F}{A} \frac{L_0}{\Delta L}$$

4.4.1 Mechanical Properties of PDMS with Different Mixing Ratios

A stress-strain relationship of a RTV 615 PDMS specimen fabricated is presented in Figure 69. At the beginning of the test, the resulting strain and the stress have linear relationship, which shows the elastic nature of the PDMS, a silicone rubber material. The Young's modulus was derived from the slope of the stress-strain curve. However, PDMS with different weight mixing ratios behaved quite differently. Figure 70 shows the change of Young's modulus versus weight mixing ratios tested. It can be clearly seen from this curve that the Young's modulus decreases as the ratio of A:B increasing. This means the higher the portion of part A in the PDMS, the lower the resulted Young's Modulus (the more soft nature). This should be considered as the contribution of the polymerization of the materials, the larger the ratio, the more un-reacted base polymer presented, the softer the final stage materials. Interestingly, the Young's modulus drops dramatically when the curing agent in the ratio is relative small (A: B >10). For example, it decreases from 2.1 MPa to 0.6 MPa when the mixing ratio is increased from 2 to 10. However, when the weight mixing ratio is greater than 10, the resulting Young's modulus of PDMS is almost independent of the mixing ratios (Figure 70). When the mixing ratio increased from 10 to 50, the change in Young's Modulus is around 0.5 Mpa only.



Figure 69 Stress-strain relationship of PDMS with mixing ratio 10:1



Figure 70 Young's modulus versus weight mixing ratio

As shown in Table 5, four pairs of PDMS are selected for the bonding test. All the samples are bonded under the same condition (heated at 80 °C for 1.5 hours right after the cure process). After bonding, small samples were cut from these bonded PDMS with the same orientation of bonding surface, showed in Figure 71.

A:B=2:1	A:B=3:1
A:B=20:1	A:B=30:1
A:B=4:1	A:B=5:1
A:B=40:1	A:B=50:1

Figure 71 bonded PDMS with the same orientation of bonding surface

As shown in this figure, with the higher dose of part A, the bonding diffusion layer is thicker than those with lower dose of part A. These samples then are mounted to the MTS test system in a way that the boundary surface is located in the middle of the testing beam and perpendicular to the applied force direction. Tensile force then is applied to the sample until it breaks. All the elongation and force change are recorded by the software. As it implies in Figure 72, the bonding strength strongly depends on the mixing ratio. The curve fitting result shows that the bonding strength changes exponentially with the weight mixing ratio and has the best value (130KPa) at the couple 30A:1B and 3A:1B. After this point, with the increase of part A, the resulted bonding strength decreases.



Figure 72 Bonding strength strongly depends on the mixing ratio

Examining the broken sample, it is found that the fracture surface occurred at the side with larger dose of part A and is close to the bonding surface. This can be explained. Because the diffusion layer has higher concentration of agent part B than the larger part A dose side, the resulting young's modulus of this thin layer is relative higher. And at the contrast, this layer has lower concentration of agent part B than the smaller part A dose side, and thus coming along with a relative smaller Young's modulus and more soft and elastic than the other side. This property is good for device design. It gives the opportunity of device failure to the device body, not the structure between two parts. In multilayer structures, this feature is important to make sure the device will work properly under load.

4.4.2 Size Effect on Mechanical Properties of PDMS

Size effect happens on stiffer materials such as silicon in the MEMS community. A question may be asked if PDMS has a similar issue. In order to answer this question, several mechanical tests were conducted to find the relationship between mechanical properties and the PDMS sizes.

Several specimens with the same thickness of $250 \ \mu\text{m}$ and the same width of 1.1 mm were prepared using Dow Corning Sylgard 184 to be tested. As seen



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Figure 73, by decreasing the ratio of length to width, the ultimate tensile stress (UTS) of the specimen increases correspondingly and approaches to its bulk material property. When the thickness decreases at the microscale, it is also seen that PDMS behaves stiffer with shorter length.



Figure 73 Ultimate tensile stress vs. length and thickness

Both PDMS from General Electronics (RTV 615) and PDMS from Dow Corning (Sylgard 184) were tested. Small differences of UTS values were observed but both PDMS samples' test curves illustrate the same scaling law effects on the material behaviors (as in Figure 73 (b)).

Another parameter that could influence the properties of PDMS is the thickness of the specimens. $30\mu m$ 50 μm , $100 \mu m$, $150 \mu m$, $200 \mu m$ and $300 \mu m$ thick specimens were studied together with bulk samples in order to find out the thickness' effect. As shown in

Figure 74, the derived young's modulus of the PDMS membrane with a mixing ratio of 10 increases dramatically when the thickness of the membrane decreases. When mixing ratio increases, more base-component exists in cured PDMS. Though the derived Young's modulus decreases with the increasing mixing ratio, the change of the properties becomes less significant than with a small mixing ratio.



Figure 74 Young's modulus vs thickness

Taking all of the parameters of the sizes into effect, it is clear that the scaling law does apply to PDMS at micro-scale. When the PDMS membrane's thickness decreases, the material behaves stiffer than in its bulk material status. Derived Young's modulus of PDMS at microscale is approximate twice of the value for its bulk material. With the geometry sizes down, the Young's modulus increases and the material behaves stiffer. At millimeter scale, scaling law effect trails off and the mechanical properties of PDMS approach to the characteristics of bulk materials.

4.4.3 Chemical Immersion Effects on Mechanical Properties of PDMS

Most microfluidic devices made of PDMS are used in bio-MEMS. The devices need to be either contacted or immersed in different solutions used in the bio-analysis and microfabrication process. Some commonly used solutions in applications are listed but not limited to Acetone, Methanol, Ethanol, Isopropanol, PCR buffer, photoresist developers and acids. Even with the natural stability of polymer, PDMS can still be affected by immersion in these chemicals. Degradation and properties may change exist during the immersion.

4.4.3.1 Organic Solvents Immersion

Figure 75 presents a stress-strain relationship of PDMS samples after 1hour and 10 hours acetone immersion.



Figure 75 Strain-stress Curve of PDMS after Acetone Immersion

As showed in the result (Figure 76), the Young's modulus of PDMS after immersion in acetone and Isopropanol (IPA) decreased as immersion time increased. Both immersion results showed a 200KPa dropping in Young's modulus. Longer immersion may lower the value of Young's modulus down to around 450KPa.



Figure 76 Young's modulus of PDMS after immersion in Acetone and Isopropanol

Another two sets of specimens which were immersed in ethanol and

methanol present different tendency (

Figure 77). Higher values of Young's modulus are noticed with the situation of longer immersion time. The effects of these two immersions saturated around 750KPa and longer immersion would not result same big influence.



Figure 77 Young's Modulus of PDMS after Immersion in Methanol and Ethanol

The ultimate tensile stress (UTS) is being influenced with chemical immersions too. Figure 78 shows the change in UTS after different immersion time in these four organic solvents. For immersion in Acetone and Isopropanol, results show a decrease in UTS with the time increase. In the same immersion time series, UTS present an ascending tendency with an increase in immersion time. Chemical immersions may bring a 2~3MPa change in UTS of PDMS membrane.



Figure 78 Ultimate Tensile Stresses of PDMS after Immersion in Acetone, Isopropanol, Methanol and Ethanol

Along with the change in UTS after immersion, the elongation of the PDMS specimens shows a change with immersion (Figure 79). After immersion in acetone and isopropanol, elongation increases by about 100% with the increase of immersion time while methanol and ethanol immersion decrease the elongation by about 100~150%.



Figure 79 Elongations of PDMS after Immersion in Acetone, Isopropanol, Methanol and Ethanol

Chemical reactions on PDMS surfaces may make contributions to these changes. Organic solvents cause the PDMS specimen to swell or strengthen which results in a change in mechanical behavior. Swollen PDMS behaves softer than normal PDMS since the invasive organic solvent weakens its structure. Acetone and isopropanol are two examples of this mechanism. Another possible reaction between the organic solvents and PDMS is that the resulted new surface or structure is more intense which results in a relatively stiffer behavior and higher UTS with smaller elongation. The organic chemical immersions did not alter PDMS's Ultimate Tensile Stress dramatically. Even though, small changes were still noticed after organic chemical solution treatment.

4.4.3.2 SU-8 Developer Immersion

In soft lithography, PDMS is always paired with the fabrication of an SU-8 mold. Some processes may require a submersion of PDMS in SU-8 developer. Most developing procedures involved in LIGA fabrication are limited to less than 30 minutes. In some extreme case, when a desired SU-8 patterns' thickness is up to several hundred microns, longer developing time is needed. A group of PDMS samples were immersed in SU-8 developer for different times and then tensile tested. Figure 80 demonstrates the slightly downtrend of Young's modulus of PDMS after immersion. The developer is an organic solvent which weaken PDMS to some extent (less than 5%) but cannot drastically change the mechanical properties of PDMS.



Figure 80 Young's modulus, UTS and elongation of PDMS after SU-8 developer immersion

4.4.3.3 Acids Immersion

Bearing in mind the nature of rubber, when confronted with acids, PDMS presents an ideal rubber behavior.

When samples were immersed into full concentration sulfuric acid, the strong acid dissolved the samples very quickly. Diluted sulfuric acid still reacts with PDMS yet with a slower rate.

A typical mechanical behavior of PDMS after acid treatment was performed using an immersion in nitric acid. The derived Young's modulus increased with the immersion time. Figure 81 shows an increase of 500% change in Young's modulus. The PDMS sample with 24 hours immersion became brittle and lost its original rubber material behavior. Along with this change, the ultimate tensile stress also demonstrated a dramatic decrease with immersion time (Figure 82). This acid immersion process also costs PDMS its plasticity. Compared to the untreated PDMS which can hold an elongation up to 400%, the nitric acid treated PDMS lost almost 75% of its elongation capability. A 24-hour nitric acid treated PDMS sample can only sustain a 50% elongation.



Figure 81 Young's modulus of PDMS after nitric acid immersion



Figure 82 Ultimate tensile stress of PDMS after nitric acid immersion



Figure 83 Elongation of PDMS after nitric acid immersion

Nitrification may be considered as the major contribution to this property change of PDMS. The longer the immersion time, the greater the nitrification of PDMS and the material behaves more brittle and stiff.

4.4.3.4 Electroplating Solutions Immersion

PDMS's stable chemical characters draw more and more attention from MEMS researchers. Some of the studies use this rubber material as a mold in micro-electroplating process to achieve pattern transfer^[109]. This application requires PDMS mold stay immersed for a relative long time in an electroplating solution during the experiments. We studied the immersion effects on PDMS with nickel and copper electroplating solutions.

A typical nickel electroplating solution is prepared following Table 6. Two temperatures of 45 $^{\circ}$ C and 60 $^{\circ}$ C were used in electroplating and were studied as different experiment parameters. Each temperature setup occupied one group of PDMS samples for different immersion time and then tensile tested.

Chemical Ingredient	Concentration
Nickel sulfate	1.54M
Boric acid	0.73M
Sodium dodecylsulfate	0.1g
Saccharin	0.1g

Table 6 Nickel electroplating solution recipe

*operating temperature 60 °C, pH 3.5~4, current density 10~25mA/cm²

Figure 84 and Figure 85 present the effect of nickel electroplating solution immersion on PDMS. The derived Young's modulus increases under both temperatures. There is a change of 100~100 KPa in PDMS's Young's modulus after 24 hours immersion as well as in the ultimate tensile stress. This change can be associated with the acid in the heated electroplating solutions. Even with a diluted concentration, once the solution is heated, the acid reacts more actively with PDMS to some extent which results in the slight hardening of the samples. It should also be notice that in both figures these samples treated in the lower temperature (45°C) possess of a larger value of both Young's modulus and ultimate tensile stress than those treated at 65 °C. This may be explained by the possibility that higher concentration or temperature can bring more intense hardening effect to PDMS but when the temperature is high, the reaction rate between the acid inside the solution and PDMS will compromise the hardening effect and results a softer material behavior on treated PDMS.



Figure 84 Young's Modulus of PDMS after Ni Electroplating Solution Immersion at 45 ∞ and 60 ∞



Figure 85 Ultimate Tensile Stress of PDMS after Ni Electroplating Solution Immersion at 45 $^{\rm C}$ and 60 $^{\rm C}$

Another group of PDMS samples were tested after being immersed in a copper electroplating solution. Table 7 lists out the components of the copper electroplating solution used.

Chemical Ingredient	Concentration			
Copper sulfate	100~150g/L			
Sulfuric acid (98%)	100ml/L			
Hydrochloride	0.13ml/L			

Table 7 Copper electroplating solution recipe

*operating temperature 25 °C, pH <1, current density 30mA/cm²

The existence of sulfuric acid in this solution leads to the results that with the accumulation of immersion time, sulfuric acid gradually attacks PDMS samples and causes the derived Young's modulus of PDMS to decrease. Due to the diluted concentration of sulfuric acid in this solution, the PDMS sample will not be able to dissolve in the solution, instead, it becomes easier to break with a decrease of 150KPa in Young's modulus (Figure 86) and 4 MPa in UTS(Figure 87).



Figure 86 Young's Modulus of PDMS after Cu Electroplating Solution Immersion



Figure 87 UTS of PDMS after Cu Electroplating Solution Immersion

Exposing PDMS to a solution with acids as ingredients can result in a change of PDMS's mechanical properties. Fully concentrated acid immersion can dramatically change PDMS's material behavior by either a hardening effect or a dissolving effect. Diluted acid immersions still have some similar effects on PDMS yet much milder.

4.4.3.5 PCR Buffer Solutions Immersion

A Polymerase chain reaction (PCR) test can achieve DNA amplification by letting the PCR buffer solution react with DNA samples through three constant temperatures for melting at 95 °C, extension at 60 °C and annealing at 77 °C. A commonly used PCR buffer solution consists of 20mM Tris-HCl (pH8.8), 10mM KCl, 10mM (NH₄)₂SO₄, 2mM MgSO₄ and 0.1% Triton X-100. Normally multiple cycles are engaged. A group of 10 PDMS samples were treated on Eppendorf Mastercycler Gradient for 30 cycles of PCR tests with the collaboration with the

Department of Plant Biology in University of Minnesota . Table 8 below shows

the results of tensile test of these treated samples.

Sample number	1	2	3	4	5	6	7	8	9	10	Average
Young's Modulus (KPa)	571.3	657.3	602.8	598.9	508.2	524.1	605	651	587.5	628.5	593.46
UTS (MPa)	9.56	8.67	9.57	7.99	7.11	8.39	8.92	8.89	8.89	9.06	8.705
Elongation	543%	434%	529%	424%	454%	510%	474%	462%	502%	480%	481.20%

Table 8 Tensile test results of PDMS after 30 cycles PCR test



Figure 88 Young's modulus of PDMS with PCR immersion

PDMS almost retains the same mechanical properties after this PCR immersion test. The average Young's modulus is around 593.5KPa which is slightly smaller than our reported value in 2007^[110]. It confirms the stability of PDMS for the use in medical and biological applications. The feasibility that PDMS microsystems provide make PDMS ideal for meeting the need of high

throughput technology in PCR tests and emerge PCR as a powerful tool in not only genetic analysis, medical diagnostics and also forensic applications.

4.4.4 Temperature Effects on Mechanical Properties of PDMS

In the MEMS applications of PDMS, this soft material is always required to sustain both a large deformation and a high working temperature, especially when it is used in a thermal pneumatic driven microsystem. It is necessary to discover how the material would behave when it is working under a high temperature for a long time period.

A group of samples were prepared as described in previous section. These samples were grouped into three sets. Samples were heated on hotplates with different temperatures of 100 \C 200 \C and 300 \C . Each set correspond to one temperature. Each set contained 25 samples. Five of them were heated for 30 minutes, five for 1 hour, five for 2 hours and five for 3 hours. Tensile tests of these samples were presented in Figure 89 and Figure 90.



Figure 89 Young's modulus of PDMS with temperature effect

The values of Young's modulus for PDMS with temperature effect exhibit a small decline with the heating time. Higher temperatures result lower Young's modulus. The decreases in Young's modulus at different temperatures were all smaller than 250K. The ultimate tensile stress also declines with the increase of heating time as seen in Figure 90. However, this effect became less noticeable with a high heating temperature.



Figure 90 Ultimate tensile stress of PDMS with temperature effect

4.5 Conclusion

The mechanical properties of materials play an important role in the performance of MEMS devices. However, borrowing the mechanical properties of materials from macro theories and applying them at micro-scale requires corrections. When PDMS becomes more and more popular in MEMS applications, the demand of understanding scaling effects on this material increases.

Direct tensile tests have been conducted to convey the relevant mechanical properties of PDMS. The results comply with the fact that materials usually behave differently on the micro-scale. Different parameters that could affect the resulted PDMS properties have been identified as thickness, length and mixing ratio. The test results comply with the scaling laws. PDMS with micro-scale size behaves stiffer with a higher length/width ratio, a thinner thickness or a higher cross linker mixing ratio.

Material challenges faced by PDMS in MEMS devices often involve not only structural deformations but also encountering with various chemical solutions. Micro-scale PDMS samples were treated in commonly used chemical solutions in microfabrication and direct tensile tests were performed to reveal the effect. The results comply with the fact that materials could behave differently after chemical immersions and reveal the influence of chemicals which are involved in most microfabrication process. As a matter of fact, lots of PDMSbased micro devices are used for biological analysis, especially PCR. The performance of PDMS in the presence of PCR buffer solutions is a concern of researchers. The tensile tests were conducted to reveal the effect and affirmed the good compatibility of PDMS in PCR buffer solution.

Temperature effect on PDMS is also noticed and studied. Long time operation at high temperature could bring PDMS a decrease in derived Young's modulus and an ultimate tensile stress

This detailed tensile test study gives out more accurate mechanical properties under various operating conditions which can facilitate and accelerate this material's various applications in MEMS and bioMEMS devices.

CHAPTER 5 CONCLUSION AND OUTLOOK

The work presented in this dissertation was to design a custom programmable microfluidic system. A systematic study has been conducted on this system in aspects of design, simulation, fabrication and initial characterization. An application of this design was adopted into a micromirror array system and tested as a deformable micromirror. The experimental results have been proven by finite elements analysis conducted. PDMS is the material of choice for the design and its properties are important to the success of this study. This research explored the mechanical properties of PDMS in terms of the effects of scaling law, chemical immersions and temperatures.

The proposed design has been developed as a PDMS-based programmable microfluidic system which can provide a universal platform for different application of microfluidic systems where microflow control is needed regardless of chemical contact. The developed microfluidic system uses both thermal pneumatic and electrostatic actuation to realize the action and control of the microvalve and microflow in the microchannel. A prototype was tested with a 5V voltage for actuation. Thermal pneumatic simulation optimizes the heat flux at a value of $5 \sim 8 \times 10^7$ W/m² to get a better actuation around 1 MHz. A voltage of 50V is applied to electrostatically drive a 20 µm thick PDMS membrane maintaining a 50 µm deflection.

An application of the developed microfluidic system conducted with specific attention was paid to preliminary results of the system itself and the material properties of PDMS. A sacrificial layer assisted process is presented to transfer reflective aluminum micromirrors into the PDMS membrane. A simplified test verified the test result from the finite element analysis. The surface profile was studied with the help of a Veeco profile system. The deformed micromirror array maintained good curvature without breaking or peeling off.

PDMS is the major material in the developed devices. Direct tensile tests were developed to test the mechanical properties of this material both as bulk material and thin film based MEMS material. PDMS at the manufacturer's recommended mixing ratio (10A:1B) has a Young's modulus of 615KPa as bulk material. By adjusting the mixing ratio of two PDMS components, different mechanical properties are gained, which can facilitate researcher by allowing various desired properties to be considered for their respective designs. As a MEMS material, the Young's Modulus and ultimate tensile stress of PDMS vary in comparison to bulk material properties. Different parameters that could affect the resulted PDMS properties have been identified as thickness, length to width ratio and mixing ratio. PDMS at micro-scale size behaves stiffer with a higher length/width ratio, a thinner thickness or a higher crosslinker mixing ratio. The use of chemicals and high working temperature during the operation of PDMS-based microfluidic systems are unavoidable and the chemical immersion effect and temperature effect on PDMS were characterized.

The results of this study provide fundamentals of developing programmable microfluidic system by using PDMS. PDMS is biocompatible and cost-efficient solution for many applications including μ TAS system, biosensors, sophisticated optical cross-connects and human retina imagining with its unique deformability.

Further applications of the custom programmable microfluidic system would be greatly promoted with expended study of the following suggested studies:

- There are still lots of room to optimize the structure of the microfluidic system for more efficient and easier fabrication. Integration of control circuits with the system was not yet been achieved. Characterization of the microfluidic system is far from enough before using the system in real µTAS.
- PDMS properties changes after chemical immersion still need a more detailed investigation into the reaction theories of PDMS with chemicals. This study could be realized with the assistance of Environmental Scanning Electronic Spectrometer and Fourier Transforms Infrared Spectroscopy.
- As discussed in Chapter 4, the micromirror application encountered difficulty with holding all the transferred reflective aluminum mirrors due to the utilization of acetone in removing sacrificial layers. An improved structure or a primer coating process is needed to improve this adhesion. Not only would it solve this challenge but also it would further benefit
securing the reflective surfaces to PDMS membrane in large deformation scenario. Further optical characterization of this deformable micromirror arrays would be able to reveal the detailed relationship between the driven power and the reflection ability.

In summary, the custom programmable microfluidic system investigated in this dissertation exhibits tremendous potential in providing a universal platform for biological and medical analysis application. PDMS's mechanical properties make it a novel MEMS material extend the application field even wider into those fields where large stroke and low power actuation are needed, such as in arrays of micromirrors. Further research that extends the current work would lead to a diverse world of new look of microfluidic systems.

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