Development of micropump for microfluidic applications

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DEVELOPMENT OF MICROPUMP FOR MICROFLUIDIC APPLICATIONS

A Dissertation

Submitted to the Graduate Faculty of the
Louisiana State University and
Agricultural and Mechanical College
in partial fulfillment of the
requirements for the degree of
Doctor of Philosophy

in

The Department of Mechanical Engineering

by

Dong Eun Lee
B.S., Mechanical Eng., Yeungnam University, 1994
M.S. Mechanical Eng., Yeungnam University, 1996
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For Mijung and Hyunwoo
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ABSTRACT

This dissertation covers the research work on two types of micropumps, one is based on magnetohydrodynamic (MHD) principle that utilizes Lorentz force for actuation, and the other is based on electrochemical actuation.

The AC-type MHD micropump was designed and analyzed as a solution to the bubble formation problem encountered in DC-type MHD micropump. A UV-LIGA process using thick layer of SU-8 negative photoresist was successfully developed to microfabricate the AC MHD micropump. Preliminary studies and tests of the AC MHD micropumps demonstrate that bubble formation was significantly reduced to permit the proper function of the micropump. A continuous flow was also successfully demonstrated with no moving mechanical parts needed. To develop the mathematical model for flow of conductive fluids between the electrodes was a challenging issue. To overcome this problem, the impedance of conductive fluids between two electrodes was measured by Electrochemical Impedance Spectroscopy, which then helped to obtain a relatively accurate mathematical model for the system. The design, simulation, fabrication, and test results of the AC MHD micropump are presented in this dissertation.

Electrochemical actuator was investigated for micropumping applications. In our research efforts to develop DC-type MHD micropump, bubble formation problem caused by electrolysis proved to be one of the most difficult issues. However, microactuation based on expanding bubbles from electrolysis effect has scale advantages compared with other commonly used microactuation mechanisms. It can therefore be used as a very
efficient actuation source for micropumping applications. We have designed, analyzed, and fabricated a microactuator based on the electrochemical principle. Preliminary experiments have proved that the bubbles generated in electrolysis can be manipulated by carefully controlling the direction and amplitude of the input signal. This has demonstrated that efficient pumping at micro volume of fluid can be realized by addition of required valves. The microfluidic system with micropump and integrated active microvalve has been successfully demonstrated. The working principle, design, simulation, and preliminary results of the electrochemical actuator have also been presented in the dissertation.
CHAPTER 1  INTRODUCTION

The development of microfluidics has rapidly expanded to a wide variety of applications in the last two decades. Microfluidics has the potentials to have revolutionarily impact on chemical analysis and synthesis, similar to the impact of integrated circuits on computers and electronics. Microfluidic devices in general can be classified into four categories: fluid control devices, gas and fluid measurement devices, medical devices, and miscellaneous devices such as implantable drug pumps. The major applications of this microfluidics include medical diagnostics, genetic sequencing, chemistry production, drug discovery, and proteomics. Of all the microfluidic devices, micropumps are the most essential ones because controlled transport of fluids in microscale is the most crucial task.

With the efforts by many researchers in the fast developing field of micro electro-mechanical systems (MEMS), many of different types of micropumps have been designed to handle small, precise volumes for chemical, medical and biomedical applications. Like in the other MEMS applications, the first approach in designing a micropump is the miniaturization of mechanical principles in macroscale. The next approaches were to apply new transport effects such as electrokinetic effects, acoustic streaming, the magnetohydrodynamic (MHD) effect, and the electrochemical (ECM) effect, which are more effective on the microscale than the macroscale. Most of these micropumps are nonmechanical pumps.

Micropumps may also be categorized as either mechanical pumps or nonmechanical pumps. In mechanical pumps, energy is periodically or continuously added by the
application of force to one or more movable boundaries. These forces then result in a
direct increase in pressure difference up to the value required to drive the fluid through
check valves or ports into the discharge line. Electrostatic, electromagnetic, pneumatic,
thermopneumatic, and piezoelectric force are the representative ones in mechanical type
of micropumps. However, most of these micropumps either suffer from high pressure
drops across the check valves or are vulnerable to mechanical fatigue of the oscillating
membranes or flapping valves which result in a reduction in performance and reliability.

1.1 Goals of the Research in this Dissertation

The object of the research work presented in this dissertation was to develop a
micropump that can precisely deliver a microscopic volume of fluidic sample to targeted
location in an analysis system. The factors to be considered include: 1) precise control of
delivering amount. Most microfluidic applications require high accuracy delivery of
sample solution and those precise cycling time of pumping. The micropumps therefore
need to be electrical controllable; 2) good connectivity. In most applications, the
micropump needs to be connected with other microfluidic components and devices, such
as micro mixer and sensors, it therefore have miniature size and be possible for cost
effective fabrication; 3) Minimal leakage. For the proper operation of the micropumps,
the leakage should be minimal, or prevented if feasible. To avoid expensive wafer
bonding, UV LIGA process, which is relatively cheaper than silicon based fabrication
technology, is preferred and therefore used in our work.

1.2 Literature Review of the Micropumps

There have been many types of mechanical micropumps based on various driving
force. Spencer et al. made a piezoelectrically actuated micro diaphragm pump that shows
one of the early technical examples of mechanical micropumps. Smits et al. created peristaltic micropump that is achieved by sequential activation of the piezo actuators. The first micropump that introduced passive check with piezo actuator was published by Van Lintel et al. at the University of Twente. Van de Pol et al. reported thermoneumatically driven micropump. Van del Pol’s device used an air-filled chamber on top of the pump diaphragm. Electrostatic actuator was introduced as a driving force to actuate diaphragm because of some advantages such as high operation frequencies (up to few kHz), lower power consumption and full MEMS compatibility for an easy integration. Zenger et al. reported the first practical electrostatic micropump that vertically-stacked chip designs in silicon. Electromagnetic actuation that uses a permanent magnet attached to the pump diaphragm and external coil was developed by few research groups. Generating high pressure and flow rate are the major advantages of these micropumps, but it also has some problems such as high pressure drops across the check valve, mechanical fatigue failure of the oscillating membranes or flapping valves which causes reduction in performance and reliability.

Nonmechanical pumps add momentum to the fluid by converting another nonmechanical energy form into kinetic energy. A number of principles such as electrohydrodynamic (EHD), electrokinetic, ultrasonic, and RF, magnetohydrodynamic and electrochemical displacement micropumps are found in literature. Bart et al. reported EHD micropump using EHD induction effect, and Richter et al. made EHD pump using EHD injection effect. Electrokinetic micropump was reported by Paul et al., who studied the electrokinetic transport of liquid in microporous media. RF and ultrasonic pumping that uses progressive mechanical wave or the quarts wind” of a vertically oscillating surface was reported by several research groups.
reported DC type Magnetohydrodynamic(MHD) pump that based on the Lorenttz effect.\textsuperscript{26} Lemoff \textit{et al.} made an AC type MHD pump, which improved the bubble generation problem of DC type MHD pump.\textsuperscript{27} Bohm \textit{et al.} reported an Electrochemical displacement micropump, which is using the electrolytic generation of gas bubbles as a primary pumping effect.\textsuperscript{28,29}

Miniature pumps or macroscale pumps are the most common solutions for flow rates more than 10ml/min. Flow rates ranging from 10ul/min to several milliliters per minutes is the typical operation range of Mechanical micropumps. For flow rates less than 10ul/min, nonmechanical micropumps are needed for an accurate control of these small fluid amounts.

1.3 Research Outline

1.3.1 Micropumps Based on Non-Contact Actuations

To achieve delivering small fluid amounts accurately in our DNA mixing and analysis system, accuracy and cost should be considered in selecting pumping mechanism. As mentioned before, the flow rate of mechanical micropumps is more than 10ml/min, and that is quite large for our application. Desired flow rate of our application is around 0.5ul/min. Many of micropumps were made of conventional MEMS processes based on silicon technology, but they have some disadvantages such as limitation of materials used in processing, relatively high processing cost, and low aspect ratio. Compared with silicon technology, the LIGA or UV-LIGA process based on polymer materials such as SU-8 and PMMA has some advantages: (1) it allows the fabrication of high aspect ratio microstructure up to one thousand of micrometers; (2) different materials such as plastics, metals, alloys, ceramics, or a combination of these materials can be used as structural materials; (3) flexibility, chemical and biological compatibility, 3D fabrication possibility
and low cost in materials and mass production. With all of these factors considered, the MHD micropump and electrochemically (ECM) actuated micropump have the advantages of simple design, scale advantages, and easy to be fabricated with the UV-LIGA fabrication technology.

### 1.3.2 MHD Micropump

The prototype of AC type micropump based on MHD principle was successfully developed using UV-LIGA fabrication. This differs from our previous AC MHD micropump\(^\text{30}\) in driving method. In previous work, an AC signal was only applied to electrodes and, the permanent magnet was used to provide magnetic force. This configuration generates not only forwarding force but also backward one too. To overcome this backward force, nozzle and diffuser structure were introduced in micropump structure. In the new design, permanent magnet was replaced with an electromagnet, and AC signals were applied to electrodes and electromagnet. To synchronize AC signals that applied to electrodes and electromagnet, currents were monitored by oscilloscope, and two current phases were synchronized using phase shift circuit. The maximum flow rate was obtained with actuations at 500Hz driving frequency, and the flow rate obtained was about 1.6\(\mu\)l/min.

### 1.3.3 Electrochemical (ECM) Actuator and Its Application

In our previously reported efforts to develop MHD micropump, bubble generation problem caused by electrolysis has proved to be one of the most difficulties at low frequency. As soon as bubbles start to form in a MHD pump chamber, the output flow jumps up dramatically. However, as soon as the bubble generations become significant, the flow stops because the dramatically increased electrical resistance stopped current
flow through the electrodes and reduced the Lorentz force significantly. It has been observed that the bubble generation is a very fast process and very powerful. If we can utilize this bubble force as a driving force of micropump with low cost fabrication such as UV-LIGA and accurate controlling electrolysis, the electrochemically actuated micropump is believed one of the best solutions that can overcome the disadvantages of MHD micropump.

In the prototype electrochemical actuator, Platinized platinum electrode that serves as a working electrode and silver chloride that serves as auxiliary and reference electrode were inserted after fabrication. Su-8 structure was fabricated on PMMA cover to create a gap between valve and cover. After bonding cover and bottom part, two reservoirs were attached to inlet and outlet port to fluid connection. Static and dynamic modeling using electrochemical analysis was successfully demonstrated.

The integrated microfluidic system that has an electrochemical active valve and micropump has been fabricated using UV-LIGA techniques, and its performance was successfully tested.

1.4 Dissertation Outline

Detailed design specifications required to our microfluidic system, working principle and specific design are presents in next chapters, followed by chapter 4 & 5 which present the static and dynamic simulation of micropumps. Device fabrication issues are covered in chapters 6 and 7. The experimental setup and preliminary test results are presented in Chapter 8. Finally summary and future work are discussed in chapter 9.
CHAPTER 2 AC TYPE MAGNETOHYDRODYNAMIC (MHD) MICROPUMP

2.1 Direct Current (DC) MHD Micropump Principle

Magnetohydrodynamic (MHD) is the theory of the macroscopic interaction of electrically conductive fluids with a magnetic field. It is of importance in connection with many engineering problems such as sustained plasma confinement for controlled thermonuclear fusion, liquid-metal cooling of nuclear reactors, and electromagnetic casting of metals. The first investigations in this field were conducted by Faraday (1836). The MHD principle has been widely used in nuclear power plant cooling systems and in MHD propulsion ship applications but has not seen widespread application in conventional pumps.

2.2 Principle of Magnetic Actuation

When an electrical current, \( i \), is supplied to a conductor in magnetic field, \( \vec{B} \), Lorentz force, \( \vec{F} \) orthogonal to both the electric current and magnetic field, is generated on the current carrying conductor. The generated Lorentz force:

\[
\vec{F} = i \times \vec{B} \cdot l
\]  

Where \( l \) is the length of the conductor, is shown schematically in Figure 2-1.

When an electrical current passes through an electrically conductive fluid filled between two facing electrodes due to ionic conduction, a similar phenomenon is occurred. These ion motions lead to the transport of charges and hence to the flow of electrical current through the electrolyte solution.
If an external magnetic field is applied perpendicular to the direction of the current, a Lorentz body force is generated. Because of this distributed body force, the conductive fluid (ions) experiences a pressure difference that drives the fluid to flow along the channel.

### 2.3 MHD Governing Equations

The fundamental equations governing the MHD principle comprise two parts:

1. Electromagnetic
2. Classical fluid dynamics
The electromagnetic governing equations can be represented by the Maxwell's equations, current continuity equation, and constitutive equations, while the classical fluid dynamics governing equations can be represented by the continuity equation and the Navier-Stokes equation, respectively. The reduced form of the MHD governing equations can be written as the Ohm's law, conservation of mass, and conservation of momentum, respectively, with the assumptions: constant magnetic, electric, and fluid properties, single phase flow, negligible free charge density, and negligible magnetic Reynolds number \( (Re_m = \sigma \mu_e U L) \), where \( \sigma \) = electric conductivity, \( \mu_e \) = magnetic permeability, \( U \) = characteristic velocity, and \( L \) = characteristic length.

\[
J = \sigma(E + \nu \times B) \tag{2.2}
\]

\[
\nabla \cdot \nu = 0 \tag{2.3}
\]

\[
\rho \left( \frac{\partial \nu}{\partial t} + \nu \cdot \nabla \nu \right) = -\nabla p + \mu \nabla^2 \nu + J \times B \tag{2.4}
\]

where \( J \) = current density, \( E \) = electric field, \( \nu \) = fluid velocity, \( B \) = magnetic flux density, \( \rho \) = density, \( p \) = pressure, and \( \mu \) = viscosity.

It has been shown that with the dimensional analysis and the assumption, applied electric field intensity across the microchannel is higher than the second term of the right hand side of Equation (2.2). This results in \( J \) that has little dependence on \( \nu \). Equation (2.2) then becomes:

\[
J = \sigma E \tag{2.5}
\]

At steady state and with low Reynolds number (\( Re \leq 2300 \)), which is expected to be the flow condition for this MHD micropump, Equation (2.4) further reduced into the form of:
\[ 0 = -\nabla p + \mu \nabla^2 \nu + J \times B \]  

(2.6)

Equations (2.5) and (2.6) are the simplified one-dimensional governing equations of the MHD micropump.

Based on this unique MHD principle of actuation, a MHD-based micropump poses four major advantages:

(1) It has an intrinsic high reliability because of no moving parts;

(2) The fabrication process is relatively simple and provides the potential for batch fabrication;

(3) Potential for remote or tele-operation by control of an applied external magnetic field;

(4) The ability to be integrated with other modules or systems with no limitation on the shape of the channels;

The simplicity results in a reduced risk of clogging and damage to molecular materials being pumped in biomedical applications, and also the reduced risk of failure due to mechanical fatigue of the check valves. Because of these advantages, research efforts have been focused on developing MHD micropumps.26, 27, 35.

We have microfabricated and tested a LIGA-based DC MHD micropump35. A 1.2 ml/min flow rate was obtained using a 2% NaOH electrolyte; however, at potentials higher than 14 volts, significant bubble generations were observed due to electrolysis which impeded the pump performance. Similar problems were also reported by J. Jang et al.26 when a DC MHD micropump was used to pump seawater. A. V. Lemoff et al.27 have reported an effort to overcome bubble generation problems by utilizing high
frequency alternating current (AC) supplied to the electrodes with an AC electromagnet to generate a phase dependent electromagnetic field. A continuous flow rate of 37 μl/min was obtained with this AC MHD micropump. The disadvantage of this AC micropump design is that it requires relatively high power consumption to produce a relatively strong and high-density electromagnetic field (250 Gauss).

2.4 Alternating Current (AC) MHD Micropump Principle

Effort has been made to develop an AC micropump based on the MHD principle to reduce or eliminate the bubble generation problems caused by electrolysis of the pumping fluids in the DC MHD micropump. This can be achieved by supplying an AC current with a sufficiently high frequency across the two facing electrodes of the micropump, causing the double layer at the metal electrodes and the pumping fluids interface to charge and discharge in the same manner as a capacitor without any electrolysis effect, as shown in Figure 2-3.

The design, analysis, simulation, microfabrication and preliminary test results of the AC MHD micropump are presented in the following chapters of this dissertation.

Figure 2-3  Charging and discharging of the electrolyte double layer on imposition of an AC current, \( i \) as a function of time \( t \)
CHAPTER 3 ELECTROCHEMICAL MICRO ACTUATOR

3.1 Working Principle

Many of micropumps have been developed in various designs, but their working principles can be categorized as mechanical or nonmechanical micropumps. In nonmechanical micropumps, the nonmechanical energy is converted to kinetic energy, and this energy adds momentum to the fluids. The ECM utilizes bubble force that is generated by electrochemical reaction during electrolysis.

Generally speaking, the bubble is a not good choice for macro sized fluidic system because surface effects inertial and other body forces are dominated forces in macro scale. In micro scale, bubble force has two advantages, such as the scaling advantage of surface effects and density difference between liquids and gases.

The bubble pressure is proportional to the surface tension and inverse proportional to bubble radius. By this advantage, bubble can have substantial force at micro scale. The pressure inside a bubble can be presented by following equation.

\[
\Delta p = \frac{2\sigma}{r}
\]  

(3.1)

Where \( r \) is radius of bubble and \( \sigma \) is surface tension of bubble.

The bubble actuators, or ECM actuators, can have very high expansion rate by the density difference between liquids and gasses. Typical density of gasses is less than three orders of magnitude of liquids.\(^{36}\) For example, 100\( \mu \)m diameter gas bubble generated by electrolysis has 617 times less density than water. So, electrolytic generation of gas bubbles from electrolyte can make a very large expansion and long throw actuation. To make micropump using gas bubble actuation (ECM), two check valves are required to
operate it. Inlet check valve will be open, and outlet valve will be closed during push out phase. Opposite operation will be carried out in suck-in phase.

3.2 Review of the Previous Valves in Microfluidic System

The active microvalves are one of the most critical components in our micropump system. In the ECM micropump, check valve will be fabricated with pump structure. Active microvalves can be categorized by their actuation principle as pneumatic, thermoneumatic, thermomechanical, piezoelectric, electrostatic, electromagnetic, electrochemical, and capillary force microvalves. Huff et al. made a bossed circular membrane which is actuated by pneumatic pressure. Vieider et al., Sim et al., Ohori et al. and Rich et al. also made silicon based microvalve with silicon rubber, thinner membranes or corrugated membranes using pneumatic pressure. Thermopneumatic microvalves were also fabricated by some research groups. In this type of microvalves, metal electrodes were introduced as a heater, and silicon, metal or silicon rubber was used as membrane material. Liseč, T., et al., fabricated solid-expansion actuator for thermomechanical valve. Chu et al., reported bimetallic materials for this type valve. Shape memory alloys also used for this valve. These type valves are utilizing thermal energy that is converted directly into mechanical stress. Piezoelectric actuators are one of the first to be used in micro valves because of the large force and commercial availability. External piezostack was assembled in silicon or glass substrate. It serves as a valve actuator to manipulate the flow through the inlet or the outlet. Efforts have been made to fabricate electrostatic actuator based on the attractive force between two oppositely charged plates. Although electrostatic actuator has a fast response, the high driving voltage and small displacement make it difficult to integrate into
microfluidic system. Pourahmadi, F., *et al.*, and several groups reported electromagnet valves that use a solenoid actuator with a magnetic core and a coil for generating the magnetic field. It offers a large displacement, but it needs external solenoid or fabrication of microcoil that make it complex to integrate. Papavasiliou, A.P. *et al.*, reported electrochemical valve that is using gas bubbles generated by electrolysis of a solution. Generated gas bubbles convert back to liquid in the reversal reaction. Platinum served as a catalyst that is able to absorb hydrogen.

### 3.3 Design of Prototype Electrochemical Actuator and Application

The basic design of prototype electrochemically actuated micropump (ECM) is shown in Fig 3-1. The structure is composed of following components: electrodes for supplying electricity, fluid channels, chamber for electrolysis (bubble generation), two reservoirs into which inlet and outlet tubes will be inserted for delivering solution, and cover.

![Figure 3-1 The schematic diagram of prototype electrochemically actuated micropump](image)

Figure 3-1 The schematic diagram of prototype electrochemically actuated micropump
SU-8 (Microchem Corp., Newton, MA) negative photoresist were selected to realize main structure of the pump, such as the flap valves of micropump, the microchannels, and reservoirs. Two different electrodes were used to realize electrochemical reaction. Ag/AgCl was used to auxiliary electrode, and platinum black was used to working electrode. These electrodes were directly inserted in the chamber. This prototype was used to determine electrochemical behavior and establish modeling of electrochemical actuation.

With the fast developing of microfluidic devices those are extensively used in micro total analysis system or Lab-on-a-Chip, the application devices are demanding integrated delivery devices includes micropump and microvalve those are essential part in handling of the fluid. The focusing areas of current researches are on developing on separated devices those are eventually assembled together through external connection. To achieve integrated microfluidic device that has pump and valve, the structure and mechanism should be as simple as possible. For this point of view, the electrochemical actuation, which is using bubble application, is promising. Recently, many research efforts have been reported on using electrochemical actuation as an alternative to other high voltage or power consumption actuators such as electroosmosis. The actuation mechanism of electrochemical actuators is based on reversible electrochemical reactions that enable gas evolution (gas bubble expansion) and reduction by electrolysis in the aqueous electrolyte solution.

The integrated microfluidic system that has an electrochemical active valve and micropump has been fabricated using UV-LIGA techniques with promising results of the prototype electrochemical actuator. The entire system is composed of two electrochemical actuators, flexible polydimethylsiloxane (PDMS) membrane structure,
and two levels of cover which is made of negative photo resist SU-8 on PMMA sheet (Figure 3-2). Recently PDMS came into the spotlight because of low cost, simple and rapid prototyping in microfabrication. The other advantages of PDMS as a microfabrication material are ease of bonding, excellent optical properties, and permeability. PDMS part and cover part were fabricated using multi layers of SU-8 process. Fill holes were fabricated together in PDMS structure. Platinum black was served as working electrode, and Ag/AgCl was served as counter and reference electrode. NaCl solution with 1M of concentration was used for electrolyte. Detailed fabrication techniques are covered in chapter 6.

![Figure 3-2 Schematic Diagram of the electrochemical actuated microfluidic system](image)

The basic principle of an ECM micropump can be schematically explained in Figure 3-3. Operation of the microfluidic system is sequentially controlled with following steps. The gas bubble that caused by electrolysis is start to push out the solution inside pump
chamber in push-out phase. When valve actuator is operating the flexible PDMS membrane, it close outlet hole, and that can stop the flow in the channel. The valve can be opened by reversal electrochemical reaction. The operation of two electrochemical actuators is done at opposite mode. The pump generates the gas that push out the solution in the chamber while the valve actuator is in reversal reaction to open the outlet hole. These sequences are repeated as a need of the application devices that connected or fabricated together with this microfluidic system. The control voltage, which is generated by a function generator, is supplied across the Pt black and Ag/AgCl electrodes for electrolysis and reversal reaction. Electrolysis process produces hydrogen and oxygen gases when the electrical potential is supplied to two electrodes. The gas bubbles in the valve chamber result in pressure building that causes PDMS membrane deflection and pushing out the solution from the pump chamber.

Figure 3-3 Schematic diagram of operation of microfluidic system; (A) Push-out phase, (B) Valve close phase
4.1 Bond Graph Dynamic Analysis of AC MHD Pump

The AC MHD micropump was designed to be without any moving parts; however, the dynamic behavior of the rapidly oscillating fluid in the microchannel is of great practical interest. To gain insight into the fluid dynamics of the MHD micropump, lumped-parameter models for different designs were considered.

In bond graph fluid system modeling, the power variables used are the pressure, $P$, as the effort variable ($e$) and the volume flow rate, $Q$, as the flow variable ($f$). Typically, three 1-port elements are used to model a section of fluid channel: an equivalent fluid resistor $R$, an equivalent capacitor $C$, and an equivalent inertance $I$.

The relationship between the pressure drop and the volume flow rate in a small section of microchannel is given by the following linear relationship:

$$ P = R \cdot Q $$

The equivalent fluid resistor for a rectangular cross-section can be derived by following equation:

$$ R = \frac{8 \cdot \mu \cdot \text{L}_{\text{pump}} \cdot (a + b)^2}{a^3 \cdot b^3} $$

Where, $a$ is channel depth, $b$ is channel width, and $\mu$ is viscosity of the fluid.

The general expression for the fluid inertance is:

$$ I = \rho \int \frac{ds}{A(s)} $$
Where, \(A(s)\) is the cross-sectional area along the path, \(s\), of the pump of interest. The inertance defined here can be used to calculate the equivalent pressure momentum for a segment of flow channel as \(P_m = I \cdot Q\). The equivalent inertances for the micro-flow channels are significant because of the small channel diameters.

The equivalent capacitance for an incompressible fluid in a segment of a microchannel can be derived as:

\[
C = \frac{Area \cdot \Delta x}{\beta}
\]  

(4.4)

Where, \(\beta\) is the fluid bulk modulus. The relationship between the pressure, \(P\), and the flow volume, \(Vol\), and the equivalent capacitance \(C\) in general is given by the following equation:

\[
P = \frac{1}{C}Vol
\]  

(4.5)

A microchannel can be subdivided into multiple segments containing the three 1-port elements. Bond graph lumped-parameter models for a microfluidic device are then constructed by combining these segments with other bond graph elements using either common flow (-1-) or common effort (-0-) junctions. Figure 4-2 shows a bond graph model for an AC MHD micropump that comprises three segments.

Three \(MSF\)s represent modulated flow source accounting for the distribution of current along the face of the electrodes. Remained \(MSF\)s are modulated flow source for electromagnet. The modulated gyrator couples the electrical sub-system with the fluidic sub-system. The usual symbols and constitutive laws are shown below:
Since magnetic field is generated from electromagnet in the system, the modulated gyrator is defined by the constitutive laws as:

\[
P = B(i_f) \cdot \frac{i}{a} \quad \quad e = B(i_f) \cdot \frac{Q}{a}
\]  \hspace{1cm} (4.6)

Where, \( B \) is the magnetic field, \( e \) is the electrical potential, \( a \) is the height of the pump channel, and \( i_f \) is the current which is supplied to electromagnet. The equation of the magnetic field of the electromagnet that has \( N \) turns of wire can be expressed as:

\[
B = \frac{\mu_i \cdot N \cdot I_0}{2\sqrt{l^2 + r^2}} \sin(\omega \cdot t)
\]  \hspace{1cm} (4.7)

Where, \( l \) is the core length of the electromagnet, \( r \) is the radius of the core, \( \mu_i \) is the permeability of the iron core, \( I_0 \) is the input current amplitude. The current, \( i \), can be expressed as:

\[
i = \frac{a \cdot \sigma \cdot L_{pump} \cdot e_0}{b} \cdot \sin(\omega \cdot t)
\]  \hspace{1cm} (4.8)

Where, \( \sigma \) is the fluid conductivity; \( e_0 \) is the input voltage amplitude, and \( \omega \) is the driving frequency.
4.2 Analysis of the Pump Electrodes as an Electrochemical Cell

The conductive fluid between the pump electrodes can be considered as an electrochemical cell. In an electrochemical cell, slow electrodes kinetics, slow preceding chemical reactions, and diffusion can all impede electron flow, and can be considered analogous to the resistors, capacitors, and inductors that impede the flow of electrons in an ac circuit. So, we can represents its performance by an equivalent circuit of resistors and capacitors that pass current with the same amplitude and phase angle that the real cell does under a given excitation. A frequently used circuit, called *Randles equivalent circuit*, is shown in Figure 4-3. Rs is the ohmic or uncompensated resistance of the solution between working and reference electrodes. Rct is the polarization resistance or charge...
Figure 4-3: Randles equivalent circuit of an electrochemical cell transfer resistance at the electrodes/solution interface. Cdl is the double layer capacitance at this interface. Figure 4-4 shows the electrical double layer between electrode and electrolyte solution.

Figure 4-4: Stern’s electric double layer model

The interactions between the ions in solution and electrode surface were assumed to be electrostatic in nature and resulted from the fact that the electrode holds a charge density which arises from either an excess or deficiency of electrons at the electrodes.
surface. In order for the interface to remain neutral, the charge held on the electrode is balanced by the redistribution of ions close to the electrode surface.

To get the values of $R$ and $C$ components, one need a special technique that is called electric measurement spectroscopy (EIS). In the EIS, an alternating signal of small magnitude with various frequencies (few mHz to 10Khz) are applied to the electrochemical cell. At high frequencies, the excitation waveform becomes much faster than charge transfer rate, the behavior of the equivalent circuit is dominated by ohmic resistance $R_s$. At the low frequencies, the capacitor acts as an open circuit and is effectively removed from the circuit. The impedance of the Randles cell is then the combined resistance values of the two series resistors $R_s$ and $R_{ct}$. The phase will approach that of a pure resistor ($0^\circ$). At intermediate frequencies, where the behavior is dominated by $C_{dl}$, the phase will approach that for a pure capacitance ($-90^\circ$). A bode plot of simple electrochemical cell is shown in Figure 4-5.

![Bode plot for a simple electrochemical cell](image)

Figure 4-5 Bode plot for a simple electrochemical cell
The bode plot of pump electrodes with 1M of NaCl electrolyte is shown in Figure 4-6. This EIS measurement was done by using potentiostats (Ametek PARSTAT 2263) and control software (Ametek PowerSuit).

Figure 4-6  Bode plot for pump electrodes with 1M of NaCl electrolyte

From the result, Rs is $50\,\Omega$, Rct is $10\,K\,\Omega$, and Cdl is $50\,nF$.

4.3 A Reconfiguration of Bond Graph with Electrochemical Cell Model

A fully augmented bond graph with equivalent circuit model is shown in Figure 4-7. $I_1$, $I_{14}$, and $I_{27}$ are the equivalent inertances (Equation (4.3)) for the corresponding channel segments. $C_{12}$, $C_{25}$, and $C_{37}$ are the equivalent capacitances (Equation (4.4)), $C_9$, $C_{22}$, and $C_{35}$ are the equivalent capacitances for electrochemical cell model. $R_5$, $R_6$, $R_{10}$, $R_{18}$, $R_{19}$, $R_{23}$, $R_{31}$, $R_{32}$, and $R_{36}$ are the corresponding equivalent flow resistances of electrochemical cell part. $R_2$, $R_{15}$, and $R_{28}$ are the corresponding equivalent flow resistances.
There are nine state variables: $Pm_1$, $q_9$, $Vol_{12}$, $Pm_{14}$, $q_{22}$, $Vol_{25}$, $Pm_{27}$, $q_{35}$, and $Vol_{37}$ in the model and nine state equations. They may be written as below:

\[
Pm_1 = B(i) \frac{MSF_{12}}{a} - \frac{R_{21}}{I_1} Pm_1 - \frac{1}{C_{12}} Vol_{12} \tag{4.9}
\]

\[
q_9 = MSF_{09} - \frac{1}{R_{10} C_9} q_9 \tag{4.10}
\]

\[
Vol_{12} = \frac{1}{I_1} Pm_1 - \frac{1}{I_{14}} Pm_{14} \tag{4.11}
\]

\[
Pm_{14} = B(i) \frac{MSF_{20}}{a} + \frac{1}{C_{12}} Vol_{12} - \frac{R_{15}}{I_{14}} Pm_{14} - \frac{1}{C_{25}} Vol_{25} \tag{4.12}
\]
\[ q_{22} = MSF_{20} - \frac{1}{R_{23}C_{22}} q_{22} \]  
(4.13)

\[ Vol_{25} = \frac{1}{I_{14}} Pm_{14} - \frac{1}{I_{27}} Pm_{27} \]  
(4.14)

\[ Pm_{27} = B(i_f) \frac{MSF_{33}}{a} + \frac{1}{C_{25}} Vol_{25} - \frac{R_{28}}{I_{27}} Pm_{27} - \frac{1}{C_{37}} Vol_{37} \]  
(4.15)

\[ q_{35} = MSF_{33} - \frac{1}{R_{36}C_{35}} q_{35} \]  
(4.16)

\[ Vol_{37} = \frac{1}{I_{27}} Pm_{27} \]  
(4.17)

Performance at several operating frequencies was simulated using **20 SIM-PRO** (Controllab Products B.V., Twente, NL). The software is based on bond graph method and no derivation of state equations is necessary. The solution of foregoing state equations was also obtained and plotted by following method.

Consider the nonhomogeneous state equation described by

\[ \dot{x} = Ax + Bu \]  
(4.18)

where \( x = n \)-vector, \( u = r \)-vector, \( A = n \times n \) constant matrix, \( B = n \times r \) constant matrix

The Laplace transform of this last equation yields

\[ sX(s) - x(0) = AX(s) + BU(s) \]  
(4.19)

or

\[ (sI - A)X(s) = x(0) + BU(s) \]  
(4.20)

Premultiplying both sides of this equation by \((sI - A)^{-1}\), we obtain

\[ X(s) = (sI - A)^{-1} x(0) + (sI - A)^{-1} BU(s) \]  
(4.21)

since \( \text{L}^{-1}[(sI - A)^{-1}] = e^{At} \)
\[ X(s) = L[e^{at}]x(0) + L[e^{at}]BU(s) \quad (4.22) \]

The inverse Laplace transform of this equation can be obtained by use of the convolution integral as follows:

\[ x(t) = e^{at}x(0) + \int_{0}^{t} e^{A(t-\tau)} Bu(\tau)d\tau \quad (4.23) \]

The output flow for the simulated dynamic response of pump is shown in Figure 4-8 and Figure 4-9.

Figure 4-8 Dynamic response curve of pump at operating frequency 500Hz using 20-SIM
Figure 4-9 Dynamic response curve of pump at operating frequency 500Hz by analytical solution
CHAPTER 5  STATIC AND DYNAMIC ANALYSIS OF ELECTROCHEMICAL ACTUATOR

5.1 Electrochemistry of Bubble Generation

The reaction at electrodes can be described as a consequence of the transfer of electric charges from one phase to another one. Suppose two phases, such as a metal and solution containing ions, are in contact. Some of the particles in the original phases tend to transfer to the other phase with their electrical charges. As a result of this reaction, one phase has an excess of positive charge, and the other has an excess of negative charge. There is a potential difference across the region between these two phases; we call this region an interphase. Two reactions at electrodes are shown in Figure 5-1.

![Figure 5-1 Reduction and Oxidation Model](image-url)

(A) Reduction and Direct Consequences

(B) Oxidation and Direct Consequences

Figure 5-1 Reduction and Oxidation Model
In a reduction reaction, a particle on the solution side of the interface accepts an
electron from the electrode. In an oxidation reaction, a particle donates an electron to the
electrode. The most direct consequences are the electrical charging of interphase and the
establishing of a potential difference across electrodes.

Figure 5-2 shows “The electrochemical substance producer.” A continuous flow of
electrons is maintained from electrical energy source. At the electron source, cathode,
electrons leave the electrode and particles of the solution are reduced. At the electron
sink, anode, the electrode takes electrons from particles, which are oxidized. Practical
example is the electrolysis of the water as shown in Figure 5-2. In acid solution,
hydrogen ions are reduced to produce hydrogen molecules, and water molecules are
oxidized to produce oxygen molecules:

Cathode \[ \text{H}^+ + \text{e} \rightarrow \frac{1}{2} \text{H}_2 \] (5.1)
Anode \[ \frac{1}{2} \text{H}_2\text{O} \rightarrow \frac{1}{2} \text{O}_2 + \text{H}^+ + \text{e} \] (5.2)

____________________
Overall Reaction \[ \frac{1}{2} \text{H}_2\text{O} \rightarrow \frac{1}{4} \text{H}_2 + \frac{1}{2} \text{O}_2 \] (5.3)

The external energy supply drives the cell in two electrodes. We can control
chemical transformations, each of which takes place only when electrons of a specific
energy are available by controlling the rate of electron transfer across the interfaces, and
the energy of the electrons.

5.2 Reversible Reaction in Electrochemical Actuator

Electrochemical actuation is operated by using reproducible production and consumption
of hydrogen bubble on the working electrodes.
Figure 5-2 The electrochemical substance producer; the electrolysis of water in acid solution.

Gas is evolved or consumed electrochemically by an oxidation or a reduction reaction at one of the electrodes. The schematic of redox reactions can be represented by:

\[ O^{\text{y+}} + n e^- \leftrightarrow R \]  \hspace{1cm} (5.4)

Where \( O^{\text{y+}} \) and \( R \) is the oxidized and reduced forms of the redox couple, and \( n \) is the number of electrons involved in the reaction. The equilibrium potential is given by the Nernst equation:

\[ E_e = E^0 + \frac{RT}{NF} \cdot \ln \frac{C_o}{C_R} \]  \hspace{1cm} (5.5)
Where $E^0$ is the standard redox potential, and $C_0$ and $C_R$ are the concentrations of oxidized and reduced components. The potential on the working electrode can be adjusted by the voltage source. We have two cases when potential $E$ differs from equilibrium value $E_e$.

(i) $E < E_e$

A gas is evolved on the working electrode by net reduction reaction.

$$O^{n+} + ne^- \leftrightarrow R \quad (5.6)$$

$$2H^+ + 2e^- \leftrightarrow H_2 \quad (5.7)$$

![Figure 5-3 Hydrogen gas is evolving at Pt Black electrode](image)

(ii) $E > E_e$

A gas bubble is shrunken on the working electrode by net oxidation reaction.

$$R \leftrightarrow O^{n+} + ne^- \quad (5.8)$$

$$H_2 \leftrightarrow 2H^+ + 2e^- \quad (5.9)$$

Some electrode materials are known for hydrogen gas evolution such as steel, Ni, Ni-coating, precious metal coating. Platinum black was preferred in our study for faster reverse reaction. With large number of small particles of platinum coated to platinum wire, the platinum black electrode can get the huge surface area, and that helps to achieve faster reduction reaction because it has a higher current exchange density than bright platinum wire. In the micro pump chamber, hydrogen bubble on the electrodes will be
generated and degenerated by reversible reaction. The speed of bubble generation can be obtained from following equation.

The number of moles of hydrogen N is given by:

$$N = \frac{Q}{nF} = \frac{i \cdot t}{nF},$$  \hspace{1cm} (5.10)

where \(n=2\), is the number of electrons required to obtain 1 molecule of \(H_2\), and \(F= 96485\) C/mol, the Faraday constant. Thus, the theoretical hydrogen production rate is:

$$\left( \frac{dN}{dT} \right) = \frac{i}{2F}$$  \hspace{1cm} (5.11)

For example, suppose the current supplied across two electrodes is 150mA, then we can get production rate of hydrogen gas.

$$\frac{150 mA}{2 \times 96485 A \cdot sec/mol} = 0.78 \mu mol/sec$$

These equations also can be used to calculate the pumping pressure by bubble formation.

### 5.3 Electrochemical Behavior of ECM

To complete electrochemical behavior of a system, cyclic voltammetry was taken with ECM pump. This is a reversal technic and is a kind of potential-scan. Figure 5-4 shows typical example of cyclic voltammetry. Figure 5-5 shows real cycle-voltammogram from prototype ECM. The potential scan was done from -1.5V to 1.5V with 10mV/s scan rate in 1.0M NaCl solution. The curve shape shows that electrochemical reactions were proceeded, and electrochemical reactions can be applied to microscale chamber. To test reproducibility of the current generation, two different potential levels were supplied across two electrodes. Figure 5-5 shows that supplied voltage and current reponse.
Figure 5-4 (a) Cyclic potential sweep  (b) Resulting cyclic voltammogram.

Figure 5-5 Cyclic Voltammogram of Prototype ECM

Even though the curve of Figure 5-5 does not following the exact shape of Figure 5-4 (a), it implies that electrochemical reaction can be progressed in micro size container.

5.4 Dynamic Analysis of Electrochemical Actuator

Bubble expansion and reduction rates were calculated by tracking the actuation of the bubble. The basic assumption in modeling and simulation of the ECM actuation is
that the gas bubble behavior and the resulting pressure and volume relation follow the ideal gas law with the ECM chamber at room temperature. Because the outlet of the flow channel was open to atmospheric pressure in our experiments and the pressure drop along from the micro chamber the outlet is assumed to minimal, the electrolysis can be assumed to occur at atmospheric pressure. The volume change can then be predicted by the following equation,

$$
\Delta V(t) = \frac{NRT}{P} .
$$  \hspace{1cm} (5-12)

Substituting equation (5) into (7) yields,

$$
\Delta V(t) = \frac{RT}{nFP}it ,
$$  \hspace{1cm} (5-13)

where $R$ is the universal gas constant, $P$ is the atmospheric pressure, and $T$ is the temperature. To simulate the current response by the potential input, the electrochemical actuator was modeled using the Randles equivalent circuit. The equivalent circuit for the electrochemical cell is shown in Figure 5-6, where $R_1$ is the solution resistance, $R_2$ is the polarization resistance, and $C$ is the double layer capacitance.

When an external voltage pulse, $E(t)$, is applied across the two electrodes, the current can be broken into two parts, one for the transient and the other for the static component, and the total current can then be calculated simply as, \(^{57}\)

![Figure 5-6 Randles equivalent circuit of the electrochemical actuator](image_url)
\[ i(t) = \left( E(t) \frac{e^{-((R_1+R_2)/R_1C)\tau}}{R_1} + \frac{E(t)}{R_1+R_2}(1-e^{-((R_1+R_2)/R_1R_2C)\tau}) \right), \] (5-14)

The equation is simplified as,

\[ i(t) = E(t)\left( \frac{R_2}{R_1(R_1+R_2)} \right)e^{-t/\tau} + \frac{E(t)}{R_1+R_2}, \] (5-15)

where time constant \( \tau \) is defined as

\[ \tau = \frac{R_1R_2C}{(R_1+R_2)} \] (5-16)

The volume change as a function of time can then be obtained by plugging Equation (5-15) into Equation (5-13),

\[ \Delta V(t) = \frac{RTE(t)}{nFP} \left[ \frac{R_2}{R_1(R_1+R_2)} e^{-t/\tau} + \frac{1}{R_1+R_2} \right] t, \] (5-17)

For any given voltage input \( E(t) \), both current \( i(t) \) flowing across the two electrodes and the volume change of gas bubbles evolving on the surface of the working electrode can be estimated using Equations (5-15) and (5-17), respectively.

Because it is very difficult to monitor the pressure generated by gas bubble expansion and reduction in the micro chamber in real time, we adopted an indirect measurement approach in which the volume of the gas bubble was measured at a given pressure level. A digital microscope camera was used to monitor the bubble expansion in the micro chamber. Since the inlet and outlet tubes of the fluidic system are left open and no other external fluidic channels are connected, it can be assumed that pressures at both the inlet and outlet are at atmospheric pressure. With the real time measurement of gas bubble volume in the reaction chamber, the pressure difference generated by the bubble expansion can be easily estimated if needed using ideal gas law.
The test setup is shown in the Figure 5-7. A mercury lamp was used to project light through the microscope onto the mixing chamber. The emission light and the reflected light from the mixing chamber were filtered using an optical filter, which permitted mainly the emission light to pass through. The images of the mixing flow were magnified with the aid of the microscope. A Nikon CV-252 digital video camera was used to monitor the mixing process. This standard NTSC video camera catches 30 frames (actually 29.97) per second, and two fields per frame. Control electropotentials were supplied to the two electrodes.

To test the dynamic behavior of the prototype electrochemical actuator, a pulse voltage source with a period of 20 seconds was supplied across the two electrodes and the current flowing through them was recorded. Since the bubble generation process in general is much shorter than that of the bubble reduction, efforts were made to minimize the bubble reduction time in order to obtain the electrochemical actuation with faster dynamic responses. Experimental results show that the maximum gas bubble reduction rate was achieved at a voltage of –1.5 V for the prototype electrochemical actuator. The bubble generation (in the reduction phase) period was selected to be about 4 seconds at a positive potential of 0.5 V. The bubble reduction (in the oxidation phase) was selected to be about 16 seconds at a potential of -1.5 V.

Figure 5-8 (A) and (B) shows the pulse voltage waveform and the measured current response curves corresponding to the input driving potentials. As can be seen from the current response curve, the currents were reproducible. This result proves that electrochemical reactions were progressed and reversed continuously, and the actuation can be reproducible.29 Because only two electrodes are used in the design for the actuator, reproductibility may be affected when a higher current is used for actuation.
The measured current curve as shown in Figure 5-8 (B) confirmed a first order character of the actuation mechanism of the actuator as discussed in early part in this section. Experiments showed that gas bubbles evolved at the surface of the working electrode (platinum black). Because both the inlet and outlet tubes of the fluidic system were left open to atmospheric pressure, the fluid in the system was driven to oscillate back and forth within the fluidic channel as the bubbles expanded and shrunk in the reaction chamber. It was found that the gas expansion and reduction cycle was 20 s,
corresponding exactly to the same driving voltage form applied across the electrodes. The basic dynamic behavior is very similar to a piston-cylinder system with the oscillating liquid body working as the equivalent piston and the gas bubble providing the actuation force.

Figure 5-9 shows two photographs of the gas bubble evolving in the micro reaction chamber during the expansion phase (Figure 5-9 (A)) and the reduction phase (Figure 5-9 (B)). Because of the limited contrast of the photographs, the boundary lines of gas bubble are marked with dashed lines. The electrode, which is located in upper side of the dashed line with bright color, is the counter electrode (Ag/AgCl). The working electrode, which is coated by Pt black, is located in down side of the dashed line. The difference between the largest length and the shortest length of the gas bubble took, marked as “stroke”, was measured to be about 600 μm.

The volume change rate was then indirectly measured using real-time monitoring of the volume change of gas bubbles evolving in the reaction chamber. Figure 5-10 shows the measured volume response, with the period being the same as those of the pulse voltage supplied as shown in Figure 5-8(A) and Figure 5-8(B). The measured expansion rate was also estimated to be about 0.0054 µl/s at -1.5 V, and the shrink rate was measured to be about 0.00135 µl/s at 0.5 V.

To help analyze the dynamic behaviors of the system, the resistances and the double layer capacitance in the Randles equivalent circuit model of the micro reaction cell were measured using electrochemical impedance spectroscopy with a pulse input. The capacitor was measured to be about 1200 μF, solution resistance $R_1$ was measured to be about 480 Ω, and the polarization resistance $R_2$ was measured to be about 1740 Ω.
Figure 5-9 Bubble shape; (A) Fully expanded during 4 s at -1.5 V (vs Ag/AgCl), (B) shrink rate during 16 s at 0.5 V (vs Ag/AgCl).

Figure 5-10 Measured volume response curve with supplied current profile

Given the pulse input $E(t)$ as shown in Figure 5-8(A), both current $i(t)$ flowing across the two electrodes and the volume change of gas bubbles evolving on the surface of the working electrode can be estimated using Equations (5-15) and (5-17), respectively. Figure 5-11(A) shows the simulated current curve and Figure 5-11(B) shows the theoretical volume change of the evolving gas bubbles.

Comparing the simulated current curve in Figure 5-11(A) with the measured current curve as shown in Figure 5-8(B), it can be seen that the equivalent system model is truly a first order one. The model can be used to predict the dynamic behavior reasonably well. The simulated current response curve and the measured one matched very well.
The predicted oscillation range of the volume changes and the measure ones are also very close. However, there is a significant difference between the experimentally measured and the simulated volume change curves. As can be observed from the measured volume response curve shown in Figure 5-10, the volume oscillates between 1.225 μL and 1.45 μL during the operation in steady state with an offset of 1.34 μL. In other words, as soon as electrical signal supplied to the actuator, bubble was evolved in the chamber in the transient stage never completely reversed. This can also be seen in Figures 5-9 (A) and (B), the bubble sizes oscillated between the two states. The simulated volume change curve shown in Figure 5-11(B), however, does not have such steady state offset. One way to increase the range of the bubble expansion-reduction stroke was to reduce the cycling frequency. Experimental results proved that reduced cycling frequency of the control electrical signal resulted in further reduced bubble size in reduction state, but not completely elimination.

There may be several possible reasons for this phenomenon. First, though the actuator was designed to operate on generation and elimination of hydrogen gas
molecules by supplying voltages in +0.5V and –1.5V, there are always a small amount of oxygen molecules generated in the chamber and can not be eliminated. At the same time, as soon as the electrical signal is supplied to the electrodes of the actuator, gas bubbles started to evolve in the reaction chamber, but some of the micro-sized bubbles may have drifted from the electrodes and move into the solution in the chamber. This may contribute to some residual gas bubbles in the solution. These small bubbles drifted in the solutions can not receive electrons for reduction reaction. The equivalent solution resistances then increased dramatically. The real solution resistance may therefore be much larger than R_1 measured using electrochemical impedance spectroscopy. This caused a significant drop of the current flowing through the electrodes. Because the reversible electrolysis process is dependent on the current to supply the free electrons, the reduced free electrons for reduction reaction resulted in less than expected gas bubble shrinkage. The reasons for reduced current may also include the effects of electric property variations of the solution in the reaction, such as ion concentration, pH of the solution, and all other factors that affect current flowing across two electrodes.

Another possible factor that may affect reversibility of the electrolysis process may be the two electrodes design of the electrochemical actuator. In general, the current supplied needs to be kept low with only two electrodes those are used in the actuator. Otherwise the potential difference may not be well controlled due to drifting or polarization of the quasi-reference electrodes when the current was not low enough.

To apply the electrochemical actuation based microactuator in microfluidic systems or other types of applications, it is often required that the fluid rate can be controlled in a closed-loop fashion. From the simulation and experimental results shown in this section, it can be seen that manipulation of the flow rate can not be easily achieved by a simple
voltage control approach. This is mainly a result of the variable resistance of the flow body in the reaction chamber resulting from electrolysis. Current control is therefore the preferred approach for flow rate control.

In the microfluidic system, the deflection of the circular membrane that is used for active valve can be calculated simply as following equation (5-18);

\[ w_{\text{max}} = \frac{3W(m^2 - 1)a^2}{16\pi Em^2h^3}, \quad (5-18) \]

Where, \( a \) is a radius of the membrane, \( m \) is a reciprocal of Poisson ratio, \( h \) is the thickness of the membrane, \( W \) is load, and \( E \) is Young’s Modulus number. The material properties and dimensions of PDMS membrane are shown in Table 5-1.

The membrane deflection curve along with built pressure was plotted in Figure 5-12 based on the equation (5-18) and parameters. Since the deflection occurs when built pressure starts to overcome atmospheric pressure, the range of interested pressure is set from near 100KPa to 300KPa.

<table>
<thead>
<tr>
<th>Table 5-1: Parameters for deflection calculation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Parameters</td>
</tr>
<tr>
<td>-----------------------------------------------</td>
</tr>
<tr>
<td>a: Radius of the membrane</td>
</tr>
<tr>
<td>( \nu ): Poisson ration of PDMS</td>
</tr>
<tr>
<td>h: Thickness of the membrane</td>
</tr>
<tr>
<td>E: Young’s Modulus of PDMS</td>
</tr>
</tbody>
</table>
Figure 5-12 The curve for built pressure vs membrane deflection

The height between valve seat and PDMS membrane is 100 µm. The graph shows PDMS membrane can achieve sufficient deflection that is enough to close the valve when if the pressure inside valve chamber is built around 270 KPa.
CHAPTER 6  FABRICATION TECHNIQUES

6.1 Introduction to Photolithography

Basically photolithography is using similar process that is used in picture development of photography. In photolithography, a photosensitive plastic is used to produce distinct patterns by exposing to light sources. The chemical change of the photosensitive polymer occurs when particular frequencies of light are absorbed. Since these polymers have a high photosensitive, the patterning by selective exposure to light followed by immersion in a chemical can be achieved by removing unexposed or exposed areas that has low molecular weight material. This process is often called ‘development’. The high molecular weighted polymer regions are only remained with a sharp pattern.

Two types of photosensitive polymers exist. Positive photosensitive polymers undergo scission in which molecular weight of the polymer chains is dramatically reduced when light is absorbed. Negative photosensitive polymers cross-link and increase their molecular weight during exposure.

A pattern transfer from the mask to a substrate via etching or adding a hard material onto the substrate using various deposition techniques is a typical application of photolithography. These processes often require the exposed polymer to be resistant to temperature variations, acids, bases, and vacuum. Polymers that show such resistances and can be patterned by exposure to light are called resists.

Spin coating is a typical method to cast the resist onto the substrate because most of them are produced in liquid form. A Centrifugal force in the spinner is used to spread the
liquid evenly across a smooth substrate while allowing for rapid evaporation of the solvent. After spin coating, the substrate is baked to remove the remainder of the solvent. A uniform solid resist coating on the substrate by these sequential processes. For solid resists, such as PMMA used in deep X-ray lithography, direct bonding of the plastic to the substrate is often performed.

6.2 Introduction to LIGA

Since high-aspect-ratio metallic devices with critical dimensions of a few microns could not be produced with traditional machining techniques such as a lathe or milling machine, the LIGA process was developed as an alternating technique. The traditional process requires the exposure of resist to X-ray radiation using a highly collimated X-ray source called a synchrotron. A conductive substrate is used for electroplating metal to form the pattern. This patterned metal is used as a master for the injection molding or hot embossing of many plastic parts. The plastic molds can be used as final products or as electroplating molds for further metallic devices as needed.

The X-ray LIGA has some excellent advantages over many MEMS technologies such as the production of bulk metallic microdevices, highest height to width aspect ratios (structures up to 3 mm tall with aspect ratios of 100:1), multi layer and angled patterns for 3-D microdevices.

However, the cost of producing micro-parts using synchrotron radiation is considerable. Only 5 synchrotrons with operational beamlines used for deep X-ray lithography of thick resist are in the United States. For these reasons such as very limited places, large initial investment has prevented the LIGA process from becoming a
mainstream industrial technology in the MEMS market, even though it has excellent advantages in MEMS fabrication.

6.3 UV-LIGA Technology

For two decades, UV-LIGA technology is widely used in MEMS process because it has couple of advantages over conventional MEMS processes based on silicon technology. First, it can achieve any lateral shape up to several hundreds of micrometers with near vertical profile. Second, the structure materials such as plastics, metals, alloys, and ceramics or a combination of these materials can be used in fabricating microstructures. In microfluidic devices, some other materials needed to fabricate together as a structure, sensor, or actuator. As point of this view, UV-LIGA technology is very suitable for the micropump and microfluidic fabrication. Finally, these complex microfluidic systems are demanding simple and low-cost processes. Compare to X-ray LIGA, UV-LIGA does need to relatively simple equipments such as UV exposure, pattern generator while the X-ray LIGA needs expensive X-ray mask and X-ray beam lines. The cost of fabrication can be reduced significantly by using the ultraviolet light source and thick negative photoresist in UV-LIGA. Another advantage of UV lithography over X-ray lithography is an exposure time. Even though very thick resist is used in the fabrication process, the exposure process can be done on the UV station within five minutes while X-ray requires an expensive exposure station and a couple of hours to finish that process. Figure 6-1 shows the basic steps involve in UV-LIGA process.

Multi-layer, multi-step UV-LIGA processes were used to fabricate microfluidic devices (MHD micropump, electrochemical microactuator) in this paper.
6.4 UV-LIGA Processes

6.4.1 SU-8 Negative Photoresist for UV-LIGA

SU-8 (MicroChem Corp., Newton, MA) photoresist was selected to fabricate the microfluidic system because of its relatively high aspect ratio capabilities and can be exposed with near-UV light source using standard UV printing technology. SU-8 is an epoxy-based, ultra thick, negative tone photoresist used in fabricating high aspect ratio MEMS structures with single or multiple spin-coatings. This photoresist can be spin-coated to thicker than 2 mm, and aspect ratio more than 20 with a standard contact lithography equipment. Moreover, in X-ray lithography, the thickness of the resist can be up to 3-5 mm, and the achievable aspect ratio can be more than 60. These results are due to the low optical absorption in the UV range. For example, 100% absorption occurs for 2 mm thickness in the 365nm-wavelength. Of course, x-ray LIGA still yields better results at a higher processing cost. In addition SU-8 is well suited for acting as a mold for
electroplating because of its relatively high thermal stability \(T_g > 200 \, ^\circ\text{C}\) for the cross-linked resist). Therefore, UV-LIGA process by SU-8 is very suitable for low-cost application.

To achieve a good quality for high aspect ratio and sidewall in UV lithography of SU-8, there are two major factors that directly affect to them. First factor is a use of a light source with suitable wavelengths. Another is significant diffraction problems due to low uniformity caused by the difficulty in spin-coating uniform and thick resist on a substrate.

One of the key requirements to achieve the best lithography results of SU-8 is exposing the resist with the proper light spectrum.\textsuperscript{58,59} The photo initiator content added in the SU-8 dominate UV absorption of SU-8 resist. The wavelengths under 350 nm result a very high absorption in the SU-8 resist. Shorter wavelengths have a high absorption rate at the surface of the resist while longer wavelengths have a much lower absorption rate, and tend to be able to penetrate deeper and can be used for exposure of thick resist. When a wide spectrum light source used for the exposure, only the lower frequency (longer wavelength) light source will be able to penetrate deep into the resist while high frequency light is absorbed at the surface layer of the resist. High frequency UV light produces a skin effect on the resist, a layer of highly cross-linked polymer with a thickness ranging from two to a few tens of microns when the surface layer was over exposed. This effect results in distorted pattern at near the surface that makes low quality microstructures. To eliminate unwanted wavelengths and overcome the skin effect in SU-8 structures, Optical filters are used in exposure process.\textsuperscript{58,59}
The diffraction effect is the second critical factor for lithography of thick SU-8 resist that also applied to any other lithography processes. Extended contact with a non-level surface prior to exposure result 5-10% error in the resist thickness across a 4” substrate in most thick SU-8 films. The error in resist thickness causes gaps between the masks and the substrate during exposure which are arguably present in almost all thick SU-8 lithography results presented to date. There are two methods to eliminate gap effect: 1) Make uniform surface by mechanical machining such as fly cutting machine, 2) use glycerin that can fill the gap during exposure process.

The fly cutting machine can make very uniform surface with a few microns by cutting soft baked SU-8 surface. To remove machining marks on the top surface, annealing at the hot plate with lower temperature of Tg of SU-8 is needed after fly cutting. Glycerin has nearly the same refractive index as SU-8. Some research results showed a significant drop in diffraction during the exposure. Structures produced using glycerin showed significant improvement in the pattern quality of 100 μm SU-8 exposures.

The residual stress in baked SU-8 films is the other challenge to overcome. The large exposed field of a thick layers of SU-8 resist those retain so much residual stress under the given parameters from the manufacturer is often de-bond from the substrate, bow or even lead to cracks in silicon substrates after exposure. Typical SU-8 procedures for post baking is cooling rapidly from 96 °C to 65 °C, held for a few minutes and then removed from the heat source and cooled to room temperature. The internal stress built up in thick resist films does not fully removed by this procedure.
The optimized post baking process for ultra thick high aspect ratio SU-8 microstructures is reported in this chapter. A multi layer spin coating process was also developed to coat uniform SU-8 resist for a thickness up to 1.1 mm.

6.4.2 Basic SU-8 Processing Steps

SU-8 processes applied to this research work were performed in CAMD*. The procedures for SU-8 process were well understood and standardized by CAMD for several years. Therefore, most processes applied to this research were based on CAMD work. However, some processing conditions were modified to improve the quality of devices, and summarized in the following sections.

i. Pattern generation of optical mask

Three 5-inch by 5-inch optical masks, chrome on glass with 1000 Å of chrome and 1 μm thick AZ 1518 positive photoresist coating (Nanofilm, Westlake Village, CA) were required based on the micropump designs using SU-8 negative photoresist and the UV-LIGA method. The mask patterns were created based on guidelines provided by Photo Sciences Inc. (St. Torrance, CA) to facilitate the conversion of an AutoCAD drawing to binary format that could be fractured into data read by the GCA Mann 3600 Optical Pattern Generator (OPG), (GCA Corp., Burlington MA). The fractured data could be viewed in the PGCAM software (Artwork Conversion Software, Inc., Santa Cruz, CA) to ensure that the fractured data was being output to the OPG correctly before the actual run.

The GCA Mann 3600 OPG at CAMD can pattern standard 5-inch by 5-inch mask plates with a high-pressure mercury arc lamp as illumination source with a minimum feature size down to 4 μm with an accuracy of 0.1 μm. The light is filtered and projected

* Center for Advanced Microstructures and Devices, Louisiana State University, Baton Rouge, LA 70806
onto a shutter which controls the duration of the exposure. The mask plate is secured onto a fine motion stage, which positions the plate at each exposure location. A set of aperture blades (vertical and horizontal), that can be rotated from 0 to 90 degrees creates rectangular images of varying sizes and orientations to form the pattern design. A focusing column images the apertures onto the mask plate.

After patterning by the OPG, each optical mask was developed in Microposit 354 positive developer (Shipley Company, L.L.C, Marlborough, MA). The developing time for each mask was determined visually based on the amount of areas being developed and the age of the developer. Typically, development took approximately 2 to 3 minutes with gentle agitation. After development, the mask was rinsed thoroughly in a DI water bath for 3 minutes to remove all the residue and developer.

The mask development process was followed by wet etching of the chrome metal layer. Commercially available chrome etch (Olin Microelectronic Materials, Norwalk, CT) which consists mainly of nitric acid was used to etch away the chrome metal layer not covered by the AZ 1518 positive photoresist after development. Again, the chrome etching time for each mask was determined using the visual inspection method under the microscope (Nikon, Optihot-88) such that when the chrome layer pattern was fully etched, the designed opening areas of the substrate became optically transparent. The etching time typically took approximately 1 to 2 minutes with gentle agitation. The mask was then rinsed thoroughly in a DI water bath for 3 minutes to remove the chrome etch and to prevent the possibilities of over-etch.
ii. Substrate preparation

The process starts with a 4-inch diameter substrate such as silicon, silicon oxide, or glass substrates. Even though SU-8 adheres well to silicon, SiO$_2$, aluminum, Al$_2$O$_3$, and gold films very well, it has poor adhesion to most commonly used metals. Patterning the metal layer or increasing the surface roughness so that the substrate has a better mechanical hold on the resist are used to overcome this limitation. In our experiment, a 1 mm thick, 4-inch diameter SiO$_2$ substrate was used to develop this process. This substrate was chosen for two reasons: the mechanical strength and the surface roughness. No significant deflection of the substrate was observed due to residual stress in the SU-8 film during process development.

Dehydration bake was performed at 200°C for 1hr to improve the adhesion of the SU-8 on the substrate. For better adhesion with the substrate, adhesion promoter for SU-8, OmniCoat, was sometimes spun on the substrate, followed by SU-8 coating. The detail process information for OmniCoat is available from the MicroChem Inc.

iii. Spin coating

Headway Research PWM101 Light Duty Photoresist Spinner was used for spin coating. The thickness versus spin speed characteristics of four different MicroChem SU-8 formulations for 30 second spin time is shown in the spin curves in APPENDIX A1. To obtain 1100 µm of SU-8 microstructure for PDMS mold on the substrate, 10 ml of SU-8 was applied to the center of the substrate and spun at 400 rpm for 25 seconds. Excess resist was removed from the edges of the substrate prior to baking to prevent resist overflowing off of the substrate during the pre-exposure bake (PEB) process. In order to minimize it, all samples also were put on a flat plane for 1 to 2 hours at room temperature after removing excessive resist from the edges of the substrate. A slope of only 1° in pre-
exposure baking on a surface can cause significant error in the uniformity of the resist, and in extreme cases, leads to over-flow of the resist off of the substrate. A short relaxation period should be used prior to exposure to reduce any residual stress.

iv. Fly cutting

Once again, flatness of coated SU-8 is very important because non-uniformity of the resist coats yields exposure non-uniformity in later processing. Fly cutting was performed to achieve a few microns of uniformity for ultra thick SU-8 layer. The fly cutting machine is a milling machine with leveling ability within ±5 microns over a 4” diameter substrate. PMMA is the most commonly leveled material. Pre-exposed optical resists, such as SU-8, can also be milled with this machine since the area excludes UV light by UV protecting curtain. The fly cutting machine is shown Figure 6-2.

Figure 6-2 The Fly cutting machine

After fly cutting, the surface of the substrate with spin coated SU-8 was flattened by putting sample on the hotplate near Tg of SU-8 resist.

v. Pre-exposure bake (Soft bake)

The temperature-controlled flat and horizontal hot plate (Figure 6-3) was used in this step. The SU-8 coated substrate was placed on the hot plate for 1 hour to reduce the
internal stress, and then the hot plate was ramped from room temperature to 96°C over a 30-minute time interval, and held for specific period of time depending on the thickness of SU-8. In this step, all solvent in the SU-8 layer is removed. Our experiments have shown that each 100 μm of resist thickness of SU-8 requires a 50 minute bake time at 96 °C. The resist was cooled with a constant ramp rate or multiple temperature steps to minimize residual stress at the end of the bake process. An additional coating was made to achieve the desired resist thickness when necessary.

![Figure 6-3 Horizontal hot plates](image)

An extended pre-baking time for each additional layer is required in multiple coatings. A hot plate temperature of 100 to 105 degrees Celsius may be required to obtain 96 °C on the top surface of the resist when a thick substrate with poor thermal conductivity or a very thick SU-8 layer is used. Figure 6-4 shows the expected bake time of SU-8 negative resist as a function of thickness in a convection oven.

**vi. Exposure**

The exposure was performed using a Quintel UL7000-OBS Aligner with a 1000 W Hg lamp A cross-linking of the SU-8 photoresist is promoted by exposure to UV light . The required dose depends on the thickness of SU-8. One can see that the exposure doses depending on the thickness of SU-8 from Table attached in APPENDIX.
These exposure doses for film less than 500 μm thick were established at CAMD and verified by numerous experiments. A thorough inspection of the absorption characteristics of the SU-8 resist was provided by the CAMD work. A broad-band UV light between the wavelengths of 350 and 550nm would provide the best exposures for SU-8 resist by the experiments.

An overexposure tends to happen at the surface of the resist due to the excessive absorption of shorter wavelength components because light components with shorter wavelengths have much higher absorption rates than the longer wavelength ones. The stepwise illumination was also performed for high exposure doses to avoid over-heating related errors with filtering of shorter wavelength components. For example, the SU-8 was illuminated for 40 seconds, cooled down for 40 seconds, and illuminated again. The presence of low power density of shorter wavelengths below than 350 nm is resulted in leaving minor skin effects on the surface, but any additional filters were not used to optimize it. The use of filter that eliminates wavelengths other than 350 – 400 nm can
improve the lithography quality.\textsuperscript{61} The pressure contact was performed in exposure process because of the existing of non-uniformity of the top surface of the thick SU-8 layer and some limitation of the equipment for using vacuum mode. The contact force was carefully controlled because excessive or lower contact force result in either adhesion between SU-8 and mask or more diffraction due to air gaps between the mask and the substrate in non-contact regions.

Some different doses were applied to exposure ultra-thick films. The dosage requirements for different patterns on the same substrate are significantly different as expected with all UV resists. Significantly higher dosages are required to open field structures, such as crosses and squares, than close field holes in the resist. Similarly, cylindrical structures required different dosages and development parameters than standard open field structures.

\textbf{vii. Post-exposure bake (PEB)}

The illuminated SU-8 is polymerized through a cationic photo-amplification mechanism during this step. The recommended PEB process by MicroChem is shown in APPENDIX A4. All samples were ramped from room temperature to 95\textdegree C. In order to minimize internal film stresses, an intermediate bake was performed at 60\textdegree C depending on the film thickness before allowing the wafers to cool to room temperature.

\textbf{viii. Development}

The standard SU-8 Developer was used at room temperature for development process. The required development time in general is proportional to the resist thickness. The substrate was rinsed with isopropyl alcohol after the development. If white traces
remain on the surfaces, it indicates that the development is not finished, and then the substrate has to be put in the developer again.

6.4.3 PDMS Molding

To construct complex 3-D structure made of PDMS membrane, microfabrication technique for stacking multi layers of SU-8 mold was used. The basic process of the PDMS mold technique is shown in Figure 6-4.

![Figure 6-5 PDMS Mold Fabrication](image)

A curing agent and PDMS prepolymer (SYLGARD 184 Silicone Elstomer Kit, Dow Corning, Midland, Mi) were mixed in a 1:10 weight ratio. Degassing was done after mixing in the mechanical vacuum chamber at 20 – 50 mtorr for 1 hour to remove any air bubbles in the mixture. The mixture was poured onto master mold made of SU-8 layers, and excessive PDMS was removed using razor knife. The PDMS premixture was cured for 3h at 75°C. Temperature was ramped up to set point and cooled down to room temperature again.
6.4.4 Copper Electroplating

Copper was used as the electrode material because it has desirable electrical properties of low resistivity and has good adhesion properties with gold that eventually electrodeposited to copper surface. Table 6-1 shows the contents of the cooper plating solution per 1 liter.

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>De-ionized Water</td>
<td>880 ml</td>
</tr>
<tr>
<td>Cooper Sulfate</td>
<td>48 g</td>
</tr>
<tr>
<td>Sulfuric Acid</td>
<td>100ml</td>
</tr>
<tr>
<td>30% Hydrogen Peroxide</td>
<td>3.5ml</td>
</tr>
<tr>
<td>Hydrochloric Acid</td>
<td>0.15ml</td>
</tr>
<tr>
<td>PC-65 Brightener</td>
<td>10ml</td>
</tr>
</tbody>
</table>

The tank is made of corrosion resistant High-Density Polyethylene which contains the chemicals along with the anode and cathode. The top is covered with two pieces of Polymethylmethacrylate (PMMA). These were cut to form a tight fit on the top allowing for the anode and cathode to be inserted and suspended into the tank.

The anode basket is made from titanium which will resist corrosion in the acidic plating solution. ½” Headed Copper Ball is filled in the anode basket. The basket is hung from the cover at one end of the tank perpendicular to the long walls. The anode bag that
made of a close-knit polypropylene fabric is covered tightly around the basket. An alligator clip used for an electrical connection near the top of the basket.

The cathode is constructed of a thin PMMA sheet cut to a size and shape sufficient to contain the sample. The copper clip used as an electrical connection. The clip is fastened and tightened to hold the sample using a nylon bolt and screw from top of the tank, and positioned opposite the anode at a distance varying between 10 and 15 cm. The power supply is a variable output DC source that capable of producing a clean current with low ripple and noise (<1mv rms). Constant current is required for good electroplating results.

The following sequences are bath make-up procedures.

(1) Fill the precleaned and leached carbon treatment tank to 75 % of the final volume with distilled or de-ionized water. Add the required amount of Copper Sulfate and mix thoroughly. Liquid Copper Sulfate Concentrate may be used without the necessity of the solution purification.

(2) Add the required amount of Sulfuric Acid to the solution with agitation.

(3) Add 2.5 ~ 5ml of 30 % Hydrogen Peroxide per 100 gallons of the premixed solution with agitation. Heat to 60° C and continue agitation for 2 hours.

(4) Add 1ml of Hydrochloric Acid per 100 gallons to raise the chloride concentration to 50-75 ppm.

(5) The copper anodes should be washed free of dirt and organic films using an alkaline cleaner.

(6) Add 1 % by volume PC 65B Brightener. 0.5 ml of Replenish brightener (PC 65R Replenisher) should be added by every Ampere hour for makeup.
6.4.5 SU-8 Removal

For the fabrication of MHD micropump, it needs a removal process of SU-8 structure after electroplating. The removal of SU-8 is extremely difficult with conventional solvent based resist strippers because SU-8 has been designed as a permanent, highly cross-linked epoxy material. The manufacturer suggest using Remover PG for minimally cross-linked SU-8 structure. For fully cured or hard baked SU-8, only use of Omnicoat (Resist stripper, Microchem) with Remover PG can remove it using lift-off process. Even though their suggestion about removal technique using Omnicoat, that also resulted a debonding problem when the sample was in developing process. Other suggestion for stripping SU-8 is using oxidizing acid solution such as piranha etch, plasma ash, RIE, laser ablation and pyrolysis. Dynasolve 185 (Dynaloy, Indianapolis, IN) was chosen for stripping SU-8 because of a limitation of equipment and Lab experimental condition. The major usage of Dynasolve 185 solvent is for deflashing, depotting, deinking, and other epoxy removal. It contains a proprietary blend of ingredients that facilitate polymer removal and prevent the depositing of particles back onto a part. Dynasolve 185 will not attack most metals used in electronic components including aluminum. This property is very useful because electroplated copper electrodes should be remain or not affected in removal process. It also disintegrates many epoxy-molded devices including UV-cured epoxies.

To remove SU-8 plating mold, the sample was immersed in Dynasolve 185 solution at 80°C for 30 minutes after electroplating. Relatively big area was started to peel off from the substrate, but specific area such as the gap area between two electrodes was removed by peeling it off using sharp tip tweezers.
CHAPTER 7  MICROFABRICATION OF MICROFLUIDIC SYSTEM

7.1  MHD Micropump

Prior to fabricate the MHD micropump that uses full fabrication steps in previous chapter, prototype of MHD micropump has been fabricated to test its feasibility of theory as explained in chapter 2. After successful test on the prototype, the MHD micropump was fabricated by fully utilizing microfabrication steps. All the components are fabricated in 20 mm X 12 mm rectangular with a height of 500 um SU-8 structures and electrodes. The channel width is also 500 um. Since the prototype of electrochemically actuated micropump that is for testing bubble actuation was fabricated without valves, both pumps have a same structure, and fabrication steps except electrode material.

7.1.1  The Fabrication of the Prototype MHD Micropump

The prototype of the MHD micropump is composed of simple structures such as SU-8 for chamber and channel, two electrode metals, and PMMA cover as seen in Figure 2-2. The diameter of the metal wires those are inserted and served as electrode is 500 um. The processes for SU-8 fabrication were optimized to this thickness.

A 4-inch diameter glass wafer (Capitol City Glass, Baton Rouge, LA) was used as the substrate. The glass substrate was first cleaned with standard Acetone/IPA/DI water cleaning method, then blown dry using nitrogen (N2) and baked in a mechanical convection oven at 95°C for 15 minutes to remove the residual moisture. To further remove any residue and promote good adhesion between the glass substrate and the thin films during the subsequent depositions, the glass substrate was cleaned in oxygen (O2)
plasma asher (Branson International Plasma Corp., Hayward, CA) for 15 minutes at 600W.

Two thin film metal layers were physically evaporated onto the glass substrate using Temescal Model BJD-1800 E-Beam Evaporator (BOC Edwards, Wilmington, MA). First, a thin chromium (Cr) layer, 200 Å in thickness that served as an adhesion layer was evaporated at 1 Å/sec onto the glass substrate, followed by evaporation of a second thin gold (Au) layer 1000 Å thick at 5 Å/sec to promote adhesion property with SU-8. The glass substrate with the evaporated Cr and Au thin film metal layers was further heat-treated (annealed) in the mechanical convection oven at 200°C for 10 hours to ensure good adhesion for the subsequent processes.

7.1.1.1 Adhesion Base Patterning

To realize base layout at a specific location on the glass substrate, a 1.5 μm layer of S1813, positive photoresist (Shipley Corp., Marlborough, MA), was spin-coated on the glass substrate using the light duty spinner (Headway Research Inc., Garland, TX) at 3000 rpm for 30 seconds, then pre-baked at 120°C for 2 minutes to evaporate the solvent.

The first optical mask was used to pattern the positive photoresist under the UV light source using a Quintel UL 7000 1000W Hg lamp (Quintel Corp., San Jose, CA) for 6 seconds. For hardening purposes and to resist chemical attacks during the metal etching processes, the exposed positive resist had a stress relaxation at room temperature for 1 hour. After stress relaxation, the positive photoresist was developed in 454 positive developer (Shipley Corp., Marlborough, MA) for 1 minute, followed by 1 minute rinse in DI water. Visual inspection method under the microscope was used to determine the completion of the development.
The Au seed layer not covered by the positive photoresist was etched using a commercial gold etch (Olin Microelectronic Materials, Norwalk, CT), which consists mainly of sodium iodide and iodine, with gentle agitation. The process was followed by rinsing the glass substrate in the DI water bath thoroughly for 2 minutes; the underlying Cr layer was etched subsequently using a commercial chrome etch (Olin Microelectronic Materials, Norwalk, CT) with agitation and followed by rinsing in a DI water bath thoroughly for 2 minutes. The remaining S1813 photoresist was stripped by immersion in an acetone bath for 1 hour.

7.1.1.2 SU-8-50 Negative Photoresist Process

Commercial SU-8-50 photoresist (MicroChem Corp., Newton, MA) was selected to be the major process photoresist for the AC MHD micropumps because of its capabilities for rapid prototyping and a single spin-coat layer up to a thickness of 600 μm. SU-8-50 also shows a very promising functionality in terms of its high degree of cross-linking, which produces good sidewall profiles, high aspect ratios, and good adhesion. It has also been investigated as a possible method to make LIGA-like micromolding material at CAMD, LSU. Given all the advantages of SU-8, however, the processing parameters have proven to be very sensitive to various designs and applications in MEMS.

As a result of that, the process parameters ranging from spin-coat, pre-bake, exposure dose, post-bake to development time were derived from the works done by CAMD researchers on determining the improved and optimized processing parameters.61 Few modifications of the optimized processing parameters were necessary in order to establish an optimized and repeatable lithography process for a thickness of 500 μm in microfabricating the prototype of the micropump.
The glass substrate with the patterned adhesion base was cleaned using Acetone/IPA/DI water and blown dry using N₂ blowing gun; the process was followed by O₂ plasma cleaning at 600W for 8 minutes to fabricate main structure such as inlet/outlet reservoir, channel, and chamber.

A 500 μm thick layer of SU-8-50 negative photoresist was deposited by spin-coating at 500 rpm for 20 seconds. The SU-8-50 photoresist was pre-baked at an initial temperature from 40° C, and ramped up to 95° C at a rate of 5~8° C/min; the photoresist was held at this temperature for 6 hours to evaporate the solvent. The substrate was then cooled to room temperature in the oven (approximately 3 hours). The second optical mask was aligned with the patterned wiring layout/electroplating base with alignment marks using UV exposure station (Quintel UL 7000 1000W Hg lamp) equipped with aligner. The SU-8-50 photoresist was exposed at a power of 23 mW/cm² for 70 seconds, which corresponds to a dose of 1610 mJ/cm². The exposed SU-8-50 photoresist was post-baked from 40° C and ramped up to 95° C at a rate of 5~8° C/min, and maintained at that temperature for 20 minutes. The heat treatment was followed by lowering the temperature to 50° C and baked for 2 hours. The substrate was then cooled to room temperature in the oven (approximately 2 hours). The unexposed SU-8-50 photoresist was developed in SU-8 developer (PGMEA) (MicroChem Corp., Newton, MA) for approximately 10 minutes in an ultrasonic bath, followed by rinsing with fresh SU-8 developer. The glass substrate was thoroughly rinsed with IPA for 30 seconds, followed by rinsing in DI water bath for 30 seconds and blow-drying with N₂. Figure 7-1 is a photograph of fabricated SU-8 structure on the patterned base layer.
7.1.1.3 Assembling Process

After fabrication, the platinum electrodes were shaped and inserted, and then PMMA cover was bonded on top of the SU-8 surface using epoxy bonding. Electrical connection was soldered to each electrode.

7.1.1.4 Fabrication Sequences

The fabrication sequences are outlined below; the schematic diagram of the sequences is shown in Figure 7-2.

(1) A 4-inch diameter glass wafer was used as the substrate. The substrate was evaporated with Cr (200 Å in thickness) as an adhesion layer and then Au (1000 Å in thickness) as the electroplating base.

(2) S1813 positive photoresist (1.5 μm in thickness) was spin-coated on the glass substrate.

(3) The resist was pre-baked to evaporate its moisture in the hot plate and cooled down to room temperature.

(4) Exposed the resist under UV light source using the first optical mask.
(5) Developed the exposed photoresist for 1 minute.

(6) Etched the Au layer. The Au electroplating base not covered by the exposed positive photoresist was etched using a commercial gold etch with gentle agitation.

(7) The underlying Cr layer was etched subsequently using a commercial chrome etch with gentle agitation.

(8) The remaining photoresist was stripped in an acetone bath, leaving adhesion base and alignment marks on the glass substrate.

(9) Spin-coated 500 μm layer SU-8-50 negative photoresist.

(10) Pre-baked (multi-step heat treatment) to evaporate the solvent and cooled down to room temperature in the oven.

(11) Exposed the SU-8-50 negative photoresist using the second optical mask under the UV light source equipped with aligner.

(12) Post-baked (multi-step heat treatment) to anneal the photoresist into its most stable state and cooled to room temperature in the oven.

(13) Developed in SU-8 developer using ultrasonic bath.

(14) Prepared electrodes were inserted and fixed using epoxy resin.

(15) An epoxy layer was spread on a 2 mm thick PMMA plate, which was bonded on top of the wafer.

(16) Two 2.0 mm diameter holes were drilled directly above the reservoirs to serve as the inlet and outlet connections for each micropump.
Figure 7-2  A schematic of fabrication for prototype MHD pump; (a) - (f): fabrication step (1) – (8), (g) – (i): step (9) – (13), (j): step (14), (k): step (15), (16)
Figure 7-3 is a picture of fabricated prototype Magnetohydrodynamic (MHD) micropump.

![MHD Micropump](image)

Figure 7-3 MHD micropump after assembling with electrodes and PMMA cover

### 7.1.2 MHD Micropump with Electroplated Electrodes

In this process, the substrate was drilled by water jet machine to make two reservoirs. The same fabrication steps from (1) to (13) in previous section are used for the MHD micropump. Copper electroplating was performed after fabrication of SU-8 structure, and all structures were leveled using fly cutting machine to remove excessively plated copper and make desired height. Then SU-8 layer was removed by using Dynasolve 185 solution. After removing electroplating structure, the micropump was fabricated repeating SU-8 process. A thin gold layer was added on the surface of the cooper electrodes to protect from the corrosion during operation of the micropump with various solutions. Each fabrication step is shown in Figure 7-4. Figure 7-5 shows fabricated MHD micropump.
(A) Drill holes on the glass substrate by water jet  
(B) Evaporating & Patterning Au/Cr Layer  
(C) SU-8 structure for Electroplating  
(D) Electroplating Cu and leveling by fly cutting  
(E) SU-8 stripping  
(F) Patterning SU-8 for final structure  
(G) Assembling with PMMA cover  

Figure 7-4 Schematic diagrams of the MHD micropump using electroplating
7.2 Electrochemical Actuator

As it was mentioned in first section of this chapter, basically a prototype electrochemical actuator has same structure as a prototype MHD micropump. Two different electrodes, which are platinum black and Ag/AgCl, are inserted to SU-8 structure instead of platinum electrode. The microfluidic system that based on electrochemical actuation has built with multiple processes using UV-LIGA technologies.

7.2.1 The Fabrication of a Prototype Electrochemical Actuator

Same fabrication steps, which were used in a prototype MHD micropump, were used for a prototype electrochemical actuator. Pt black was chosen for working electrode, Ag/AgCl was chosen for counter electrode. Both electrodes were prepared by electroplating from platinum wire and silver wire. Figure 7-6 shows the schematic of platinum black coating setup.

Figure 7-5 A photograph of the UV-LIGA fabricated prototype MHD micropump
Platinizing solution was prepared by dissolving 3 g chloroplatinic(IV) acid hexahydrate \( \text{H}_2[\text{PtCl}_6] \times 6 \text{H}_2\text{O} \) and 30 mg lead acetate in 100 mL distilled water. Before used, the new platinum electrode was degreased and etched for a few minutes in hot aqua regia (50 °C) (3 parts conc. HCl and 1 part conc. HNO3) to ensure proper adherence of the platinum black coating. Fume cupboard needs to be used and the necessary protective measures (safety glasses, lab coat and gloves) need to be taken. After plating work is done, the samples need to be rinsed well with distilled water.

Platinum electrode being coated was connected to the negative pole as shown in Figure 7-6. Another platinum electrode was connected to the positive pole as auxiliary electrode. The current was slowly increased by reducing the variable resistance until the electrode being coated begins to blacken and a slight gas evolution becomes visible (the actual current depends on the electrode surface or on the cell constant of the conductivity measuring cell). After about 5 minutes, by which time the coating should be a deep black color, the current was turned off. Afterwards the electrode was rinsed well by DI water.

Finally, electrolyzing was done in a 2% solution of sulfuric acid, using the same procedure as for the platinization. The current was adjusted with the variable resistance.
so that a strong gas evolution occurred. With conductivity measuring cells, the polarity was reversed a few times. The electrode was rinsed well with distilled water and leave immersed in water overnight.

Figure 7-7 shows two SEM images of surface of the platinum electrodes coated with platinum nano-particles. The large number of platinum nano-particles coated on platinum electrodes dramatically increased the effective surface area of the electrode, and this in turn helps to enhance the electrochemical reaction. The effective rate of the reversible electrolysis process can therefore be enhanced, leads to significantly faster operation rate ECM actuators.

![SEM images of platinum black nanoparticles on platinum electrodes: (A) magnification at x 15,000, (B) magnification at x 30,000](image)

Ag/AgCl counter and reference electrode was electrolytically deposited by the following steps. Silver electrode being coated was connected to the positive pole as shown in Figure 7-8, and platinum electrode was connected to the negative pole. The current was slowly increased until a slight gas evolution becomes visible at the platinum electrode. After 5 minutes, the current was switched off, and Ag/AgCl electrode was rinsed well by DI water.
Two electrodes were carefully inserted into the fabricated SU-8 structure after deposition of electrode materials (Pt Black, Ag/AgCl). Epoxy bond was applied to inserted electrodes to fix them, and then two holes were drilled on the PMMA cover. After bonding PMMA cover to the fabricated structure using epoxy bond, it was cured for an hour under 5lbs weight.

7.2.2 Fabrication of the Microfluidic System

7.2.2.1 Bottom Part for Electrochemical Actuators and SU-8 Base

For electrochemical actuators, two thin film metal layers were physically evaporated onto the Si/SiO₂ substrate using a Temescal Model BJD-1800 E-Beam Evaporator (BOC Edwards, Wilmington, MA). A thin layer (200 Å) chromium (Cr) layer, which served as an adhesion layer, was evaporated onto the silicone substrate. A thin gold (Au) layer of 1000 Å thickness, which served for electroplating seed layer, was then evaporated. A positive photoresist (Shipley Corp., Marlborough, MA) was used for patterning to realize the base layout at a specific location on the silicone substrate.
Since a thin film of Au/Cr layer can be damaged during electrochemical actuation, Au layer up to 3 um was electroplated using commercial gold plating solution. Then silver layers were electroplated only on the counter electrode and reference electrode. The commercial Cy-less silver plating solution and silver anode from Technic Inc. (Technic Equipment Division, Pawtucket, RI) was used for electroplating with home-made plating bath. The current density of 3 mA/cm$^2$ was applied for 30 minutes. Approximately 1.5um of silver layer was achieved, and more than 30 minutes of plating time resulted in peeling off the entire layers including Au/Cr seed layer because of plating stress. Ag/AgCl layer was electrolytically deposited from a 1 M solution of NaCl after electroplating silver layer. SU-8 (MicroChem Corp., Newton, MA) structure was patterned to protect connector line with alignment pillars after plating of counter and reference electrodes. Finally, working electrodes, which covered with plated gold layer, were selectively platinized. Platinum black coating and electrolyzing was done using the same procedures as used in a prototype electrochemical actuator. The fabrication processes explained above are sequentially shown in Figure 7-9.

7.2.2.2 PDMS membrane fabrication

The PDMS membrane structure serves not only as valve actuation but also as chamber that contains the solution to get electrochemical actuation. Three SU-8 layers were sequentially fabricated to make PDMS mold. A 300 um height of the first SU-8 layer was fabricated on the silicon substrate using SU-8 processes that was described in chapter 6 except development. Then the second layer for the actuator’s chamber processed with height of 1000 um. Finally, the third layer to fabricate PDMS membrane
was added to the substrate. After post exposure baking, the sample was developed in SU-8 developer, and carefully cleaned out by IPA and DI water.

Figure 7-9 Schematic diagrams of an electrochemical actuators and SU-8 Base: (A) Evaporating Au/Cr, (B) Patterning Au/Cr layer and electroplating additional Au, (C)Ag/AgCl layer deposition, (D) –(F): SU-8 base fabrication, (G) Platinum black coating
PDMS pre-mixture that made of 1:10 ratio of curing agent and PDMS prepolymer was poured on the master mold made of SU-8 after degassing in the mechanical vacuum chamber at 20 – 50 mtorr for 1 hour to remove any air bubbles in the mixture. The excessive PDMS was removed using razor knife, and premixture was cured for 3h at 75°C. Figure 7-10 is a schematic diagram of PDMS process.

![PDMS Diagram]

7.2.2.3 PMMA Cover Structure

The cover structure was fabricated using similar process that was used in PDMS master mold. The PMMA substrate was chosen for the cover material because it has an excellent adhesion property with SU-8 photoresist and also provides transparent view that needs to observe flow of the solution through microscope. Since Tg of PMMA is less
than typical curing temperature of SU-8, baking and curing was done at low temperature with longer time. Two SU-8 layers were used fabricated on the PMMA substrate. The outlet hole and valve seat surface were exposed in the first layer, then second layer was spun on, the channel area was exposed. After development process, the 2 mm hole was drilled on the PMMA substrate to connect capillary tube. The fabrication processes explained above are sequentially shown in Figure 7-11.

Figure 7-11 PMMA cover structure fabrication; (A) Fist layer for outlet hole and valve seat, (B) Second layer for channel, (C) Develop two layers, (D) Drill on the PMMA substrate using micro drill.

(A) Top view of SU-8 Base and Actuator       (B) PDMS mold for membrane part

Figure 7-12 Pictures of fabricated parts
(C) PDMS mold for pump chamber        (D) PDMS structure after peeling off

(E) PMMA cover structure                            (F) Assembled microfluidic system

Figure 7-13 Pictures of fabricated parts (continued from page 78)

Figure 7-13 is showing assembling process. Sequential numbers in the figure represents order of assembling process. Figure 7-14 is a picture finally assembled microfluidic system.
Figure 7-14 Assembling process of the microfluidic system

Figure 7-15 A picture of assembled microfluidic system
CHAPTER 8 EXPERIMENTAL TEST AND RESULTS

8.1 Tests on the AC MHD Micropump

There are two key factors that need to be studied to operate AC MHD micropump. One is driving current and its frequency that applied to electrodes; another is the characteristic of the electromagnet. As reported by our research effort, bubble generation can stop the function of the micropump. To eliminate bubble generation, threshold value of current and its frequency was measured that does not cause bubble generation.

The characteristic of the electromagnet is very important in AC MHD micropump because the phase shift of magnet field directly affects the performance of the micropump. The magnetic flux density was measured using a LakeShore 420 gaussmeter (LakeShore Corp., Westerville, OH) is plotted in Figure 8.1. The magnetic flux density was significantly decreased beyond 1kHz.

![Figure 8-1 Magnetic flux density at frequency span 10 to 1kHz](image-url)
This characteristic of the electromagnet can be explained as a resonance RLC circuit. The electromagnet can be considered as a serial RLC circuit, and its resonance frequency can be expressed as following equation.

\[ f_0 = \frac{1}{2\pi\sqrt{L \cdot C}} \]  

(8-1)

When operation frequency become a resonance frequency, the impedance of the serial RLC circuit is minimized, thus the current in the circuit can have maximum value. Since the magnetic field density is proportional to applied current, its maximum value can be obtained at resonance frequency. The calculated resonance frequency of the electromagnet is 32.24 Hz that is similar value from the measured one.

To drive continuous flowing of the solution, this magnet field phase should be synchronized to the current that supplied to electrodes. The phase shift circuit was successfully designed and made for the micropump system to solve phase shifting. Circuit design and phase shifting angle equation are shown in Figure 8-2 and Equation (8-2). The phase shift angle varies from -12° to 168°, and this variation is enough to cover the phase shift of magnet field at the input frequency, which is span to few kHz.

\[ \theta = 2 \cdot \tan^{-1}(2\pi \cdot f \cdot Ci \cdot Ri) \]  

(8.1)

The operating frequency was set to be 500Hz because bubble-free operation can be allowed at higher frequency with higher currents. Figure 8-3 shows a measurement setup for AC MHD micropump system. The electrodes and the electromagnet are driven by a single function generator. The phase shifter was connected to power amplifier to synchronize the input signal of electromagnet.
Figure 8-2 Phase shift circuit drawing
Figure 8-3  Measurement setup for AC MHD micropump

To measure the flow rate, the microscope was positioned above the micropump. A CCD camera, which is connected to video capture software, was attached to the microscope. The 1M NaCl solution was mixed with 3.9 μm polystyrene beads, and this enabled us to measure flow speeds by tracking the moving distance of the beads in a given time.

Figure 8-4 shows the movements of two beads between two frames 50 seconds apart. The calculated flow rate was 1.62ul/min.
Figure 8-4 Video capture of 3.9 μm polystyrene beads flowing in micro channel

8.2 Tests on the Electrochemical Actuator

To verify the bubble force as a source of the pumping, simple experiment was done. The one side of the chamber of the pump was blocked, and the function generator was connected to two electrodes to supply driving potential. Figure 8-5 is a sequential picture of the pumping.

Figure 8-5 Sequential picture of the pumping by the bubble formation
The circles in the sequential pictures indicate the boundary between the gas, which was generated by electrolysis, and solution. 1M NaCl was served as a test solution, and the frequency of the input signal was 5 Hz of sinusoid with electrode potential of ±15Volts. Calculated flow rate was 12 μl/min. This experiment proved that the bubble force can be a very powerful and efficient source of the pumping.

Efforts were also made to demonstrate the feasibility of fast reaction and reverse reaction that actuates ECM micropump. This driving principle can be achieved by controlling the duration of operating potential. Highest potential was chosen for -1.5 V because largest bubble was achieved in the micro chamber at given potential. The bubble was expanded at positive potential 0.5V for 4 seconds, and was shrunk at negative potential -1.5V for 16 seconds. Figure 8-6 shows the bubble shape when fully expanded and shrunk during 20 seconds of a cycle.

![Figure 8-6 Bubble shape of expansion and shrinking](image)

(A) Expansion phase  (B) Shrink phase

8.3 Tests on ECM Microfluidic System

In the microfluidic system, function test for the valve actuator was performed before test of entire microfluidic system. Test set up is shown in Figure 8-7.
The end of outlet tube is connected to PMMA cover hole, which serve as a pathway of outlet flow in the pump chamber when the pump is start to push out the water. The water was filled in both of two chambers through fill hole in the PDMS structure. When the active valve starts to expand PDMS membrane by electrochemical actuation, the valve blocks flow of the water. The purposes of this test are; (1) the performance of the electrochemical active valve, (2) valve function test in microfluidic system.

For the performance test of the valve, valve electrodes were connected to the Potentiostat (PS-205/API, ELCHEMA, Potsdam, NY) that can control electric potential level through three electrodes system. To control valve operation, the voltage pulse signal come from the function generator is connected to the Potentiostat. The membrane deflection was measured using micrometer that attached with microscope by focusing different two different heights to verify operation of the valve. Figure 8-8 shows a schematic of deflection measurement, pictures of deflected membrane. The applied potential was -1.5V as same as a prototype electrochemical actuator, and was continuously supplied through electrodes until observation of notable deflection. From the measurement, deflection height ($\delta$) was achieved up to 300 um. After it reaches at height of 300 um, no significant deflecting move was observed. The dashed line in
picture is an edge of the membrane, and the white light dot inside the line is near top of the deflected membrane due to electrochemical actuation.

To check maximum deflection with 100 um thickness of PDMS membrane, lower negative potential up to -7.0 V was applied to the electrochemical actuation valve for 10 seconds. Very large displacement was observed, and the measured deflection height was approximately 3 mm.

![Deflection measurement](A) Deflection measurement                   ![Deflected membrane with -1.5V](B) Deflected membrane with -1.5V

![PDMS membrane before deflection](C) PDMS membrane before deflection   ![Deflected PDMS membrane with -7.0V](D) Deflected PDMS membrane with -7.0V

Figure 8-8 Deflection measurement of PDMS membrane

Operation at lower negative potential made quick pressure accumulation in valve chamber because the electrochemical reaction occurred not only in working electrode but also in reference electrode. Figure 8-9 is a picture the valve when it was operated in
lower voltage negative potential and it show that gas bubbles are generating from two electrodes.

Since valve seat height is 100 um, the operation with this deflection is sufficient to stop the flow of the solution that pumped from pump actuator chamber. The calculated built pressure inside the valve chamber was calculated using equation (5-18), it results 790KPa with 300 um deflection and 7.9MPa with 3mm deflection. The PDMS membrane was lasted with 3mm deflection, and it was not break at relatively high pressure.

Figure 8-9 A picture of valve actuation at lower negative potential

The reversal reaction, which makes PDMS membrane return back to original position by gas shrinkage, took relatively longer time than expansion phase. The pictures in Figure 8-10 are sequential images of PDMS membrane when it starts to shrink from maximized deflection position, and each frame was taken 7.5 seconds apart. The reversal reaction was continued until the membrane return back to the original position. More than 20 minutes later, the membrane deflection was reduced, but it did not returned back completely. As it was discussed in chapter 5, residual micro bubbles, generated oxygen bubbles and electrodes location may result in slow reversal reaction. For the reversal reaction, the generated gas bubble should contact with electrodes those are located in bottom of the chamber. After oxidation reaction for few minutes, the water is fulfilled
between electrodes and gas bubbles, and this makes hard to contact hydrogen gas on the electrodes.

![Sequential images of the PDMS membrane at reversal mode](image)

The valve function test was done with integrated micropump. First, a voltage of -1.5V was supplied to the electrochemical actuator in the pump chamber that results gas bubble push out the solution through micro channel and connected tube. The valve was activated in middle of the pump actuation with -2.5V of potential. Positive potential of 0.5V applied to the pump actuator for reversal reaction while valve stops the flow of the solution. Then negative potential was applied to the pump actuator to push out the solution again, and positive potential of 0.5V was applied to the valve actuator to reopen the outlet hole by reversal electrochemical reaction. Figure 8-11 shows pictures of the valve function test.

Dyed DI water was filled on the top of the DI water to help the observation. Since Dyed DI water was filled through the connected tube, it was not completely filled to the pump chamber, and that made the gap between two boundaries.
As it can be seen from the photograph, as the valve was opened, the water level rises as marked by the two arrows. The position of the boundary between two dyed and not dyed DI water rose up after the valve was opened and more water pumped out. After valve was reopened, the pump was started again to push out the solution from the
chamber. The interval between close and reopen mode was 6 minutes because of sloe reversal reaction in the valve chamber.

The test results of the microfluidic system show that it could be used in various microfluidic applications. The valve system can be integrated to PCR or LDR system for mixing of analysis solutions. It also can be used micropump by PDMS membrane actuation. The compact syringe pump with very large displacement can be made by attaching simple structure to micro valve. The performance of this micropump such as, pumping rate and pressure can be precisely controlled by adjusting electrochemical reaction rate.
CHAPTER 9  SUMMARY AND FUTURE WORK

9.1 Summary

9.1.1 MHD Micropump

- AC-type MHD micropump was designed, analyzed, and tested as a possible solution to the bubble formation problem encountered in DC-type MHD micropump. A complete and detailed model of the AC MHD micropump with the electrochemistry sub model was developed and analyzed using simulation tool.

- UV-LIGA process was successfully implemented for inexpensive microfabrication of AC MHD micropump.

- The test result shows that the bubble formations were significantly reduced, and flow rate was observed as fast as 1.62 μl/min.

9.1.2 Electrochemical Actuator

- Mathematical model of electrochemical actuator was developed for predicting pumping pressure using the ideal gas law.

- Prototype of electrochemical actuator was fabricated and tested to verify the bubble force as a source of actuator. The observed flow rate was 12 μl/min when the potential of ±15V was continuously supplied to the electrodes, which are located in one side blocked chamber. Test of electrolysis and reversal reaction was achieved as fast as 20 seconds at 0.05Hz, ±2V, -0.5V DC offset.

- Optimized SU-8 UV-LIGA processes for various microfluidic components such as PDMS structure fabrication with multilayer of SU-8, SU-8 structure fabrication on PMMA substrate with lower temperature process, were successfully established.
• The performance test was done for the microfluidic system. The valve membrane was deflected 300 um with -1.5 V potential, and it reached maximum deflection 3mm at -7.0V of potential.

• The PDMS membrane with a thickness of only 100μm was found to be able to stand a pressure up to 7.9MPa and large deflection.

• The sequential operation of the electrochemical valve and pump was successfully demonstrated with electrochemical reaction controlling.

9.2 Future Work

• Particle Image Velocimetry (PIV) Test for AC MHD Micropump

The flow of MHD micropump can be visualized by mixing the micro particles to a carrier solution. The high resolution two-dimensional velocity fields can be obtained through this experiment, and it can lead to the better understanding of microfluidics in MHD micropump chamber. Due to the limitation of equipment available in our laboratory, the prototype micropump had to be fabricated in such a way that observation of flow can only be through backside. The fabricated sample has two outlet holes on the glass substrate. This design makes it possible to conduct PIV test by covering the top surface with a plastic sheet.

• Performance Test of AC MHD Micropump with Non Aqueous Solvents

The AC MHD pump was tested only with one conductive solution (saline water). This caused the electrolysis problem in the MHD pump. If MHD pump is operated with non aqueous solution, we can use DC operation. This may help to remove complex AC driving circuit by replacing an electromagnet to permanent magnet. Tetrahydrofuran was
used to check feasibility of operation in MHD micropump, but it dissolved the PMMA cover sheet. The SU-8 structure is found to be resistant to this solution, but PMMA was not. It needs other covering material that does not affect from dissolving issue such as SU-8. Further effort that is fabricating a thin SU-8 layer on the thin glass substrate has been investigating. Figure 9-1 shows a schematic of the glass cover fabrication with SU-8. A thin layer of SU-8 is spin coated on the glass substrate. After hard baking of SU-8, the glass substrate is put on the surface of MHD micropump with small weight in the convection oven at 65ºC for 1 hour. The soften SU-8 can be bonded and then exposure and hard bake process is followed.

Figure 9-1 A schematic of assembling glass cover on MHD micropump

- **Electrochemical Actuator Test with Microfluidic Application**

  Eventually the micropump need to be connected to other microfluidic system such as micro mixer or sensing unit for various applications. Further tests need to be conducted to check feasibility of using these micropump with the ECM active valves for handling various solutions.
REFERENCES


APPENDIX A : SU-8 PHOTORESIST PROCESS

A1. Spin Coating process

Figure A-1 Spin speed vs. thickness curves for SU-8 50

A2. Soft Bake Process

Table A-1 Recommended soft bake process [MicroChem Inc.]

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### A3. Exposure Process

#### Table A-2 Exposure dose versus thickness [CAMD]

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<td>770</td>
<td>2490</td>
</tr>
<tr>
<td>220</td>
<td>840</td>
<td>500</td>
<td>1680</td>
<td>800</td>
<td>2580</td>
</tr>
</tbody>
</table>
### A4. Post-exposure Bake

Table A-3  Recommended PEB Process [MicroChem]

<table>
<thead>
<tr>
<th>Product</th>
<th>Thickness (µm)</th>
<th>Soft Bake Time (m: minute)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Step 1: 65 °C</td>
<td>Step 2: 95 °C</td>
</tr>
<tr>
<td>SU-8 5</td>
<td>5</td>
<td>1 m</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>1 m</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>2 m</td>
</tr>
<tr>
<td>SU-8 10</td>
<td>10</td>
<td>1 m</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>2 m</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>3 m</td>
</tr>
<tr>
<td>SU-8 25</td>
<td>15</td>
<td>1 m</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>3 m</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>4 m</td>
</tr>
<tr>
<td>SU-8 50</td>
<td>40</td>
<td>2 m</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>5 m</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>10 m</td>
</tr>
</tbody>
</table>
APPENDIX B : S1813 PROCESS

B1. Spin-coating
i  Clean the substrate with acetone, IPA and DI water (long rinse); dehydration bake @ 120° C for 1/2 hour (convection oven)
ii Spin-coat for 30 sec (2.5 μm)
iii Let the wafer sit for 10 min to allow the resist to relax
iv Bake cycle - put into hot plate @ 95° C for 1.5 min.
v Let the sample sit for about 1/2 hour

B2. Exposure
i Expose 100mJ/cm² using Quintel in vacuum mode
ii See the data sheet for the relationship between dose and thickness

Let relax after exposure for at least 30 min

B3. Development by Microposit 354 Developer or PD523 AD
i Use 2 baths of Microposit 354 developer
ii 2 min in first bath (used developer can be used). Agitate mildly by moving the developer tray
iii 2 min in second bath (fresh developer). While in this bath stop development soon after red clouds of resist stop coming off the wafer.
iv Rinse in DI water
Dong Eun Lee was born on April 11th, 1970, in Daegu, South Korea. He received the degree of Bachelor of Science in Mechanical Engineering in February of 1994 and the degree of Master of Science in Mechanical Engineering in February of 1996 from Yeungnam University in Kyoungsan, South Korea. In 1996 he joined Samsung Electronics Storage System Division in Kumi, South Korea, as a mechanical engineer. In the Fall of 2001, he entered Louisiana State University (LSU) Baton Rouge, Louisiana and joined graduate program in mechanical engineering as a member of Microsystems Engineering Team. He expects to receive the degree of Doctor of Philosophy in mechanical engineering in May 2007.