Acoustic Streaming Pump for Microfluidic Applications

by

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A thesis submitted in conformity with the requirements for the degree of Master of Applied Science

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A prototype acoustic streaming pump for microfluidic applications was developed. A novel integration scheme was devised based on the acoustic reflector concept. Numerical simulations were conducted to predict the flow patterns around the transducer. Ultrasound transducers using P(VDF-TrFE) as the piezoelectric element were fabricated using lithography-based microfabrication technology. Silicon channels were fabricated using anisotropic etching. A heat-press bonding technique was adopted to bond the transducers with the silicon chips using CYTOP fluoropolymer as the adhesive. The piezoelectric transducers were characterized to have a resonance frequency of 82 MHz. Micro-PIV experiments were performed in the near and far-fields of the ultrasonic transducer/pump. The near field experiments showed complex flow patterns that could enhance mixing. Estimates of the pumping pressure were obtained using transient flow velocities in the far-field. Conservative estimates indicate the total back pressure the micropump can pump against is 39 Pa. Future research directions were suggested.
Acknowledgements

I would like to first thank my supervisors Axel Guenther and Anthony Sinclair for their support and guidance during the past two and a half years. They have taught me that perseverance is the key to success in many aspects of life. I am grateful to the funding provided by the Natural Sciences and Engineering Research Council. Next I would like to thank Michael Huang and Dr. Vahid Safavi for their initial work on this project. I am especially indebted to Dr. Safavi for our technical discussions and his patience in answering my long and numerous questions. The training provided by Dr. Henry Li, Yimin Zhou and Dr. Edward Xu at the ECTI microfabrication facilities are also deeply acknowledged. Next I would like to acknowledge my colleagues at the Guenther research lab for their help and friendship during the past years. Specifically I would like to thank Lian Leng for her training sessions on soft-lithography fabrication; Sanjesh Yasotharan for his invaluable help in many different areas of my experimental work and Milad Abolhasani for our many discussions on science and life in general. I would also like to thank Oren Kraus for his feedbacks on my thesis presentation and Amir Saffari for his company in the lab for the past two years. Lastly, I would like to thank my family for their patience and continual support during my graduate project.
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\( \rho \) ........................................................... density
\( \rho_0 \) ....................................................... density at rest
\( x_i, x_j, x_k \) ........................................... directions in Einstein notation
\( u_i, u_j, u_k \) ........................................ velocity components in Einstein notation
\( p \) ........................................................... pressure
\( f_i, f_j, f_k \) ........................................ force components in Einstein notation
\( u \) ........................................................... x component of velocity
\( v \) ........................................................... speed of sound for a material, flow speed
\( \mu \) ........................................................ first viscosity coefficient
\( \lambda \) ..................................................... second viscosity coefficient, wavelength
\( t \) ........................................................ time, thickness
\( f \) ........................................................ frequency
\( \omega \) .................................................... angular frequency
\( A \) ....................................................... wave amplitude, area
\( \alpha \) .................................................... absorption coefficient, angle from horizontal plane
\( F \) ........................................................ force per volume
\( I \) ........................................................ acoustic intensity
\( I_o \) ....................................................... initial acoustic intensity amplitude
\( c \) ........................................................ speed of sound in working fluid
\( R_I \) ..................................................... intensity reflection coefficient
\( Z_o \) ..................................................... acoustic impedance
\( \theta_i \) ................................................... incident angle
\( \theta_t \) ................................................... angle of transmission
\( k_t \) ..................................................... thickness coupling factor
\( m \) ....................................................... maximum size of mesh element
\( Re_h \) ...................................................... Reynolds number based on hydraulic diameter
\( C_h \) ...................................................... constant relating friction factor and \( Re_h \)
\( D_h \) ...................................................... hydraulic diameter
\( C_g \) ...................................................... constant relating average and maximum velocity
\( C_l \) ...................................................... lumped constant relating pressure gradient to maximum velocity
\( Q \) ....................................................... volumetric flow rate
1 Introduction

In the last few decades, ability to manipulate fluids through microscale flow networks has fueled advances in analytical and synthetic chemistry, cell molecular and biology, materials science, soft matter physics and in clinical medicine [1-3]. Lab-on-a-chip (LOC) devices have been successfully developed to operate with a variety of liquids and in performing numerous analytical tasks. One of the main attractions of microfluidic platforms is the opportunity to conduct experiments that are difficult to implement in macro environments due to the differential scaling of physical properties. As the channel dimensions become smaller, the surface area to volume ratio increases, and this characteristic can be exploited to expedite chemical reactions [4]. The move towards smaller reagent volumes can also reduce the cost of experiments.

The pumping device used to control the flow of fluids within the microchannels is an important component to microfluidic systems. Two major types of pumping devices are commonly used. The first type consists of external pumps such as syringe pumps and the second type consists of micropumps that are integrated within the microfluidic device. Integrated micropumps do not require external tubing and therefore can further reduce the quantity of fluids used in the application. They can also provide flows for complex networks by integrating an array of these pumps within the microfluidic system. For these reasons, the development and characterization of a prototype micropump will be the focus of this research project.

We chose to adopt acoustic streaming for the development of our micropump. Acoustic streaming is the physical phenomenon in which the momentum from a propagating mechanical wave is transferred into the fluid medium causing the fluid to move in the direction of the wave propagation [5]. Compared to other continuous micropumps, acoustic streaming pumps do not induce electrochemical reactions in the fluid and are therefore compatible with a wide range of biological applications. The planar geometry of the transducers also allows the pumping system to be scalable and modular.

1.1 Project Objectives
The goal of this project is to design, demonstrate and characterize a scalable pumping strategy for microfluidic applications using the principle of acoustic streaming. We will first review the literature on the physical background and current state of development of acoustic streaming pumps, and outline improvements that we can contribute to this topic. The next step is to devise our own micropump design and integration scheme, and evaluate the performance of our design using finite element simulation packages. The results of the numerical simulations could then be used to improve the initial designs. The third and the most time-consuming step is to fabricate a prototype of the acoustic streaming micropump. This would involve the fabrication of the transducers and the microchannel networks as well as the development of the integration protocol for these two components. The last step of the project is to conduct experiments using the fabricated devices from the previous step. Electrical impedance and C-scans will be conducted on the piezoelectric transducers to characterize their resonance frequency and active areas. Flow visualizations experiments will be conducted with the integrated micropumps to test the flow rates provided by the devices.

1.2 Outline for Remaining Chapters

Chapter 2 will give a comparison of the different micropumps reported in the literature. We will compare their relative advantages and disadvantages and explain why we decided to adopt the acoustic streaming mechanism for powering our micropump. This will be followed by a discussion of the theory of acoustic streaming. Finally, a list of the detailed design goals will also be given in this chapter, based on the physics and previous research experience described in earlier Sections of Chapter 2.

Chapter 3 will present the design scheme that we devised for meeting the design goals listed in the previous chapter. It will contain results from numerical simulations and a description of how the overall trends observed in the simulations were used to improve the design of our micropump. 3D simulations of the operation of our micropumps, plus analysis of the results, will be shown towards the end of the chapter.
Chapter 4 will present the device micro-fabrication sequence and the decisions that were made in the development of the fabrication steps. Some failed processes will also be discussed. This chapter is divided into three main sections. The first one will discuss the fabrication of the piezoelectric transducers. The second one will discuss the fabrication of the silicon microchannel networks while the last section will describe the bonding technique used to combine the two components into a working microfluidic system.

Chapter 5 will give describe the performance of the fabricated devices. Electrical impedance and C-scan results will be given. Micro-scale particle image velocimetry results will show the flow patterns generated by the micropumps, and an analysis to estimate the total pumping pressure and power will be presented.

The final chapter will provide a summary of our work and list developments that can be pursued in the future.
2 Background and Motivation

An important component to microfluidic systems is the pumping device used to control the flow of fluids inside the channels. Commercially available flow control devices include syringe pumps and geared rotary pumps. Although these devices are programmable and widely adopted, they require the use of external tubing which reduces the robustness of the system and negate a major advantage of microfluidics—reduction of the quantity of fluids used for experiments. It is also difficult to control complex flow networks using external pumps. For these reasons, it is desirable to utilize micropumps that are integrated into the microfluidic system.

The goal of this project is to develop a prototype pumping system for microfluidic applications that can provide good temporal and spatial control of the fluid flow. The pump needs to be compatible with biological fluids (such as blood cells) and be able to provide constant flow rates for an extended period of time. In addition, the pumping system should ideally be modular so that different channel networks can be integrated to the same pumping component. Such modularity would allow for rapid design changes to the channel networks for adaptation to different applications.

Before we decided on the actuation mechanism for our micropump, we consulted the literature to review the strengths and weaknesses of the current micropump designs. After the literature review process, we chose to utilize the acoustic streaming effect for our micropump. The reasoning for this decision will be made transparent after a comparison of the different types of micropumps in the section 2.1. It will be followed by an explanation of the acoustic streaming mechanism in section 2.2. Section 2.3 will discuss the inadequacies of the current acoustic streaming micropumps and what we plan to improve for this research project.

2.1 Comparison of Micropumps

There has been ongoing research on the field of micropumps for the past three decades. Many review articles have been published categorizing the pumps according to their working principles and comparing their pumping performance [6-9]. Micropumps can generally be divided into two
major categories. The first category consists of pumps that use a mechanical device to create a moving boundary to displace a liquid volume. The second category consists of non-displacement pumps that continuously add energy into the fluid to increase its momentum or pressure to induce fluid flow.

2.1.1 Displacement Pumps

Displacement pumps usually employ the deflection of a membrane to create a change of volume to create fluid flow. Many design schemes have been developed to move the fluid in the desired direction. These different design schemes are summarized below.

One of the most common types of displacement pumps is the check-valve pump. Check valves are passive components with high reverse-to-forward flow resistance ratio. When a forward pressure is applied, the check-valve would open and allow the fluid to pass through. Conversely, when pressure is applied in the reverse direction, the valve would close and prevent the fluid from leaking through. A schematic diagram of this working principle is presented in figure 2.1.
Many different actuation principles have been used to create the volume displacement in the check-valve devices. The most common actuation mechanisms include thermopneumatic [10, 11], electromagnetic [12], electrostatic [13] and piezoelectric [14].

The second common type of displacement pumps is the valveless rectification pumps. The constructions of the valveless displacement pumps are similar to the check-valve pumps. The major difference is that instead of the use of passive check-valves, non-moving nozzle/diffuser structures are used to direct fluid flow. The benefit of this design is that it avoids the clogging, wear and fatigue problems that are associated with moving valves. The elimination of moving parts also allows further miniaturization of the micropumps. Examples of such valveless displacement pumps are presented in [15] and [16].
A third variation of the volume displacement pumps is the peristaltic pump. These pumps leverage on peristaltic action created by sequencing opening and closing of displacement chambers. A schematic representation of peristaltic actuation using three chambers is shown in figure 2.2.

Since a peristaltic pump does not require a large actuation pressure to open or close the flow valves, the most important optimization factor is to increase the deformation of the flow chamber. Early pumps were made with piezoelectric actuators and pump chamber etched in silicon [17]. More recently with the advance of rapid prototyping soft lithography technology in silicone rubber (PDMS), the peristaltic pumping principle has been successfully adopted in soft elastomer microfluidic systems [18]. Since the Young’s modulus of PDMS is more than 5 orders of
magnitude lower than that of silicon (around 700 kPa compared to 180 GPa) [19, 20], the deformation of the membrane is much larger for the same applied actuation pressure. Consequently, the valve chambers can be drastically reduced in size. This allows a dense integration of flow channels and actuation chambers.

2.1.2 Disadvantages of Displacement Pumps

Both the check-valve and the nozzle/diffuser displacement pumps are devices that are integrated with the channel geometry. In other words, the channels must be designed to accommodate these devices at the beginning of the design process. This severely limits the usability of these systems in a research environment where channel designs are modified frequently. The need for a displacement chamber also limits the ability for miniaturization.

In comparison, the peristaltic pumps described in [18] are more promising since they can be fabricated on a layer on top of the flow channels. However, the reliance on the large deflections of PDMS severely limits its usefulness in chemical applications because many reagents would attack the soft elastomer channels.

2.1.3 Continuous Pumps

The second category of micropumps directly adds energy into a microfluidic channel to create fluid flow. Although the flow rates and maximum back pressures reported by these pumps are often lower than their displacement counterparts, they have the advantage of providing very constant fluid flows since they are not affected by the transient effects of cycling displacement chambers. In addition, the low flow rates can also be an advantage if the application requires a slow but well-defined fluid movement in the system.

Electro-osmotic flows make use of electrokinetic effect. In general, when an electrolytic liquid is in contact with a solid wall, the surface of the solid substrate develops a negative charge [21]. Consequently, the positively charged ions of the electrolytic liquid would be attracted to the liquid-solid interface. If electrodes with a potential difference are placed at the two ends of the
channel, the positively charged liquid at the walls would be attracted to the anode [22]. Since the liquid particles around the circumference of the channel all move at about the same velocity, the result is a “plug flow” meaning the velocity profile across the cross-section of the channel is constant. One major disadvantage of electro-osmotic flow is that the flow rate is very sensitive to the pH or ionicity of the solution. In addition, the electrodes immersed in the fluids can produce unwanted electrochemical reactions in the channels. This is very undesirable in biological applications. Lastly, many biological molecules would attach to the walls of the channel under electrokinetic situations and are therefore precluded from the use of electro-osmotic pumps.

A second type of continuous micropump is the electrohydrodynamic (EHD) pump. There are two major types of electrohydrodynamic (EHD) pumps—the EHD induction pump and the EHD injection pump. The EHD induction pump is based on the induced charge at the material interface. A traveling wave of electric field pulls the charged liquid particles in the direction of the propagating wave. In contrast, EHD injection pumps injects ions into the fluid by a electrochemical reaction [7]. Richter et al. [23] have demonstrated examples of both the EHD induction and injection pumps in 1990. The major disadvantage of EHD pumps is that they rely on induced charges in the liquid. This is often undesirable in biological applications where the cells are handled at in vitro environments.

A third category of continuous micropump is the ultrasound acoustic streaming pump. There are two different acoustic phenomena that can be used for providing fluid flow. The first of which is called “quartz-wind” acoustic streaming, named after the movement of air observed in the vicinity of quartz crystal transducers [24]. Quartz wind pumps use longitudinal propagating sound waves to transfer momentum into the working fluid. When an ultrasound wave is sent in the axial direction of a liquid filled channel, its sound energy would be absorbed causing movement of the fluid in the direction of wave propagation. The second type of acoustic streaming pumps uses surface propagating waves (such as Rayleigh waves or flexural plate waves) to move the fluid near the vicinity of the surface.

In comparison to the previously described micropumps, acoustic streaming devices do not initiate electrochemical reactions in the channels and are therefore suitable for biological applications.
Also, since the activation areas are composed solely of planar piezoelectric transducers, modular systems can be developed that would allow for frequent design changes of the channel geometry without affecting the transducer design. Lastly, acoustic streaming pumps fabricated in silicon-based microsystems can be used to conduct many chemical experiments that are incompatible with PDMS-based microfluidic systems.

Because of these advantages, we decided to design our micropump using the acoustic streaming principle. From the two possible acoustic streaming actuation schemes, we chose to use “quartz wind” streaming due to the following reasons. The first reason is novelty. Most of the research to date have focused on traveling wave devices and very few works have concentrated on quartz wind streaming. To our knowledge, the only quartz wind micropump was reported by Rife et al. [25] and it only provided a very small pump pressure (0.13 Pa). The second reason is that traveling wave streaming is only effective at moving a very thin layer of fluid above the transducer. In comparison, quartz wind streaming is capable of transferring momentum to the entire volume of a microfluidic channel. Consequently, quartz wind is a more powerful transport mechanism for typical microfluidic channel dimensions that are of the order of 100 μm [22, 26].

2.2 Principles of Acoustic Streaming

Before we begin our discussion of acoustic streaming, let us introduce the governing equations of fluid flow. For brevity, we will not discuss the derivation of these fundamental equations. The reader can refer to many fluid mechanics textbooks, such as [27] for more details.

2.2.1 Governing Equations for Fluid Mechanics

The first important equation of fluid mechanics is the mass conservation, or continuity equation. For a general compressible fluid, it is written in the form:

\[
\frac{\partial \rho}{\partial t} + \frac{\partial}{\partial x_k} (\rho u_k) = 0
\]  

(2.1)
Where $\rho$ is the density and $u$ is the velocity of the fluid. Notice here we have used the Einstein notation where the subscripts $i, j, \text{ and } k$ represent the $x, y, \text{ and } z$ components of a vector. In addition, repeated subscripts (seen on the second term of the LHS) represent summation over all three components. Physically, the first term represents the time-transient change in density while the second term represents the convective change of density.

Often it is sufficient to assume negligible compressibility when the fluid is in a liquid state. For such instances, the continuity equation can be simplified to:

$$\frac{\partial \rho}{\partial t} + \rho \frac{\partial u_k}{\partial x_k} = 0 \quad (2.2)$$

The second important governing equation in fluid mechanics is the Navier-Stokes equation. This equation combines the conservation of momentum with the constitutive equations for a Newtonian fluid to provide closure between the systems of governing equations and number of fluid properties in fluid dynamics.

For a compressible Newtonian fluid, the Navier-Stokes equation in Einstein notation is given as:

$$\rho \frac{\partial u_j}{\partial t} + \rho u_k \frac{\partial u_j}{\partial x_k} = -\frac{\partial p}{\partial x_j} + \frac{\partial}{\partial x_j} \left( \lambda \frac{\partial u_k}{\partial x_k} \right) + \frac{\partial}{\partial x_j} \left[ \mu \left( \frac{\partial u_k}{\partial x_k} + \frac{\partial u_j}{\partial x_i} \right) \right] + \rho f_j \quad (2.3)$$

Here $p$ is the pressure of the fluid and $\mu$ and $\lambda$ are the first and second viscosity coefficients. This result can be greatly simplified for incompressible flows. For incompressible flows, the divergence of flow field is zero, hence the term involving the second viscosity coefficient is identically zero. After more simplification, the Navier-Stokes equation for an incompressible fluid is:

$$\rho \frac{\partial u_j}{\partial t} + \rho u_k \frac{\partial u_j}{\partial x_k} = -\frac{\partial p}{\partial x_j} + \mu \frac{\partial^2 u_j}{\partial x_i \partial x_i} + \rho f_j \quad (2.4)$$
Physically, the first and second terms on the left-hand-side represent the temporal and convective acceleration of the fluid. On the right-hand-side, the first term is the pressure gradient, the second term the viscous resistance to flow and the last term a body force, such as gravity, acting on the fluid.

### 2.2.2 Reynolds Stress and Quartz Wind Streaming

Physically, acoustic streaming occurs due to the transfer of momentum from the sound wave into the fluid. We can illustrate this concept mathematically through an analysis using the concept of Reynolds stresses [24].

Reynolds stresses usually refer to turbulent fluctuations in fluid momentum that cause a stress tensor to develop in the fluid. The resultant stress would cause the fluid to move in a preferred direction. This concept can be extended to wave-fluid flows by treating the resultant time-averaged acoustic streaming fluid velocity as the average fluid velocity and replacing the turbulent fluctuations with ultrasound wave oscillations.

Let us first clarify the mathematical notations and the ensemble rules of averaging that will be used before conducting the analysis. Since the tensor notation is employed, the first order sound perturbation and second order flow streaming velocities are represented by superscripts $u^{ac}$ and $u^{dc}$ respectively to avoid interference with the summation subscript $i$ and $j$. The bar at the top of following entities represents temporal averages over a wave period. The following averaging rules apply:
For our analysis, we will assume the flow is incompressible to significantly reduce complexity. Now if we let $u$ to be $(u^{dc} + u^{ac})$ and $p$ to be $(p^{dc} + p^{ac})$, the governing equations become:

\[
\frac{\partial (u_i^{dc} + u_i^{ac})}{\partial x_i} = 0
\]

\[
\rho \frac{\partial (u_i^{dc} + u_i^{ac})}{\partial t} + \rho (u_j^{dc} + u_j^{ac}) \frac{\partial (u_i^{dc} + u_i^{ac})}{\partial x_j} = - \frac{\partial (p^{dc} + p^{ac})}{\partial x_i} + \mu \frac{\partial^2 (u_i^{dc} + u_i^{ac})}{\partial x_j \partial x_j}
\]  

(2.6)

Notice that we have neglected the external body force term in the Navier-Stokes equation since it is not needed in the derivation of the acoustic streaming equations. The second term on the LHS can be simplified by chain differentiation and continuity. The corresponding Navier-Stokes equation is:

\[
\rho \frac{\partial (u_i^{dc} + u_i^{ac})}{\partial t} + \rho \frac{\partial (u_i^{dc} + u_i^{ac})(u_j^{dc} + u_j^{ac})}{\partial x_j} = - \frac{\partial (p^{dc} + p^{ac})}{\partial x_i} + \mu \frac{\partial^2 (u_i^{dc} + u_i^{ac})}{\partial x_j \partial x_j}
\]  

(2.7)
Now we can average the various terms to generate the Reynolds averaged Navier-Stokes equation. The temporal average of the first term of the momentum equation \( \rho \frac{\partial (u_i^{dc} + u_i^{ac})}{\partial t} \) can be written as \( \rho \frac{\partial u_i^{dc}}{\partial t} \) since the density here is constant and the last ensemble average rule states that averaging only occurs inside of the differential operator. Using the second ensemble averaging rule \( \rho \frac{\partial (u_i^{dc} + u_i^{ac})}{\partial t} \) becomes \( \rho \frac{\partial u_i^{dc}}{\partial t} \) which finally can be represented by \( \rho \frac{\partial u_i^{dc}}{\partial t} \) as the average value of a sinusoidal oscillation is identically zero.

Similar logic can be applied to the first and second terms of the right hand side to obtain:

\[
\rho \frac{\partial (u_i^{dc})}{\partial t} + \rho \frac{\partial (u_i^{dc} + u_i^{ac})(u_j^{dc} + u_j^{ac})}{\partial x_j} = -\frac{\partial (p^{dc})}{\partial x_i} + \mu \frac{\partial^2 (u_i^{dc})}{\partial x_j \partial x_j} \tag{2.8}
\]

To simply the second term on the left, the product \((u_i^{dc} + u_i^{ac})(u_j^{dc} + u_j^{ac})\) must be first multiplied out:

\[
(u_i^{dc} + u_i^{ac})(u_j^{dc} + u_j^{ac}) = u_i^{dc}u_j^{dc} + u_i^{dc}u_j^{ac} + u_i^{ac}u_j^{dc} + u_i^{ac}u_j^{ac}
\]

Now taking the average of the RHS, we have:

\[
\overline{u_i^{dc}u_j^{dc}} + \overline{u_i^{dc}u_j^{ac}} + \overline{u_i^{ac}u_j^{dc}} + \overline{u_i^{ac}u_j^{ac}} = \overline{u_i^{dc}u_j^{dc}} + \overline{u_i^{dc}u_j^{ac}} + \overline{u_i^{ac}u_j^{dc}} + \overline{u_i^{ac}u_j^{ac}}
\]

Of the four terms on the right-hand-side of the above equation, only the first and last ones survive. Consequently, the Reynolds averaged governing equations take the form:

\[
\rho \left[ \frac{\partial (u_i^{dc})}{\partial t} + \frac{\partial (u_i^{dc}u_j^{dc})}{\partial x_j} + \frac{\partial (u_i^{ac}u_j^{ac})}{\partial x_j} \right] = -\frac{\partial (p^{dc})}{\partial x_i} + \mu \frac{\partial^2 (u_i^{dc})}{\partial x_j \partial x_j} \tag{2.9}
\]
Finally, by assuming a steady state solution and further simplifying the equation, one arrives at the final form of the acoustic streaming governing equation:

$$
\rho u^{jc}_j \frac{\partial (u^{jc}_i)}{\partial x_j} = - \frac{\partial (p^{jc})}{\partial x_i} + \mu \frac{\partial^2 (u^{jc}_i)}{\partial x_j \partial x_j} - \rho \frac{\partial (u^{ac}_i u^{ac}_j)}{\partial x_j} \tag{2.10}
$$

Physically $\rho \frac{\partial (u^{ac}_i u^{ac}_j)}{\partial x_j}$ can be considered as a body force acting on the fluid. $\rho u^{ac}_i u^{ac}_j$ is viewed as the $x_j$ component of sound wave momentum, $\rho u^{ac}_j$, transported into the $x_i$ direction by the oscillating velocity $u^{ac}_i$. The force per unit volume in the $j$ direction is the gradient of the momentum flux with respect to $x_j$ and the minus sign ensures decreasing momentum flux results in a net positive force.

Having derived the governing equations for acoustic streaming, it is time to examine the forcing function in a little more detail. We will first consider a 1D propagating plane wave and then generalize the results for an arbitrary wave.

For a plane wave propagating in the positive $x$ direction, the sound oscillations only occur in the $x$ direction and can be completely represented by the $x$-component $u^{ac}$. Consequently, the body force described in the last term of the RHS of equation (2.10) becomes:

$$
F(x) = 2 \rho_0 u^{ac} \frac{du^{ac}}{dx} \tag{2.11}
$$

The oscillating fluid perturbation in the $x$-direction, $u^{ac}$, is represented as an exponentially decaying sound wave with initial wave amplitude of $A$:

$$
u^{ac} = A e^{-ax} \cos \left[ \omega \left( t - \frac{x}{c} \right) \right] \tag{2.12}$$
where $\alpha$ is the absorption coefficient and has units of m$^{-1}$. The next step is to calculate the derivative of $u^{ac}$ with respect to $x$:

$$\frac{du^{ac}}{dx} = -A \left\{ \alpha e^{-\alpha x} \cos \left[ \omega \left( t - \frac{x}{c} \right) \right] + \frac{\omega}{c} e^{-\alpha x} \sin \left[ \omega \left( t - \frac{x}{c} \right) \right] \right\}$$  \hspace{1cm} (2.13)

Substituting $du^{ac}/dx$ and $u^{ac}$ back into equation (2.11), the forcing function is equal to:

$$F(x) = -2\rho_0 A^2 e^{-2\alpha x} \left\{ \frac{\omega}{c} \sin \left[ \omega \left( t - \frac{x}{c} \right) \right] \cos \left[ \omega \left( t - \frac{x}{c} \right) \right] + \alpha \cos^2 \left[ \omega \left( t - \frac{x}{c} \right) \right] \right\}$$  \hspace{1cm} (2.14)

The temporal average of the first term on the RHS is zero due to the orthogonality of cosine and sine functions. The second term averages out to $\frac{1}{2}$. Therefore the acoustic streaming force per unit volume becomes:

$$F(x) = -\rho_0 A^2 \alpha e^{-2\alpha x}$$  \hspace{1cm} (2.15)

We can see that this body force decreases exponentially along the axis of propagation. To find the pressure distribution, we can simply integrate equation (2.15) with respect to $x$. This pressure distribution is:

$$P(x) = \frac{\rho_0 A^2 e^{-2\alpha x}}{2}$$  \hspace{1cm} (2.16)

It is worthwhile to express this forcing function in terms of the sound intensity $I(x)$. The intensity $I(x)$ is proportional to the square of the wave amplitude. Consequently, its amplitude decays at a rate of $e^{-2\alpha x}$. The intensity can be related to the sound wave amplitude by the following relation [28]:

$$I(x) = I_0 e^{-2\alpha x} = \frac{\rho_0 c A^2}{2} e^{-2\alpha x}$$  \hspace{1cm} (2.17)
Here \( c \) is the speed of sound in the fluid medium. Substituting equation (2.17) into (2.15), the force per unit volume has the alternate form of:

\[
F(x) = -\frac{2\alpha I(x)}{c}
\]  
(2.18)

Finally for an arbitrary wave with intensity components in all three directions, equation (2.18) takes the form:

\[
F_j = -\frac{2\alpha I_j}{c}
\]  
(2.19)

The simplicity of equation (2.19) makes it very useful for numerical simulations. One simply needs to solve for the acoustic intensity field then multiply the intensity by a constant to find the force field imposed by acoustic streaming.

2.3 Addressing the Inadequacies of Current Quartz Wind Micropumps

To our knowledge, the only quartz wind micropump reported was by Rife et al. [25]. We believe the major reason for the relative neglect of quartz-wind streaming in microfluidic applications is due to the difficulty of producing a propagating wave in the axial direction of a channel. Rife et al. used a custom manufactured BaTiO₃ piezoelectric array attached to a micro-machined PMMA block in their quartz wind device. Their approach requires custom machining which severely limits further miniaturization of the device (their transducer and channels have lateral dimensions of the order of mm’s). This integration method is also not scalable which prevents the development of complex flow networks.

The goal of this project is to overcome the short comings of current quartz wind micropumps. The integration scheme of the piezoelectric transducers with the flow channels must be scalable and modular to allow for the design of complex flow networks in the future. The processes in the fabrication of the micropump should be compatible with lithography-based microfabrication
procedures to facilitate the adoption of the system. Lastly, the micropump system will be designed to accommodate a wide range of biological and chemical experiments.
3 Design Scheme and Simulations

As was mentioned at the end of chapter 2, one of the most technologically challenging aspects of implementing a quartz wind acoustic streaming micropump is to direct an ultrasound wave along the axial direction of a channel. The most direct approach is to position the transducer vertically on a wall that faces the end of a microfluidic channel. However, this solution is incompatible with lithography-based MEMS fabrication process and would require the development of a customized fabrication sequence.

The approach used in this work is to design and fabricate planar piezoelectric ultrasound transducers using conventional microfabrication processes and then redirect the vertical sound waves emitted by the transducers into horizontally propagating ones by the use of an acoustic reflector.

3.1 Acoustic Reflector Solution

The idea is to use an inclined solid surface to reflect the vertically propagating sound wave into horizontally propagating ones. This concept is illustrated in figure 3.1.

![Figure 3.1: Schematic diagram of the acoustic reflector concept](image-url)
This design is only feasible if most of the wave energy can be reflected at the liquid-reflector interface. The ratio of reflected to incident energy is given by the intensity reflection coefficient, $R_I$, expressed as [29]:

$$R_I = \left[\frac{Z_{o2} \cos \theta_i - Z_{o1} \cos \theta_t}{Z_{o2} \cos \theta_i + Z_{o1} \cos \theta_t}\right]^2$$  \hspace{1cm} (3.1)

For our case, $Z_{o1}$ and $Z_{o2}$ are acoustic impedances of the working fluid and the solid reflector, and $\theta_i$ and $\theta_t$ are the angles of incidence and transmission. For an incident wave that is perpendicular to the interface, the reflection coefficient can be simplified to:

$$R_I = \left(\frac{Z_{o2} - Z_{o1}}{Z_{o2} + Z_{o1}}\right)^2$$  \hspace{1cm} (3.2)

As can be seen from equations (3.1) and (3.2), there needs to be a large impedance mismatch between the working fluid and the reflector material for most of the energy to be reflected. This design criterion immediately eliminates the possible use of PDMS for accommodating microfluidic channels (acoustic impedance of PDMS and water are approximately 1.1 MRayl and 1.5 MRayl [30]). If PDMS is used as the channel layer, the wave energy would be transferred into the PDMS where it would not be available to initiate directed fluid flow.

Consequently, we decided to use silicon as the material for the acoustic reflector. Its high acoustic impedance (around 20 MRayl [30]) ensures that most of the wave energy would be redirected along the axis of the microfluidic channel in the scheme shown in Figure 3.1. In addition, we can take advantage of anisotropic etching of silicon to produce slanted sidewalls with 54.7° from the horizontal plane. Although this angle is different from the ideal reflector angle of 45 degrees, it is sufficiently close so that most of the wave energy will still be directed along the channels after reflection off a slanted side wall. A more detailed discussion of silicon anisotropic etching will be discussed in chapter 4.
3.2 Numerical Simulation

Before designing the complete microfluidic system including the piezoelectric transducers and the silicon micro-channels, numerical simulations were conducted to predict the overall flow patterns of a quartz wind acoustic streaming device, using the concept depicted in Figure 3.1. The commercial finite element software COMSOL was used for this purpose (version 3.5a, Burlington, MA). This software package was chosen because of its ease of conducting simultaneous multiphysics simulations. For example, the piezoelectric vibrations of a transducer can be transferred to the nearby fluid medium and the resultant pressure waves of the fluid are transferred to the channel walls as localized wall acceleration. The vibrations of the walls can then be sent back into the fluid and piezoelectric elements. This simultaneous approach yields a more accurate solution compared to staggered methods where the solution from one physical system is stored and later manually transferred to a different computer software to solve a separate physics problem.

The decision to use a 3D or 2D numerical model involved the following considerations. 3D simulations can provide rich details about the physics of the phenomenon but are slower than their 2D counterparts and would require much more memory resources. Consequently, a coarser mesh is required for 3D simulations. However, to accurately model pressure waves, it is necessary to have a minimum of two quadratic mesh elements within the distance of one wavelength. That is because a minimum of two parabolas are needed to approximate a sinusoidal signal.

For a sound wave propagating in water with an excitation frequency of 50 MHz. The maximum element dimension, \( m \), would be:

\[
m = \frac{\lambda}{2} = \frac{c}{2f} = \frac{1500 \left[ \frac{m}{s} \right]}{2 \times 50 \times 10^6 [s^{-1}]} = 15 [\mu m]
\]  

(3.3)

Due to the small size of these elements and the need to compute simultaneous coupled physics calculations, we chose to use 2D models for COMSOL simulations in this project.
3.2.1 T-junction Simulation

Figure 3.2 shows the pressure distribution of one of the COMSOL simulations we conducted. This geometry represents the top view of a T-junction in a microfluidic network. Note that these simulations differ from the real situation in which the channel lengths are of the order of a few cm’s. Due to this difference, the flow resistances in these simulations are much lower than in the actual device and the acoustic forcing is much stronger than in the real life (since the acoustic body force decays exponentially with propagating distance). Consequently, the flow rates obtained here are much higher than the results reported by the literature. However, these simulations provide us insight on what the flow patterns would be like in the near-field and medium/far-field of a transducer.

For this simulation, the transducer is placed on the left side of the geometry and attached to a channel side wall. This simulation was conducted to test the ability of COMSOL to model the transfer of momentum from an ultrasonic wave into a working fluid, although this exact pumping geometry is not available with our microfabrication techniques. The boundaries of the side channels were set as the inlets and the end of the main channel was set as the outlet. The hydrodynamic pressures at all inlets and outlets were set to be zero so that the fluid patterns were not influenced by external pressure differences. An excitation frequency of 50 MHz was used for this simulation.

From figure 3.2, we see that part of the pressure wave is distributed to the side channels while the majority of the pressure wave is directed to the central channel. A corresponding sound intensity distribution is shown in figure 3.3.
Figure 3.2: Numerical simulation of the acoustic pressure distribution of a T-junction

Figure 3.3: Numerical simulation of the sound intensity distribution of a T-junction
The sound intensity, as defined in equation (2.17), is a measure of energy propagation per unit area. As shown in figure 3.3, the majority of the sound energy is directly towards the main channel. Having now found the acoustic intensity, we can make use of equation (2.19) to find the acoustic streaming body force within the fluid and calculate the streaming flow velocity. The velocity profile at this T-junction is shown in figure 3.4.

![Figure 3.4: Numerical simulation of the streaming velocity of a T-junction](image)

We can see the fluid is drawn in from the side channels and expelled along the central channel. The suction of the fluids from the side can be explained by two related phenomenon. The first one is mass conservation where fluid must enter to replace the mass that was pushed away by acoustic streaming. The second phenomenon is that a low hydrodynamic pressure zone is created immediately in front of the transducer. Note that the hydrodynamic pressure is the steady pressure distribution in the streaming flow field and is a separate parameter from the oscillating acoustic pressure of the sound field. A plot of the hydrodynamic pressure distribution is shown in figure 3.5.
3.2.2 Reflector Simulation

Having completed the simple T-junction simulation, we next simulated the near-field conditions for an acoustic reflector geometry. The pressure distribution for this geometry is shown in figure 3.6. In figure 3.6, the plane of the transducer piezoelectric disk is oriented horizontally at the top of the channel, and firing vertically down into the working fluid at an angled wall. To model the out-of-plane influx of the working fluid, we set one of the walls (highlighted in red) to be a fluid inlet (the pressure is set to be zero).
Compared to figure 3.2, we can see the pressure field is a lot more chaotic and less directional than the case for the T-junction. That is because there are multiple reflections in the vicinity of the reflector. However, the directionality of energy propagation is more evident in the plot of acoustic intensity as shown in figure 3.7.
As can be seen from the directional arrows, most of the intensity is reflected off the acoustic reflector into the horizontal channel. This indicates that most of the streaming force is directed down the channel. This observation is confirmed by taking a look at the fluid velocity plot in figure 3.8.
Figure 3.8 shows the fluid enters the channel via the inlet, and then is pushed downward and into the horizontal channel by the acoustic streaming body force. There is also a weak vortex underneath the transducer where the fluid is drawn in by a low pressure zone and then pushed downwards. The hydrodynamic pressure distribution of this geometry is shown in figure 3.9.
Figure 3.9: Numerical simulation of the hydrodynamic pressure in a reflector geometry

Again there is a low pressure zone near the transducer.

3.2.3 Trends from Numerical Simulations

The numerical simulations of an ultrasonic micropump have shown that an acoustic reflector can be used to effectively redirect the momentum transfer from the vertical direction to the axial direction of a micro-channel. We have also learned that a low pressure zone is created near the transducer surface. A successful pump/network design should utilize this effect to help draw the fluid in from the side channels and hence increase the flow rate of the device.

3.3 Integration Scheme

With the trends of the numerical simulations in mind, we devised the following network designs utilizing KOH anisotropic etching to create systems of microchannels with acoustic reflectors. The L-corner design is shown in figure 3.10.
In figure 3.10, the working fluid is drawn in from the left and pushed out through the top along the vertical channel. The piezoelectric transducer (shown as a circular disk), is positioned on top of the slanted sidewall that is parallel to the bottom of the page. The round interior corner of this design is created by convex angle etching inherent in the KOH etching process. One could alternatively use the perfect corner approach described in [31] to obtain right-angled bends. However, doing so would significantly increase the complexity of the fabrication process. In addition, the rounded corner can aid the fluid through the turn and minimize the effects of flow separation at the corner [32].

An alternate T-corner design for testing a prototype micropump is presented in figure 3.11. Fluid streams are drawn in from the left and right channels and expelled out through the top. This design can also serve as a mixer if different fluids are fed through the two inlet channels.
Figure 3.11: T-corner acoustic reflector design using KOH etching

The following chapter will discuss the fabrication process for implementing the designs shown in figure 3.10 and 3.11.
4 Device Fabrication

4.1 P(VDF-TrFE) Transducer

A major component of this project is the design, fabrication and characterization of the piezoelectric transducers that serve as flow actuators using the principle of acoustic streaming. Drawings of the integration schemes used for our micropump prototypes are included figures 3.10 and 3.11.

For our application, we require the transducer to be embedded inside a microfluidic channel. Consequently, the transducers must have a small active area (~0.1 mm in diameter) and be chemically resistant to the working fluid. Since the purpose of the transducer is to deliver acoustic power to the working fluid, the acoustic impedance of the piezoelectric material should be low to minimize the amount of power that is reflected at the transducer-fluid interface. In addition, since the acoustic absorption coefficient of an ultrasound beam is proportional to the square of the actuation frequency (see section 2.2), the transducer was designed to operate at high frequencies (> 50 MHz) so that most of the acoustic power is absorbed within the length of the microfluidic channel. The requirement for high frequency operation dictates that the transducer must be thin (a few micrometers) since the resonance frequency of the transducer is inversely proportional to the thickness of the piezoelectric layer.

Given these performance requirements, a piezoelectric polymeric material was selected according to its good acoustic impedance match with water and its compatibility with deposition by spin-coating [33]. Since spin-coating is a well-characterized cleanroom fabrication process, it allows us to easily adjust the piezoelectric layer thickness and thereby obtain the desired operation frequency.

From a list of available piezoelectric polymers (see Table 4.1), the copolymer vinylidene fluoride combined with tetra fluoroethylene, P(VDF-TrFE), was chosen because of its overall favorable piezoelectric properties and lower dielectric and elastic losses compared to pure PVDF polymer. In addition, P(VDF-TrFE) does not require a stretching procedure to convert the non
piezoelectrically polarizable \( \alpha \)-phase into the polarizable \( \beta \)-stage prior to poling as PVDF does [34]. The fact that stretching is not required simplifies the fabrication process and allows for direct deposition of the copolymer onto a substrate and consequently enhances the acoustic performance and mechanical robustness of the transducers. From a study conducted by Koga and Ohigashi [35], it was determined that the optimal molar fraction of VDF should be between 65 and 80\% to achieve the highest electromechanical coupling coefficient \( k_t \). Therefore, we used 75/25 mol \% P(VDF-TrFE) pellets. (Donation by Mr. Mitch Thompson from Measurement Specialties based in Hampton, VA, USA)

<table>
<thead>
<tr>
<th>Property</th>
<th>PVDF</th>
<th>P(VDF-TrFE)</th>
<th>P(VDF-TeFE)</th>
<th>P(VDCN-VAc)</th>
<th>Nylon-11</th>
<th>Nylon-7</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density [kg/m(^3)]</td>
<td>1780</td>
<td>1880</td>
<td>1900</td>
<td>1200</td>
<td>1023</td>
<td>1115</td>
</tr>
<tr>
<td>Longitudinal Velocity [m/s]</td>
<td>2200</td>
<td>2400</td>
<td>2200</td>
<td>2620</td>
<td>2000</td>
<td>2000</td>
</tr>
<tr>
<td>Acoustic Impedance [MRayl]</td>
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<td>4.32</td>
<td>4.18</td>
<td>3.14</td>
<td>2.05</td>
<td>2.23</td>
</tr>
<tr>
<td>Clamped Dielectric Constant</td>
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<td>4</td>
<td>5.5</td>
<td>6</td>
<td>2.2</td>
<td>2.5</td>
</tr>
<tr>
<td>Dielectric Loss Tangent</td>
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<td>0.12</td>
<td>0.19</td>
<td>—</td>
<td>0.25</td>
<td>0.22</td>
</tr>
<tr>
<td>Mechanical Quality Factor</td>
<td>13</td>
<td>25</td>
<td>17</td>
<td>—</td>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>Thickness coupling Factor</td>
<td>0.15</td>
<td>0.3</td>
<td>0.21</td>
<td>0.22</td>
<td>0.11</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Table 4.1: Table of piezoelectric properties of different polymers (from [33])

Another important decision that had to be made was the selection of the optimal material for the transducer electrodes. The electrode material needs to be corrosion resistant as the transducers are located at the bottom of the microfluidic channels and could be subjected to attack from the working fluid. In addition, it should have good electrical conductivity to minimize electrical resistance. Silver was the first metal tested for the electrodes. However, experimentation showed that electron beam evaporated silver showed poor adhesion to both glass substrate material and a chromium adhesion layer. Consequently, a 150 nm layer of gold was used instead for the top and bottom electrodes despite of its much higher material cost and substantial mass loading on the piezoelectric material.

The last important component of the transducer is the protective layer that was deposited over the active area. Initially, we did not plan to include an acoustic matching layer over the top electrode due to the good acoustic impedance match between P(VDF-TrFE) and the working fluid.
However, we discovered that a significant portion of the piezoelectric polymer surrounding the designed active area above the bottom electrode also became active during transducer operation (see section 5.1 for more details). We explain this effect by the presence of fringe electric fields that occur during high voltage poling and the effect of electrically conductive fluids that enlarge the effective active area of the top electrode during electrical excitation. To eliminate this problem, we decided to add an electrically insulating layer over the top electrode and thereby remove the possibility of having the fluid acting as the counter electrode (see figures 5.2 and 5.3).

Although the main function of this protective layer is electrical insulation, it can also perform as an acoustic matching layer if the acoustic impedance of the material and its thickness are chosen carefully. The acoustic impedance of P(VDF-TrFE) and water are 4.3 and 1.5 MRayl respectively [36]. Consequently, a quarter-wave matching layer should have an acoustic impedance of approximately $\sqrt{(4.3)(1.5)} \approx 2.5$ MRayl [37]. Most materials with acoustic impedance in this range are polymers [30]. This result is encouraging as polymers are usually good electrical insulators. Parylene C was ultimately used for this layer because of its ideal acoustic impedance value of 2.6 MRayl and its excellent electrical insulating properties (volume resistivity of $6 \times 10^{16}$ $\Omega$-cm at room temperature) [38]. An additional advantage of Parylene C is that it can be conveniently deposited using a low pressure chemical vapor deposition (LPCVD) process with good thickness control.

In summary, our final transducer design has five main layers. The bottom most layer consists of a 2” × 3” borosilicate glass slide on which all subsequent layers are deposited. Its excellent optical transparency allows the use of microscopy techniques for flow measurements. Its high acoustic impedance compared to P(VDF-TrFE) allows the transducer to operate at quarter-wave resonance. The second layer contains the bottom gold electrodes while the third layer is the piezoelectric material itself. The fourth layer accommodates the top gold electrodes. The overlapping portions of the top and bottom electrodes define the active area of the transducer. The final fifth layer is a Parylene C protection layer which also functions as an acoustic matching layer. Descriptions of the fabrication sequences for each layer are presented in the following section.
4.1.1 Fabrication Sequence

The fabrication sequence is performed in two cleanrooms located at the ECTI Bahen prototyping facility and at the Toronto Microfluidic foundry. We have reviewed several P(VDF-TrFE) fabrication procedures reported in the literature [39-41] and have revised these procedure to suit our own design requirements. The final fabrication sequence (revised to address several obstacles encountered) is summarized in figure 4.2.

Figure 4.1: Schematic of the fabrication process. A: deposition of chromium/gold layer. B: patterning of bottom electrodes and contact pads for top electrodes. C: spincoating of P(VDF-TrFE) layer. D: deposition and patterning of top electrodes and soldering connections to contact pads. E: deposition of protective parylene layer.

Detailed descriptions of all processing steps are presented in the following sections.

4.1.2 Glass Substrate Cleaning

Experiments have shown that the cleanliness of the glass substrates is crucial to the adhesion of the bottom electrodes. Consequently, we have developed a cleaning protocol to remove small particles and traces of solvent residue from the glass slides. Although the use of “piranha etch”
(mixture of hydrogen peroxide and sulfuric acid) is a common way to clean microfabrication substrates [42], the use of concentrated acid makes this process inherently dangerous. Consequently, we have used an alkaline cleaning solution (Hellmanex II, Hellma Analytics, 8% by vol. in deionized water,) in place of piranha etch.

The glass substrates are first gently scrubbed with a cleanroom wipe that was sprayed with isopropyl alcohol. This step helps remove the more resistant residue marks that were created during the manufacturing and packaging of the glass slides. The slides were subsequently rinsed with the organic solvents isopropyl alcohol and acetone (ACS grade, Caledon Laboratories Ltd., Georgetown, ON, Canada) to remove the particles that were introduced in the previous scrubbing step. A nitrogen gun was used to completely dry the microscope slides. The next step involved placing the glass slides into the solution of Hellmanex II alkaline cleaning liquid for one hour. The alkaline solution is routinely used for cleaning glass substrates and is therefore very effective at removing any organic contaminants from the borosilicate glass slides. After the one hour cleaning session, the glass substrates were rinsed with de-ionized water and dried with a nitrogen gun.

4.1.3 Bottom Electrode Deposition and Patterning

An e-beam evaporator in the Bahen cleanroom (Auto 306, BOC Edwards, Crawley, UK) was used for depositing the bottom and top electrodes. The electron beam caused the atoms of the source material to evaporate and coat the target located above the source material inside the vacuum chamber [43]. A thin adhesive layer (~20 nm) of chromium was first deposited on glass followed by a 150 nm layer of gold. It is imperative to coat the gold on top of the chromium layer without interrupting the vacuum during the transition. If the sample is exposed to the ambient atmosphere before gold is evaporated, a thin oxide layer forms on the chromium and would prevent the gold layer from consistently adhering to the chromium.

After the entire glass slide was covered with chromium and gold, a positive photoresist (S1818 Shipley, Marlborough, MA) was spincoated on the glass slide and patterned using standard lithography. The transparency masks were designed in Adobe AutoCAD (San Jose, CA) and
printed by CAD/Art Services (Bandon, OR). Once the photoresist was exposed and developed, wet etchants type 1020AC and type TFA (Transene Copany, Danvers, MA) were used to selectively remove the gold and chromium layers respectively from the areas outside of the electrodes. After patterning the bottom electrodes, a photoresist remover (MS2001, Fujifilm Electronic Materials, North Kingstown, RI) was used to remove the unexposed positive photoresist on top of the electrodes.

4.1.4 Piezoelectric Polymer Deposition

The P(VDF-TrFE) pellets were first placed in a solution of methyl ethyl ketone, MEK (ACP Chemicals, Montreal, Quebec) at a weight ratio of 1:4.5. This optimal ratio was obtained from a series of spincoating experiments. A higher weight ratio of copolymer pellets to MEK solvent would increase the viscosity of the solution and reach a higher layer thickness after spincoating. However, if the weight ratio was too high, the solution became very viscoelastic which renders the uniform distribution of the solution over the entire sample challenging. After the copolymer pellets were placed in MEK, the solution was heated to 80 °C and a teflon coated stirring magnet was inserted to enhance the dissolving of the pellets into the solvent. A stirring time of approximately 2 hours was needed onto completely dissolve the copolymer pellets. Once the solution was prepared, it was spincoated on the sample at 1000 rpm for 60 seconds and provided a uniform coating with a thickness of approximately 7 micrometers according to profilometer measurements.

4.1.5 Top Electrode Deposition and Patterning

The top electrode layer was deposited and patterned in the same fashion as the bottom electrode layer. However, since a layer of gold was deposited on top of the piezoelectric layer, back side alignment was required to align the mask pattern on top of the sample with the bottom electrode pattern visible from the back side of the device. The MA6 aligner (Karl-Suss, Garching, Germany) in the ECTI Bahen prototype cleanroom performs back side alignment by storing an image of the top side mask pattern using the top optics and then aligning this fixed image with
the live image transferred by the bottom optics. Using this setup, we were able to achieve alignment accuracies of better than 10 micrometers.

Another difference to the procedure for patterning of the bottom electrodes is that MS 2001 cannot be used to remove the photoresist after wet-etching of the gold and chromium metals. Experiments revealed that Fujifilm MS 2001 chemically attacks the P(VDF-TrFE) copolymer film and causes it to delaminate. Consequently, a different method was employed to remove the layer of positive photoresist attached to the top electrodes. After performing photolithography and etching of the top electrodes, the device was exposed again to UV light of (365nm: 16 mW/cm²; 405nm: 29.6 mW/cm²) for 20 seconds to ensure the photoresist was completely exposed and therefore soluble in the developer. Afterwards, the photoresist was developed again and stripped. An optical microscope image of the aligned top and bottom electrodes is shown in figure 4.2.

4.1.6 High-voltage poling of P(VDF-TrFE)

The next step was to pole the P(VDF-TrFE) co-polymer to align its dipoles and render the material piezoelectric. A previously constructed high-voltage DC poling device was used, with output voltage can be variable from 0 to 5000V. A voltage of 1500V was used because it was determined experimentally that a higher voltage difference across the piezoelectric material
would cause an electrostatic spark. Electrostatic sparks are undesired because they are uncontrolled releases of energy that would locally destroy the P(VDF-TrFE) film itself. We performed the poling procedure before the deposition of the protection coating because to avoid exceeding the dielectric field strength of the parylene C protection layer.

4.1.7 Acoustic Matching/Protective Layer

The desired thickness of the acoustic matching layer was initially determined as follows. Since the P(VDF-TrFE) layer is deposited on a mechanically stiff substrate, we expect the co-polymer layer to act at the quarter-wave resonance mode. The acoustic matching layer, it should also be one quarter wavelength in thickness to optimize the transfer of acoustic energy from the co-polymer to the working fluid.

The thickness of the piezoelectric layer is therefore given by \( t_c = \frac{\lambda_c}{4} = \frac{v_c}{4f} \), where subscript c designates the piezoelectric material, \( \lambda \) is the wavelength, \( v \) is the speed of sound in the material and \( f \) is the excitation frequency. Similarly, the thickness of the matching layer is \( t_m = \frac{v_m}{4f} \). Consequently, the thickness ratio of the matching layer to the piezoelectric layer should be \( \frac{t_m}{t_c} = \frac{v_m}{v_c} \). Since the speeds of sound in P(VDF-TrFE) and parylene C are approximately 2400 and 2200 m/s, respectively [44], approximately 0.92 is expected for the ratio.

The thickness of the coating is controlled by the mass of dimer placed in the Parylene C evaporator. Every gram of Parylene C dimer would result in an incremental increase to the coating layer thickness of approximately 0.6 µm. Once the dimer is put into the evaporation boat, the furnace is heated to 690 °C and the dimer begins to evaporate under vacuum conditions. However, the coating chamber remains at room temperature because the parylene monomer carries little thermal mass. The room temperature condition makes the coating process compatible with P(VDF-TrFE).

4.1.8 Electrical Connections
Since the melting temperature of P(VDF-TrFE) is approximately 160 °C [35], it is not suitable to use regular soldering materials for making electrical connections. Instead, we have used a 52 percent indium/48 percent tin eutectic alloy solders (AIM Special Materials, Cranston, RI) with a melting temperature of only 118 °C. The soldering iron was set at a temperature at 130 °C during the application of the solder. Although this temperature approaches the Curie temperature of the piezoelectric copolymer (approximately 135 °C for 75/25 mol ratio P(VDF-TrFE) [33]), the electrical contact surfaces were sufficiently removed from the active area that local heating at the electrical tabs did not degrade the piezoelectric performance of the transducers. A picture of the finished transducers is presented in figure 4.3.

![Figure 4.3: Completed transducer on 2” × 3” glass substrate with BNC connector attached.](image)

4.2 Microfluidic Channels in Silicon Substrate

The second fabrication component of this project involved the design and fabrication of the microfluidic channels that are compatible with the P(VDF-TrFE) transducers. The physics of acoustic streaming dictates that the body force created by wave attenuation acts in the direction of decreasing wave intensity (see section 2.2). Consequently, the bulk fluid flow initiated by acoustic streaming is tangential to the direction of wave propagation. Having this notion in mind, the microfluidic channels must be designed as wave-guides in addition to functioning as fluid flow networks.
In order for the acoustic wave to be guided along the channels, a large acoustic impedance mismatch is required at the interface between the fluid and the channel substrate material. This design criterion immediately eliminates the possible use of PDMS for accommodating microfluidic channels (acoustic impedance of PDMS is around 1.1 MRayl [30], [30]), which is similar to that of many biological fluids that might be flowing though a microfluidic network.). If PDMS is used as the channel layer, much of the wave energy would be transferred from the fluid into the PDMS which renders it very difficult to initiate directed fluid flow.

Another important selection criterion for the microfluidic channel material is the ability to fabricate small slanted surfaces that are approximately inclined from the vertical by 45 degrees. Since the P(VDF-TrFE) transducers are planar, an angled reflector is needed to translate the vertically propagating acoustic waves into horizontal waves so that the wave attenuation and hence the body force is along the longitudinal directions of the channels. This requirement can potentially pose significant fabrication challenges as it is very difficult to micro-machine slanted surfaces at such small scales. We used anisotropic silicon etching to solve this technical challenge.

Anisotropic etching of silicon occurs due to differential etching rates along the different crystal planes of a silicon wafer [45]. Using a wet etch agent such as potassium hydroxide (KOH), the etching speed in the <111> crystal direction will be much slower than in the <100> direction [45]. Consequently, for <100> wafers, KOH etching would form sidewalls at an angle defined by the <100> and <111> direction vectors. This angle from the horizontal, $\alpha$, can be easily calculated as follows.

$$\alpha = \cos^{-1}\left(\frac{\sqrt{1^2 + 0^2 + 0^2}}{\sqrt{1^2 + 1^2 + 1^2}}\right) = \cos^{-1}\left(\frac{1}{\sqrt{3}}\right) \approx 54.7^\circ$$

(4.1)

Although this angle is different from the ideal reflector angle of 45 degrees, it is sufficiently close so that most of the wave energy will still be directed horizontally along a channel after reflection off a slanted side wall. The ability to produce consistent slanted sidewalls and its high acoustic
impedance (approximately 19 MRayl [30]) makes silicon the most suitable candidate material to accommodate the microfluidic flow network.

4.2.1 Fabrication Sequence

Before we started experimenting with KOH wet etching, we consulted the literature [46] to determine the etch rates of the various processes to determine the processing time required and which materials should be used as the masking layers. These decisions will be explained in the following sections.

4.2.2 Silicon Nitride Deposition

Since the etch rate of 30 weight % KOH on positive photoresist is greater than 18 µm/min, the use of positive photoresist as the masking layer for KOH channel patterning is impractical. Therefore, a layer with a higher selectivity to KOH is needed to protect the areas outside of the microfluidic channels during KOH etching. Since KOH etching is usually conducted in a bath, both sides of the wafer must be protected to prevent unwanted etching. As suggested by Williams et al. [46], a highly selective masking material for KOH etching is LPCVD silicon nitride, for which the etch rate is virtually zero in KOH.

Initially we had attempted to deposit silicon nitride on N-type <100> wafers using the PECVD machine in the ECTI Bahen cleanroom. However, the deposition process proved to be very time-consuming as only one side of a single wafer can be processed at a time. In addition, the adhesion of the silicon nitride film on the wafers is very sensitive to the cleanliness of the wafers prior to deposition. Due to these reasons, we decided to purchase commercially available LPCVD nitride deposited wafers from University Wafers (South Boston, MA). These wafers have 300 nm of LPCVD stoichiometric silicon nitride deposited on each side.

4.2.3 Method for Patterning Silicon Nitride
According to [46], reactive ion etching (RIE) is one of the most efficient ways to remove silicon nitride. The etch rates of RIE using the gases SF$_6$ + O$_2$ and CF$_4$ + O$_2$ on silicon nitride are 150 and 120 nm/min, respectively. Consequently it would take only a few minutes to completely remove the 300 nm of silicon nitride deposited on the sample wafers. According to Williams et al. [46], SF$_6$ + O$_2$ and CF$_4$ + O$_2$ RIE treatments etch positive photoresist at rates less than 200 nm/min. Both gasses are therefore compatible with standard lithography processing using S1818 photoresist since the resist layer is a few micrometers thick. Despite their similar etch rates for silicon nitride and positive photoresist, SF$_6$ + O$_2$ and CF$_4$ + O$_2$ exhibit drastic differences at etching plain silicon. SF$_6$ + O$_2$ etches <100> silicon wafers at a rate of 1500 nm/min whereas the etch rate for CF$_4$ + O$_2$ is only at 95 nm/min. This result shows that CF$_4$ + O$_2$ or a similar RIE treatment should be used as it prevents undesired etching of plain silicon during the silicon nitride removal process. After a discussion with the staff at the ECTI cleanroom, we learned that CF$_4$ is not available but CHF$_3$ can be used as a substitution. Experimentation has shown that CHF$_3$ is effective at etching silicon nitride but has negligible effects on silicon and S1818 photoresist.

4.2.4 Micro-Channel Fabrication Sequence

To provide fluid access into the microfluidic channels, inlet and outlet holes through the back side of the wafer are required to externally supply fluid to the chip and remove fluid from it after it has flowed through the microfluidic network. KOH etching was again employed for this task in order to reduce the complexity of the fabrication sequence. Since the access holes and channels were designed to have different depths, a time-staggered etching sequence was used starting with the deepest features. The etching sequence for the microfluidic channels are shown in figure 4.4.
Figure 4.4: Schematic of the etching process. A: deposition of silicon nitride on both sides of the 500 um thick wafer. B: patterning of silicon nitride on the back side. C: timed etching of the back side access holes D: patterning silicon nitride for the top channels E: etching of the wafer on both sides.

S1818 photoresist was used to first pattern the back side of the wafers (the surface from which the inlet and outlet holes are etched). To eliminate unwanted etching on the frontside of the wafer during RIE etching, photoresist must also be spincoated onto the frontside. The back of the wafer was then patterned using standard lithography. During alignment, the patterns were aligned to the <110> directions of the wafer (indicated by the primary and secondary flats) so the resultant features have straight side walls after completing the etch.

After patterning the photoresist, the wafer back side was etched with CHF₃ + O₂ RIE for 10 minutes. The elongated treatment time ensured the complete removal of the exposed nitride layer. The over-etching of silicon is insignificant compared to the feature depths (~100 ums). After nitride patterning and subsequent removal of photoresist, the entire wafer was immersed in a 30 weight % KOH bath controlled at a temperature of 80 °C for approximately 80 minutes. This would allow the inlet/outlet holes to gain a “head start” and reach a greater depth compared to the microfluidic channels once the entire etching process was completed.
After the back side access holes had reached a desired depth (approximately 100 µm), the wafer was removed from the KOH bath and subsequently cleaned with piranha (1:3 by volume mixture of hydrogen peroxide and sulfuric acid). This cleaning step was important in preventing peeling of the silicon nitride thin film during subsequent KOH etching. The microfluidic channel patterns were transferred to the front side of the wafer by S1818 photoresist. Back side alignment was used before exposure to align the fluid access holes with the channel patterns. Once the photoresist was patterned, the front side is RIE etched to expose the silicon substrate.

The S1818 photoresist was then removed and the entire wafer was placed in a 30% KOH bath for approximately 3 hours. Visual inspection was used to determine the time of intersection of the back side access holes with the front side channels. The wafer was etched for an additional 15 minutes after the time of intersection to enlarge the through holes at the bottom of the microfluidic channels. After KOH processing, the wafer was cleaved along the parting lines that were included in the front side channel pattern. The silicon chips were then ready to bond with the piezoelectric transducers.

4.3 Bonding of Piezoelectric Transducers to the Microfluidic Channels

The final phase of the fabrication of the acoustic streaming flow actuator prototype involves the integration of the fluid channels with the piezoelectric transducers. The active area of the transducer must be aligned to the corner edge of a channel for the sound wave to be reflected along the longitudinal direction of a channel (refer to figure 4.3). We had initially experimented with vacuum and pressure sealing systems but these techniques proved to be ineffective. Since the surfaces of the piezoelectric transducers and the channel chips consist of rigid surfaces, small imperfections such as dust particles and scratches would cause the device to leak due to incomplete sealing. Independently, alignment was also a major technical difficulty for the non-permanent sealing systems.

Due to the problems experienced in the reversible sealing methods, we decided to integrate the flow actuators with the silicon channel chips using a permanent sealing method. The use of a permanent seal enables us to separate the bonding and flow characterization tasks into two
separate steps. Consequently, the bonding step can now be conducted in a cleanroom environment where the presence of dust particles is minimized. In addition, the microfluidic channels can now be joined to the transducer using an optical alignment system in a cleanroom. The next step was to determine the most suitable bonding method to use. The most important constraint of the bonding procedure is that the process itself cannot exceed the melting temperature of P(VDF-TrFE) which is around 160 °C [35]. This requirement immediately eliminates the possible use of anodic bonding as an integration technique for our prototype. Instead, we had to adopt a low temperature bonding technique. Most low temperature bonding techniques reported in the literature make use of an intermediate adhesive layer to combine the two components. For our application, it is imperative that any such adhesive layer be sufficiently thin that the wave energy produced by the transducer would not be drastically attenuated by the adhesive. The adhesive must also not flow into the microfluidic channels which are of the order of 100 µm deep.

4.3.1 Experimentation with Adhesive Layers

There are many reports of the successful use of low temperature adhesives in the literature. The sealants include UV-curable adhesives[47-49], fluoropolymers[50], and parylene[51-53] among others. The UV adhesive NOA-81 (Norland Products, New Jersey, USA) was tested first for this project because it does not require the application of heat or high pressure during bonding. However, it was difficult to obtain a thin uniform layer of the adhesive on the transducer. The literature describes a technique of placing a slab of PDMS on top of a thin layer of NOA-81 adhesive before UV curing [48]. The working principle is that oxygen molecules present at the surface of a gas-permeable PDMS layer inhibit the free radical polymerization of NOA-81 under UV excitation. We found the reproducibility of the method to be low due to the manual placement of the PDMS slab. The resultant adhesive layer also contained voids due to the presence of air gaps between the PDMS and the adhesive. Due to these setbacks, we next experimented with Parylene C as the adhesive layer to bond the transducer to the silicon wafer containing the microfluidic channels.
Parylene C was a promising material to use as the adhesive layer because it was already being used as the acoustic matching layer of the transducer. Consequently, the deposition method and its physical properties were well understood. According to the literature [51-53], Parylene bonding is conducted by depositing half of the desired thickness of the Parylene layer on each surface to be bonded and pressing the two surfaces together under elevated temperature and pressure. It is theorized that when a compressive stress is applied between two Parylene-coated surfaces that are heated to a temperature higher than the glass transition temperature (~100°C), the polymer chains on opposite side of the interface will physically entangle and thus form a permanent bond[51]. According to Kim and Najafi, a bond fabricated at 125 °C with an applied pressure of 102 kPa has an ultimate failure strength of 1.6 MPa, which is sufficient for our application and significantly greater than the highest pressure in the channel. However, our experiments have shown that the resultant adhesive bond is non-uniform in our application and prone to leakage from the micro-channels. The exact failure mechanism of the bond is still unknown and would require further investigation.

Because of the inability to achieve uniform bonds with effective seals using the previous methods, we then experimented with the fluoropolymer CYTOP to link the silicon chip to the piezoelectric transducer. The bonds obtained using CYTOP as the adhesive were more uniform than those obtained with Parylene C or NOA-81, and were mostly leakage free. The bonding procedure using CTYOP will be documented in the sections below.

4.3.2 CYTOP Fluoropolymer Application and Silicon Chip Preparation

The CYTOP fluoropolymer used for our experiments was of type CTL-809A (donation by Mr. Kunio Watanabe of Asahi Glass Company, Ibaraki, Japan). According to the technical brochure provided by Asahi, type A CYTOP has a –COOH end functional group and therefore requires a silane surface treatment to be applied to a silicon or silicon nitride substrate to form a covalent bond with the fluoropolymer. The chemical bonding mechanism is described in figure 4.5.
Consequently, we treated the silicon channels with trichloro (1H, 1H, 2H, 2H-perfluorooctyl) silane (Sigma-Aldrich Chemistry, St. Louis, MO) before fabricating the adhesive bond. The silicon chip containing the microfluidic channels was placed in a dessicator with an opened vial of the silane solution at the side. The dessicator was then evacuated to a pressure of -25 inHg gauge so that the silane could evaporate and coat the silicon sample. This treatment rendered the surface of the silicon hydrophobic to de-ionized water.

The application of the CYTOP layer to the piezoelectric transducer was conducted using a spincoating process. A spin speed of 650 rpm was used to obtain a layer thickness of 3 μm. The sample was then heated to 100 °C for 20 minutes to completely remove the CYTOP solvent. It was important to use gradual temperature ramp ups and ramp downs (5°C per minute) to eliminate the occurrence of wrinkles on the CYTOP surface.

4.3.3 Aligning and Bonding Procedures

Equipment limitations forced the separation of the alignment and bonding operations into two separate operations, each with its own set of equipment as follows.

The first step was to “pre-bond” (weakly bond) the silicon chip to the piezoelectric transducer. The purpose of this step was to align the two components and form a temporary bond so that the
combined components could be transported to another facility for the final bonding step. The alignment was performed using an OAI Hybralign Series 200 aligner (OAI, San Jose, CA). An in-house designed heating unit was built incorporating a resistive heater and a temperature controller (Model CN8200-R1, Omega, Stanford, Connecticut) to regulate the temperature of the two components during this process.

After optical alignment of the two components, they were brought into contact by raising the z-axis stage of the OAI aligner. The surface of the stage was then heated to 135 °C using the in-house designed heating unit. This temperature was chosen because it was higher than the glass transition temperature of CYTOP (108 °C). After a heating time of 30 minutes and partial curing of the CYTOP adhesive, a temporary bond was formed between the transducer and silicon chip with microchannels. The combined specimen could then be safely transferred to a pressing station where a much higher bonding pressure, and higher temperature, could be applied.

According to [50], an applied pressure of approximately 7MPa and temperature greater than 135 °C is needed for a successful bond. To apply such large pressures at an elevated temperature, we used the temperature-regulated hydraulic press (Model M, Carver, Wabash, Indiana). The samples were stacked in the manner described in figure 4.6 for this final curing step.

Figure 4.6: Stacking of different layers during bonding.
The purpose of the copper shim beneath the transducer glass substrate was to provide rigid support to prevent the glass from cracking. The rubber on top of the silicon chip distributed the loading evenly to achieve a more uniform bond. After numerous iterations, an optimized temperature and compression pressure of 148 °C and 5.5 MPa were selected to obtain repeatable, uniform leak-free bonds. A close up photograph of a final successfully bonded system is presented in figure 4.7.

Figure 4.7: CYTOP bonding microfluidic channel with piezoelectric transducer.
5 Experimental Results and Discussion

After completing the fabrication sequence described in chapter 4, we proceeded to perform experiments using these devices to test their performance. The first experiments were conducted to evaluate the properties of the piezoelectric transducers and are described in Section 5.1. Flow measurement results are described in Section 5.2.

5.1 Piezoelectric Transducer Characterization

The fabrication of the piezoelectric transducers was one of the most technically challenging aspects of this project. It involved a sequential multi-layer process with a yield that is the product of the success rates of each individual steps. An error made in any of the intermediate steps could render the entire device useless. For this reason, the piezoelectric transducers were tested before they were combined with the silicon microfluidic channels.

5.1.1 Electrical Impedance Measurements

Electrical impedance measurements were conducted to find out the resonance frequency of the fabricated transducers. A network analyzer (Advantest Model R3754A, Santa Clara, CA) was connected to a Standing Wave ratio (SWR) bridge to measure the reflectance coefficient and hence the electrical impedance of the transducer. Typical results for the electrical impedance measurements are shown in figure 5.1.
Piezoelectric transducers that are attached to a rigid substrate (in this case, glass) are suited for operation in the quarter-wave resonance mode [37]. This means that the thickness of the transducer is equal to one quarter of the wavelength inside the piezoelectric material during excitation at the resonance frequency. Using this relationship, the theoretical resonance frequency can be estimated as:

\[ f_R = \frac{c}{\lambda} = \frac{c}{4t} \]  

(2.1)

Using values of \( c = 2400 \text{[m/s]} \) for P(VDF-TrFE) [44] and a measured thickness of 7.3 μm for the transducers, the theoretical resonance frequency comes out to be 82 MHz. It is noted from Figure 5.1 that a typical piezoelectric transducer fabricated using the procedure detailed in chapter 4 has a resonance at around 85 MHz. This is very close to the calculated theoretical value of 82 MHz.
The good agreement between the theoretical and measured results is an indication that the mass loading effects of the gold electrodes on the transducer is minimal for the deposited metal thickness (~150 nm).

As seen in figure 5.1, the application of a Parylene C coating decreases the resonance peak of the transducer. This suggests that more of the wave energy is passed on to the fluid medium due to quarter wave acoustic impedance matching [54], i.e., the resonance is broadened and its amplitude is decreased by energy removal (see section 4.1 for more details).

### 5.1.2 Active Area Measurements

After finding the resonance frequency of the fabricated transducers, we conducted ultrasound C-scans to determine their active area. For these experiments, the fabricated transducers were used as the pulsers that send out bursts of ultrasound signal in a water tank and a commercial 100 MHz ultrasound probe was used to detect the sound bursts. The commercial probe was positioned on a computer controlled two-axis translation stage and therefore was able to map the active area of the fabricated transducers.

It is important that the active area be small so that most of the input energy is directed into the working fluid (see figure 3.10 for the integration scheme). The first C-scan was conducted on a piezoelectric transducer without a Parylene protection layer and the result is shown in figure 5.2.
From figure 5.2, it is observed that the active area extends beyond the overlapping area of the top and bottom electrodes, i.e., beyond the area for which the transducer had been designed. In fact, the device was piezoelectrically active at areas defined by the (larger) bottom electrode. We explain this phenomenon by the presence of fringe electric fields that occur during high voltage poling and the effect of electrically conductive fluids serving as an extended area for the top electrode during electrical excitation. To eliminate this effect, we decided to add an electrically insulating layer (Parylene C) over the top (counter) electrode, and thereby remove the possibility of having the fluid acting as the counter electrode. A C-scan conducted after the deposition of the protective layer is shown in figure 5.3.
After the application of the Parylene layer, the active area was reduced to the overlapping area of the top and bottom electrodes, as desired for our pump design.

These characterization experiments showed the fabricated transducers had characteristics consistent with our intended design. We then bonded these piezoelectric transducers to the completed silicon channel networks using the process described in section 4.3. We then conducted flow experiments to characterize the pumping performance of our prototype quartz wind acoustic streaming micropumps, as described in Section 5.2.

5.2 Microscale Particle Image Velocimetry Measurements

To provide the sinusoidal voltage signal to power the acoustic streaming micropump, a signal generator (Wavetek model 3006, Indiana) was connected to a radio frequency power amplifier (Booton Radio Type 230A, Boonton, New Jersey). The signal generator is capable of generating sinusoidal voltage signals with amplitudes up to 1V and the amplifier can increase the voltage signal to 15 V rms when it is loaded with a 50 Ω device.
The inlet and outlet access holes on the microfluidic network were connected to pipette tips to facilitate the measurement of head difference in the flow experiments. The pipette tips also served as fluid reservoirs so that tubing connections to an external reservoir could be eliminated. A finished prototype with these pipette tips is shown in figure 5.4.

![Prototype acoustic streaming micropump with pipette types attached.](image)

To measure the velocity field in the microchannels, we employed the micro-scale particle image velocimetry (μPIV) technique. During PIV experiments, small particles are seeded inside the working fluid. Two images of the particle distributions closely spaced in time are captured by the PIV instrument; by correlating the change in the particle positions over the time difference of the two image frames, the flow velocity field is determined [55].

The schematic diagram of the florescence μPIV experimental setup that was used for our flow visualization experiments is shown in figure 5.5. A laser light (frequency doubled Nd:YAG, 532 nm) fires into a dichroic mirror cube and is reflected into the micropump. The tracer particles (1 μm polystyrene beads labeled with nile red fluorescence dye) would then fluoresce due to the laser excitation. Since the fluorescence of the nile red dye is at a different wavelength than the
Nd:YAG laser, the florescence light signal can then pass through the dichroic filter and be focused onto the imaging camera.

![Schematic setup of a florescence μPIV experimental setup.](image)

The particle imaging software used in our setup is Davis version 7.2 made by Lavision (Goettingen, Germany).

5.2.1 Near-Field Flow Measurements

The first μPIV experiments were conducted in the vicinity of the piezoelectric transducers to study the near field flow profile. Since the velocity profile in the near field is very complex and has components in all three directions, we took 2D flow measurements at different depths of an
L-corner in order to gain insight on the overall flow patterns. The experiment was conducted with the network in the horizontal plane, with the transducer aimed downwards. Flow enters the L-shaped corner from the right, and exits at the top of each picture. The velocity fields at different depths are shown in figures 5.6 to 5.11. Note that the outline of the electrodes and the channel walls as focused on the top plane \((z = 0)\) are superimposed on the flow profiles to better illustrate the location of the measurements. The \(z\)-axis is used to measure depth into the channel; it has an origin at the transducer-silicon interface and the positive direction is towards the bottom of the channel. The depth of the channel is approximately \(250 \, \mu m\). The influx of fluid from the right was not readily observable in these experiments, as the electrodes blocked optical access to this part of the network; the electrode leads should be redesigned in the future to minimize this problem.

Figure 5.6: Time averaged velocity at \(Z = 0 \, [mm/s]\).
Figure 5.7: Time Averaged Velocity at \( Z = 50 \text{ um} \) [mm/s].

Figure 5.8: Time averaged velocity at \( Z = 100 \text{ um} \) [mm/s].
Figure 5.9: Time averaged velocity profile at Z = 150 um [mm/s].

Figure 5.10: Time averaged velocity profile at Z = 200 um [mm/s].
Looking at figures 5.6 to 5.11, we can see that the fluid was sucked into this region of the microfluidic network near the top of the channel of the outlet (where Z is small) and expelled at a marginally greater overall flow rate at channel depths that are 100 μm or deeper, as indicated by the velocity directional arrows. This is similar to the COMSOL numerical simulation results where the fluids are drawn into the low hydrodynamic pressure zone near the transducer surface. From figure 5.8, we can also see a clockwise vortex near the center of the L-corner. We explain this result by the presence of a large channel breadth at this location created by convex corner KOH etching. Since the cross-sectional area at the convex corner is large, the overall flow velocity through the corner is small because of the conservation of flow rate along the microfluidic channel network. Consequently, the slow moving fluid near the interior corner is easily drawn in to replace the fluid mass that was pushed away by acoustic streaming.

A final observation is that the flow velocity near the middle of the channel (Z ~ 125 μm) is higher than the velocity near the top or bottom of the channel. This is expected because the wall shear stresses are dominant near the channel walls.
Overall, the near field velocity field agrees with the trends observed from the numerical simulations. However, the flow is less directional and more chaotic than expected. Further discussions about the implication of these results and future improvements will be presented in chapter 6.

**5.2.2 Far-Field Flow Measurements**

After performing the near field flow characterization, we proceeded to perform flow experiments in the far field of the pump’s transducer, downstream from the pump location. The far field experiments are needed to determine the pressure and the flow rate that this pump can provide. The most direct way to measure these performance characteristics is to first let the system fully equilibrate (so the fluid is stationary) with the fluid reservoirs (pipette tips) at the inlet and outlet of the network at equal heights such that there would be no externally applied net pressure to the system. We then turn on the acoustic streaming micropump. After a short transient response, the pump provided its maximum flow rate because the externally-applied head difference that it opposes was still close to zero. This maximum flow rate was measured. The pump is then allowed to keep running until the net flow rate slows to zero due to the build of a hydrostatic pressure drop between the inlet and outlet pipette tips. The maximum head at zero velocity can then be measured from the difference in reservoir heights at the inlet and outlet.

Although the described procedure is direct and simple, it proved difficult to implement for our prototype micropump. The major problem was that the beam profile fired out from the transducers was not broad enough to fill the entire cross-section of the channels. Consequently, the transducer was prone to induce recirculation vortices even in the far field of the channels. In addition, the head pressure provide by the micropump was too small to be accurately measured by the height of the water columns in the pipette tips.

Consequently, we have devised an alternative method to measure the flow rates and pressure provided by our micropumps. In this method, a small hydrostatic pressure difference was provided to the pipette tips so that the fluid would flow slowly in the desired direction of the pump. The micropump was then turned on and the difference in velocity before and after the
pump operation was recorded. Since the flow was already moving in the desired direction, vortices were somewhat suppressed.

The *increase* in the maximum flow speed can then be used to back calculate the approximate pressure provided by the pump. For laminar pipe flow in a noncircular duct, the relation between applied pressure and flow velocity can be expressed as [32]:

\[ \Delta p = \frac{C_h}{Re_h D_h} \frac{l \rho V^2}{2} \]  

(2.2)

Where \( C_h \) is a numerical constant based on the geometry, \( D_h \) is the hydraulic diameter, \( V \) is the averaged flow velocity and \( Re_h \) is the Reynolds number based on the hydraulic diameter. By substituting in the definition of the Reynolds number and after some rearranging, it can be shown that:

\[ V = \frac{2\Delta p D_h^2}{C_h \mu l} \]  

(2.3)

where \( \mu \) is the dynamic viscosity of the working fluid. As can be seen from equation (5.3), the average flow velocity is linearly proportional to the applied pressure and inversely proportional to the channel length. The average velocity can also be related to the maximum velocity by a numerical constant \( C_g \). This constant is dependent on the geometry of the cross-section of the pipe. Consequently, equation (5.3) can be rewritten in the form:

\[ V = \frac{2\Delta p D_h^2}{C_h \mu l} \]

\[ V_{max} = \frac{2\Delta p D_h^2}{C_g C_h \mu l} \]  

(2.4)

Although analytical solutions for \( C_g \) exist for different geometries [56] and one could consult fluid mechanics handbooks for values of \( C_h \), the use of such a hydraulic diameter estimate in equation (5.4) could lead to errors as large as 30 percent [32]. To obtain more accurate results, we
lumped $C_h$, $C_g$, $D_h$ and $\mu$ into one constant and conducted a simple COMSOL simulation to find the lumped parameter $C_l$.

The lumped equation can be expressed as:

$$\Delta p = C_l V_{max} l \quad (2.5)$$

Note that parameter $C_l$ has units of [kg/m$^3$·s]. For the COMSOL simulation, we drew a triangular cross-section with the same dimensions as the fabricated channel and extruded it to have a length of 2 mm. Different pressures were applied at the inlet and the resultant maximum velocities were recorded. Since we used a 1:10 volume mixture of glycerol to water as the working fluid, the effective viscosity and densities were calculated to be:

$$\rho = \frac{10(998) + 1261}{11} = 1023 \frac{[kg]}{[m^3]}$$

$$\mu = \frac{10(1.02 \times 10^{-3}) + 1.2}{11} = 0.109 \frac{[Pa \cdot s]}{}$$

A diagram of the COMSOL simulation is shown in figure 5.12.
By plotting $V_{\text{max}}$ against the pressure gradient and fitting a linear trendline in figure 5.12, the value for $C_L$ can be obtained.

Figure 5.11: COMSOL simulation of flow through a triangular cross-section channel

Figure 5.12: Simulated pressure gradient vs. $V_{\text{max}}$ plot to find the lumped parameter $C_L$
The value for $C_l$ was found to be $5.41 \times 10^7$ [kg/m$^3$·s] from our COMSOL simulation. A numerical study was also conducted to find the constant $C_g$. By using the definition in equation (5.4), we can see that the volumetric flow rate, $Q$, can be written as:

$$Q = VA = C_g V_{max} A$$  \hspace{1cm} (2.6)

Where $A$ is the cross-sectional area of the microchannel and is equal to \(\frac{(300 \times 10^{-6})(212 \times 10^{-6})}{2} = 3.18 \times 10^{-8}[m^2]\). Figure 5.13 shows the numerical results of average velocity $Q/A$ vs. $V_{max}$.

![Figure 5.13: Simulated $V_{max}$ vs. $V_{avg}$ plot to find the geometric parameter $C_g$](image)

By using the trend line, the geometric constant $C_g$ is found to be 0.47.

Having now determined all the required coefficients, we will now return to the $\mu$PIV experiments. We ran two separate experiments on two different micropumps with the same geometry to test the repeatability of the results. (Additional runs were not possible due to difficulties in fabricating defect-free pump/microfluidic networks). The time averaged velocity vector plots for the two runs, immediately after turning on the pumps are shown in figures 5.14 and 5.15. Although the goal was to turn on both pumps when the systems were initially at
hydrostatic equilibrium, it proved difficult to achieve this initial condition to the desired level of accuracy.

Figure 5.14: Time averaged velocity in the farfield for the first run.
For the second run, the initial hydrostatic pressure was greater and therefore the baseline flow velocity was higher. To extract useful data about the pump performance, it is therefore better to look at the maximum flow velocity in the channel as a function of time when the pump was being turned on. This yields the resultant *increase* of flow velocity due to the actuation of the piezoelectric transducers. The transient response plots for the first and second runs are shown in figures 5.16 and 5.17.
Figure 5.16: Transient response for the first run, maximum velocity vs. time [mm/s]

Figure 5.17: Transient response for the second run, maximum velocity vs. time [mm/s]
It is seen that the second run has a much higher increase in the maximum flow velocity compared to the first run. However, the oscillation amplitude for the second run is also substantially higher. The reason for this vast variation is still unknown and requires further investigation. It is interesting to note that tracer particle concentration was very evident in the first run while it was absent in the second run. This might have led to inaccuracies in the measurement of the velocity profile since the beads did not follow the fluid velocity closely. A fluorescent photograph of the bead concentration is shown in figure 5.18.

![Figure 5.18: Bead concentration observed in the first run.](image)

Figures 5.16 and 5.17 indicate that the maximum channel flow velocity $V_{max}$ increased by roughly 0.032 [mm/s] when the pump was turned on in the first microfluidic network, and by approximately 0.32 [mm/s] in the second network. Using equation (5.5) and a channel length of 22.6 mm, we find the total pressure differences added by the pumps were:
\[ \Delta p_1 = 5.41 \times 10^7 \left[ \frac{kg}{m^3s^1} \right] \times 0.032 \times 10^{-3} \left[ \frac{m}{s} \right] \times 22.6 \times 10^{-3} [m] = 39.1 [Pa] \]
\[ \Delta p_2 = 5.41 \times 10^7 \left[ \frac{kg}{m^3s^1} \right] \times 0.32 \times 10^{-3} \left[ \frac{m}{s} \right] \times 22.6 \times 10^{-3} [m] = 391 [Pa] \]

With these numbers we can also find the total power delivered by the pumps. Power is defined as the product of the pressure and the volumetric flow rate. For the two runs, the total kinetic power delivered was:

\[ P_1 = \Delta p_1 \times Q_1 = 39.1 [Pa] \times 0.47 \times 0.032 \times 10^{-3} \left[ \frac{m}{s} \right] \times 3.18 \times 10^{-8} [m^2] = 1.88 \times 10^{-11} [W] \]
\[ P_2 = \Delta p_2 \times Q_2 = 391 [Pa] \times 0.47 \times 0.32 \times 10^{-3} \left[ \frac{m}{s} \right] \times 3.18 \times 10^{-8} [m^2] = 1.88 \times 10^{-9} [W] \]

So there is an order of magnitude difference between the flow rates provided by the micropumps and two orders of magnitude of difference in terms of the kinetic power delivered. This could potentially be caused by the presence of a bubble in the first device that drastically increased the flow resistance of the channel. This could also explain the smaller flow fluctuations in the measurements of the first run due to the damping effects of a bubble. More experiments should be conducted to better understand the causes of the performance difference.

Due to time constraints, only two successful experiments were performed in the far fields of the micropumps. More experiments should be conducted in the future to test the repeatability of the results.
6 Conclusions and Future Work

6.1 Project Summary

During the span of this research project, we have successfully designed, fabricated and characterized a microfluidic pumping system using the working principle of acoustic streaming. A summary of our work and discussions of the implications will be presented in the following sections.

6.1.1 Design Phase

In the design phase, a novel transducer integration scheme was devised based on the acoustic reflector concept. By using a slanted reflector inside the microfluidic flow network, a planar transducer deposited on the base substrate can now be used for providing a propagating sound wave along the axial direction of a channel. This design scheme allowed us to use standard lithography-based fabrication technologies that have been developed in the semiconductor and MEMS industries. The modularity of the transducer substrate from the flow channels would allow the development of complex flow networks based on acoustic streaming in the future. We envision that researchers in the future will utilize a standardized transducer grid with integrated control circuitry and design their own flow networks to address their unique application needs.

We have also conducted finite element simulations to predict the flow patterns around the transducer and the feasibility of the acoustic reflector design. These studies showed that slanted sidewalls are capable of redirecting the wave momentum into the desired flow direction. Trends of the simulated hydrodynamic pressure distribution were also used to improve the design of the microchannel geometry.

6.1.2 Fabrication Phase

Due to the difficulty of obtaining commercial transducers with the desired high operating frequency and active area for our application, we developed and fabricated our own piezoelectric
transducers using the co-polymer P(VDF-TrFE) as the active piezoelectric element. Although other researchers had reported general descriptions of how to fabricate such transducers, there were a number of technical challenges during the development of the fabrication procedure; however, the overall development process was completed successfully.

The next major component of the fabrication phase was the etching of silicon channels used for both the flow network and the acoustic reflector. We developed a time-staggered etching procedure that would produce both the microchannels and fluid access holes at the back side of the silicon chip. We have learned that over-etching can lead to defects in the channels despite the fact that the channels have already reached a stable configuration. For this reason, a more accurately timed etching process should be employed in the future to increase the yield of the process.

The last crucial component of the fabrication phase was the bonding of the piezoelectric transducer substrate to the silicon chips. We faced many technical challenges in this step and the overall progress of this project was therefore delayed. Clogging of the channels and non-uniform bonding were the most severe problems that we faced. We learned that the adhesive must be very thin (~few micrometers) so that the adhesive would not reflow into the channels. After many trials, we finally achieved uniform bonds by the use of a heat pressing technique applied to a fluoropolymer. These devices showed no signs of leakage after repeated use.

6.1.3 Experimental Phase

After fabrication of the acoustic micropump systems, we tested these devices to evaluate their operational characteristics. Experiments were conducted to find the resonance frequency of the transducers and their active areas. These experiments found that a top electrical insulation layer was needed to prevent the working fluid from extending the active area of the top electrode. Consequently, a Parylene C layer was added to provide mechanical and electrical protection to the transducers. The thickness of the Parylene C layer was also carefully chosen so that it served as a quarter-wave acoustic impedance matching layer to water.
Micro-PIV experiments were performed in both the near and far fields of the ultrasonic transducer/pump. The near field experiments showed a very complex flow pattern. Flow directions changed as a function of the channel depth and steady vortices were formed. We attribute the formation of the vortices to the large cross-sectional area in the channel corner compared to the width in the straight-run regions, and compared to the effective diameter of the ultrasonic beam. Since the average flow rate in the corner is small, fluid in the vicinity of the transducer is easily drawn into the low pressure zone created near the transducer surface. This situation can be improved by using more aggressive corner compensation or a multi-step masking technique to produce perpendicular corners without any interior corner etching. From a different perspective, the chaotic flow in the near field can be utilized as an effective stirrer if the goal is to mix multiple fluid streams.

The far field experiments gave us estimates of the total pressure delivered by the micropumps. Unexpectedly, we observed very different performances from two different chips with the same nominal channel geometry: there was an order of magnitude difference between the flow rates provided by the two micropumps. This was possibly caused by the presence of a bubble in the first device that drastically increased the flow resistance of the channel. This could also explain the small fluctuations in the measurements due to the damping effects of a bubble. More experiments should be conducted to better understand the causes of the performance difference.

Overall, this work has met the design goal of building and testing a micropump using the principle of acoustic streaming and has laid the foundations for future research on this topic. The pumping system we developed is compatible with lithography-based microfabrication techniques and highly scalable. Initial estimates of the pump pressure are also much better than the ones reported by Rife et al [25] (39 Pa vs. 0.13 Pa).

6.2 Future Work and Improvements

There are many changes that could be explored on the current system design to seek improvements to its performance. In addition, this technology is sufficiently flexible that it
allows for integration into a much more complex microfluidic network in the future. In the following section, we will list some of the possible future developments for this line of research.

6.2.1 Short Term Tasks

An improvement that can be easily realized to the system design is the electrical impedance matching of the transducers. It is known that maximum power can be delivered to a load if the load impedance is the complex conjugate of the impedance in the voltage source [57]. Since the source impedance (for many standard commercial function generators or amplifiers) is usually purely real and has a value 50 $\Omega$, the load impedance should also be 50 $\Omega$. From figure 5.1, we can see that the transducers have a capacitive component to its electrical impedance. By connecting the transducer to a variable inductor, we can tune out the capacitance and increase the power delivered to the transducers. A Matlab calculation of the effects of impedance tuning is shown in figure 6.1.

![Load Power vs. Frequency](image)

Figure 6.1: Comparison of power delivery with and without inductor tuning
Another important short term task is to conduct more far-field velocity measurements to gain a more quantitative understanding of the performance variations among individual micropumps with the same nominal design. We suspect the large performance difference of the current experiments might be attributed to the presence of entrapped bubbles. Repeating the same experiment using a vacuum-degassed working fluid would eliminate this possible source of error.

More experiments can also be performed in the near field of the acoustic reflector to better understand the formation of the vortices. New microchannel designs with more aggressive corner compensation can be used to decrease the corner cross-sectional area. This should lead to a simpler flow pattern. One could also feed two different working fluids into the inlets of the T-corner design shown in figure 3.11 to test the mixing capabilities of the acoustic streaming micropump.

6.2.2 Long Term Initiatives

This work has developed a technological platform that can be custom-tailored to different applications. An attractive application of this technology is the ability to build fluid feedback devices. By closing the loop of the microchannels, the pump can run in a closed-circuit mode where the momentum delivered by the transducers is exactly balanced by the friction forces at the channel walls. Such a device would accommodate the necessary residence times of 1-10 minutes that are required for carrying out a variety of chemical reactions in a microfluidic format at a drastically reduced footprint. One example is microfluidic preparation of nanoparticles demonstrated by deMello et al. [58]

Another possible future development is the design of a highly complex fluid network with active flow control using a grid of transducers. By selective switching off and on the different transducers, the fluid can be controlled with high spatial and temporal resolution. Such a system would require a microprocessor with information feedback (from a flow sensor) to accurately assess the flow situation.
References


