Design and test of lead-zirconate-titanate flexural plate wave based actuators

Sriram Akella

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Design and Test of Lead-Zirconate-Titanate Flexural Plate Wave Based Actuators

by

Sriram Akella

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Electrical Engineering
Department of Electrical Engineering
College of Engineering
University of South Florida

Major Professor: Shekhar Bhansali, Ph.D.
Tom Weller, Ph.D.
John Bumgarner, Ph.D.
Scott Samson, Ph.D.

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Dedication
To my Parents
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ABSTRACT

Current MEMS development is driven by the need to develop various “Miniaturized Total Chemical Analysis Systems” (µTAS), biological and chemical sensing, drug delivery, molecular separation, microfiltration, amplification, and sequencing systems. In this work, the use of flexural plate wave devices as an actuator has been investigated.

This research was done with the aim of developing a platform to build FPW devices for use in System-On-Chip applications. It is well known that acoustic forces generated by a flexural plate wave (FPW) device can cause fluid motion, by the principle of acoustic streaming. Also the proven ability of FPW devices to cause mixing, filtration and to work as a chemical-biological sensor can be used towards building a micromachined µTAS.

The effects of the IDT finger width, spacing, aperture, membrane thickness, and driving conditions on the device performance was studied to understand the impact of IDT design on device performance. For this research bidirectional IDT’s were used, and at a later stage unidirectional IDT’s on one end and bidirectional IDT’s on the other end can be used for devices that need to function both as a pump and as a sensor.
The devices were tested under various conditions to understand the device performance. The devices were tested with and without a ground. The device performance was also studied when operated under the poling condition. Also the response of the device was tested before and after etching the membrane and a significant improvement in the output response was observed with the reduction in the transmission loss.

The FPW device performance was studied and it is observed that uniformly spaced IDT’s which have an acoustic path of at least 25 wavelengths long are required for good device performance. It is also observed that the devices need to be isolated from one another piezoelectrically for better device performance.
Chapter 1 Introduction

A revolution in understanding and utilizing micromechanical devices has started. The utility of these devices will be enormous, and with time these microdevices will fill the niches of our lives as pervasively as electronics. What form will these microdevices take? What will actuate them, and how will they interact with the environment? We cannot foresee where the developing technology will take us. The number of possible things we could try is beyond possibility [1].

Micromachining has numerous applications in microfluidics, and its use in this area has become even more important as people strive to create complete fluidic systems. A. Manz, in 1993, identified one of the possibilities as “Miniaturized Total Chemical Analysis System” (µTAS) [2]. Some of the applications include chemical analysis, biological and chemical sensing, drug delivery, molecular separation, microfiltration, amplification, sequencing and other applications. These systems are examples of the integration of flow channels, mixers, pumps, valves, sensors, etc. which from the building blocks for these “µTAS” systems. In this context, we present the work on design and testing of Flexural Plate Wave (FPW) devices for possible integration into fluidic systems for biological and chemical sensing, filters and mixers.

1.1 Acoustic Wave Devices

Acoustic wave devices are attractive for use in sensors because of the fact that the wave velocity and damping are sensitive to the surrounding parameters. Acoustic wave based delay line sensors have the following features: high sensitivity, very small in size,
have a high signal-to-noise ratio is an oscillator set-up. The acoustic wave devices, based on piezoelectric crystals, which allow transduction between electrical and acoustic energies, have been fabricated in a number of configurations the most commonly utilized for sensing application are:

1. Thickness Shear Mode (TSM) resonator
2. Surface Acoustic Wave (SAW) device
3. Acoustic Plate Mode (APM) device
4. Flexural Plate Wave (FPW) device.

Each of these devices are depicted in Figure 1.1

![Figure 1.1 Schematic sketches of the four types of Acoustic sensors [3]
(a) TSM resonator (b)SAW device (c)APM device (d)FPW device](image)

The waves in most cases are excited using an interdigital transducer (IDT), which consists of an interdigital metal pattern on a piezoelectric substrate. An illustration of an IDT electrode is shown in Figure 1.2.
In the Figure 1.2, “d”, determines the periodicity of the fingers and is equal to the wavelength of the excited wave. The frequency $f_0 = \nu / p$ is the resonant frequency of the device. A is the aperture and $d/4$ is the finger spacing.

1.2 Thickness Shear Mode (TSM) Resonator

The TSM resonator, also called as the quartz crystal microbalance (QCM), typically consists of a thin disc of AT-cut quartz with circular electrodes patterned on both side. Due to the piezoelectric properties of the quartz crystal, the application of a voltage between the electrodes results in a shear deformation of the crystal. The TSM resonators are generally used to measure metal deposition rates.

1.3 Surface Acoustic Wave (SAW) Device

Lord Rayleigh in 1887 discovered the surface acoustic wave mode of propagation in which the energy is confined very near to the surface of an isotropic solid. Rayleigh waves are useful in sensor application due to the surface confinement of the energy, making them sensitive to surface perturbations. The surface acoustic wave is most
conveniently excites on a piezoelectric material using an IDT pattern. An application of an alternating voltage between the alternately connected electrodes causes a periodic electric field to be imposed on the crystal generating a periodic strain field that produces a standing surface acoustic wave. The standing wave produces a propagating wave in both the directions with the wavefronts parallel to the transducer fingers. A cross-sectional view of the strain field generated by the wave propagating along the surface of the crystal is shown in Figure 1.3. The penetration depth of these waves is just over one wavelength. Typically these devices operate at around 100MHz.

Figure 1.3 Deformation filed due to the SAW propagating to the right along the solid surface [4]

1.4 Acoustic Plate Mode (APM) Devices

Acoustic plate mode devices utilize the shear-horizontal (SH) acoustic plate mode of vibration, which was developed for sensing in liquids. SH modes have particle displacements parallel to the device surface and normal to the direction of propagation as depicted in Figure 1.4. The absence of a surface-normal component of displacement allows each SH plate mode to propagate in contact with liquids without coupling excessive amounts of acoustic energy into the liquid. These devices utilize thin single-crystalline quartz plates that serve as acoustic waveguides, confining the acoustic energy between the upper and lower surfaces of the plate. Due to this the sensitivity of the SH-APM to mass loading and other perturbations depends on the thickness of the substrate
and both the faces of the crystal can be undergo displacements, hence detection can take place at both the surfaces.

![Figure 1.4 Schematic of the SH-APM device showing the shear horizontal displacement as it propagates between the input and output transducers [3]](image)

### 1.5 Flexural Plate Wave (FPW) Device

In a FPW device, the acoustic wave is excited in a thin membrane. The unique feature of the FPW device is that its phase velocity is lower than that of most liquids. When the FPW device is immersed in a liquid, a slow mode of propagation exists in which there is no radiation from the plate. The acoustic waves generated in a FPW are the first order symmetric and asymmetric lamb waves, illustrated in Figure 1.5
1.6 Organization

The following chapters describe the methods used in the development of the ultrasonic FPW device. Chapter 2 introduces the theory behind the system studied and the operation of the FPW device. Chapter 3 deals with the PZT thin films and their processing parameters. Chapter 4 describes the fabrication issues and process flow development. Chapter 5 presents experimental setup and results.
2.1 Active Fluid Pumps

Fluid flow in micro channels has very high flow impedance, i.e., pressure gradient per unit volume flux. This in a cylindrical channel varies as the inverse fourth power of the channel radius. In micro channels with radii in the order of micro meters, the hydrodynamic loads are enormous. In discrete pumping, there is one or more discrete pumping stations, each driving the hydrodynamic load associated with a channel length of the channel. However the number of pumping stations or the strength of these pumps must increase drastically to keep up with the drastically high flow impedances in micro-channels.

An alternative approach is to use distributed pumping as opposed to discrete pumping or the so called “Active pumps”. The fluid is driven all along the flow circuit via a body force [5]. Since each section of the channel is “active” it needs to drive only its own impedance; the channel itself acting as a pump. Such an active pump can be realized by a phenomenon known as Acoustic Streaming as shown by White et al [6]. The FPW device is an example of a device which causes fluid motion by the principle of acoustic streaming.

2.2 Flexural Plate Wave Device (FPW)

A typical FPW device consists of a piezoelectric film on a 1-µm thick silicon nitride membrane supported by a silicon frame. The piezoelectric layer is sandwiched
between a thin continuous bottom electrode and the interdigital transducers (IDT) top
electrode. In a FPW device, when a sinusoidal excitation is given to the IDT’s, they
generate an acoustic wave that propagates across the membrane. The IDT’s can either be
unidirectional (generate an acoustic wave traveling in one direction) or bidirectional
(generates an acoustic wave that travels in both the directions). Figure 2.1 and Figure 2.2
illustrate a FPW device with bidirectional and unidirectional IDT’s.

![Diagram of a FPW device with bidirectional IDT’s]

Figure 2.1 Flexural plate wave device with bidirectional IDT’s

Figure 2.2 shows a unidirectional IDT configuration, this device when excited by
two sinusoidal electrical drives operating in quadrature, where one signal lags the other
signal by 90° in phase, launch acoustic waves in only one direction, appropriate for FPW
device as an actuator. Figure 2.1 shows a sensing device with bidirectional IDT’s that
launch acoustic waves to the right and left across the membrane. Richard Moroey and
Chuck Bradley showed that FPW’s with unidirectional IDT’s are effective micropumps
[5, 6]. This pumping is caused by a phenomenon called acoustic streaming. Acoustic
streaming is a non linear effect that results in the time-averaged velocity field having a non-zero component parallel to the membrane.

![Diagram of Flexural Plate Wave Device](image)

**Figure 2.2 Flexural plate wave device with unidirectional IDT's**

A unique feature of the FPW device is that it can be designed such that its phase velocity is lower than that of most fluids. When the FPW is in contact or is immersed in a fluid, a slow mode of propagation exists in which there is no radiation from the plate. This makes the FPW device to function well in a liquid environment and can be used effectively for pumping as well as chemical and biological sensing in liquids.

### 2.3 Acoustic Streaming

The various mechanisms of fluid actuation are rotating cylinders, diaphragm motion, electrophoresis and other physical phenomena. One mechanism that received limited attention is the phenomena of acoustic streaming. Acoustic streaming is a steady fluid motion, which is created when high amplitude acoustic waves travel through a dissipative medium. Part of the energy lost through acoustic dissipation is imparted to the fluid as a steady momentum by means of nonlinear hydrodynamic coupling [7].
Acoustic streaming can be classified into three types depending on the scale of the fluid motion:

1. Eckart type: streaming generated in a non-uniform free sound field, whose scale is much larger than the acoustic wavelength.

2. Rayleigh type: vortex-like streaming generated outside the boundary in a standing wave field, whose scale is comparable to the wavelength.

3. Schlitching type: vortex motion generated in a viscous boundary layer on the surface of an object placed in a sound field, whose scale is much smaller than the wavelength.

Acoustic streaming offers two distinct advantages for applications in microfluidics [8].

1. Streaming helps to overcome the large viscous forces associated with microfluidic flow. This is because (a) Streaming is generated through acoustic attenuation, with very viscous losses that limit most of the microfluidic devices and (b) the streaming force is a body force and hence present wherever the acoustic field is present in significant amplitude and thus resulting in pumping throughout the fluidic network.

2. The streaming forces scale favorably as the diameter of the channels decrease. This is due to the generation of streaming in the acoustic boundary layer, resulting in very large forces over a small area.

In a FPW device, the flexural plate waves near the micromachined thin membrane excites an acoustic filed in the neighboring fluid and the acoustic filed in turn causes
streaming. This actuation mechanism is gentle yet effective means of transporting fluids through flow channels.

2.3.1 Flexural Plate Wave Driven Streaming

Determination of the source boundary condition is the first step towards applying the streaming theory to the FPW-driven streaming problem. The ultrasonically vibrating plate is the source of the acoustic energy; the source motion is that of the flexural waves traversing in the plate. The waves are viscous loaded zeroth-order antisymmetric Rayleigh-lamb mode waves. The streaming is primarily caused by the first-order acoustic field composed of acoustic and shear viscous modal fields. For fluid loaded FPW’s the acoustic field is evanescent, with a penetrating depth of ~ 20\(\mu\)m, and the viscous filed penetration depth is about 0.3\(\mu\)m.

![Figure 2.3 The forms of the acoustic, shear viscous and force fields near the plate surface [7]](image)

The force field is directed near along the direction of propagation of the wave, with several oscillations within ~3\(\mu\)m of the surface and an eventual exponential decay as shown in Figure 2.3

Forces that are generated due to the acoustic streaming can be classified into lateral forces (i.e., act in direction parallel to the device membrane) and vertical forces (i.e., act in a direction perpendicular to the device membrane) [9, 10, 11].

The lateral forces include the Strokes drag force and the force from the radiation pressure due to the traveling and standing wave. The vertical forces include the lift force,
2.4 Acoustic Wave Generation

The generation and detection of acoustic waves in a FPW is accomplished by the use of an IDT. The comb like structure of the IDT is depicted in Figure 3.4. The design of the IDT determines the electrical impedance of the device as well as the operating frequency, bandwidth, and sensitive area. The IDT excites an acoustic wave in the piezoelectric material (PZT) when a RF-voltage is applied to it. This applied voltage results in a synchronously varying deformation of the PZT substrate and the generation of an acoustic wave.

The wavelength $\lambda$ most effectively excited by the IDT is equal to the periodicity of the IDT pattern. The electrical impedance of the IDT depends on a variety of factors like the electromechanical coupling factor ($K^2$), the dielectric permittivity of the substrate ($\varepsilon_r$), and the geometry of the IDT: electrode width, electrode spacing, number of finger pairs, and acoustic aperture (IDT finger overlap length) [3].

For the low-loss and low-noise operation of the device it is important that its impedance be matched as closely as possible to the external components. The impedance of the IDT is largely capacitive due to its physical nature and this capacitance is be tuned out by placing an appropriately chosen inductor in series with the non-grounded comb of the IDT. The number of finger pairs in the IDT affects the bandwidth (BW) of the device: $BW \approx f_0/N$, where $f_0$ is the centre frequency and $N$ is the number of finger pairs. The generation of the wave for both bidirectional and unidirectional waves is explained below.
2.4.1 Bidirectional IDT

A bidirectional acoustic wave is generated by bidirectional IDT’s. In the standard bidirectional FPW device, two sets of transducer fingers are interdigitated and spaced so that the fingers are half a wavelength apart (center-to-center).

A single sinusoidal drive voltage is split by a power splitter, which produces two signals, “Signal I” and “Signal II”, which are $180^\circ$ out of phase with each other. Since the Ti/ TiN/ Pt ground electrode stack is always at a voltage half-way between the “Signal I” and “Signal II”, and “Signal I” and “Signal II” are symmetric about 0V, the ground plane will remain at 0V at all times. This is why devices with bidirectional IDT’s do not need an actual account to the ground. Though unnecessary in principle, the ground plane connection improves stability of the device in practice.

![Figure 2.4 Electrode layout for bidirectional IDT's](image)

The piezoelectric nature of PZT is used to create the periodic tension and compression in the upper half of the membrane. When the signal applied to an electrode is positive with respect to the ground plane, the PZT beneath it expands in a horizontal
direction. When the signal applied is negative with respect to ground plane, the PZT beneath the electrode contracts in a horizontal direction. This behavior is described by

\[ S_3 = d_{31} E_1 \]  

(2.1)

Where \( S_3 \) is the strain in the horizontal direction, \( d_{31} \) is the piezoelectric coefficient (m/V) and \( E_1 \) is the applied electric field (V/m).

Firstly, consider the rightward traveling displacements in a device operating at resonance in Figure 2.5. The compressions and tensions under the electrode propagate outward at a velocity determined by the dimensions, and the material properties of the membrane and any fluid in contact. The horizontal expansion that begins at time \( t_o \) under the left most “Signal I” electrode will propagate to the right so that at time \( t_2 \), it is under the neighboring “Signal II” electrode. When the device is at resonance, this expansion arrives just as the voltage at the “Signal II” electrode is inducing its own horizontal expansion, the net result being an even larger horizontal expansion. The same thing happens under the second “Signal I” electrode at time \( t_4 \). As this horizontal expansion passes under successive electrodes, the resultant expansion continues to grow in magnitude. For a horizontal contraction under “Signal II” electrode at time \( t_o \), a similar process takes place resulting in a growing magnitude of the horizontal contraction as it passes under successive electrodes.

The generation and growth of the leftward-traveling horizontal expansions and contractions occur in a similar way. The resulting sequence of tension and compression in the PZT layer (upper half of the membrane) causes periodic flexure, which propagates both to the right and to left.
An anti-symmetric traveling wave is generated when the PZT layer is approximately half of the thickness of the membrane. When the PZT layer is considerably thicker, when compared to the total membrane thickness, a bulk acoustic wave propagating in the ±x directions might be expected.

![Figure 2.5 Pictorial view of acoustic wave generation by bidirectional IDT's in a FPW device](image)

**2.4.2 Unidirectional IDT**

The working on a unidirectional IDT for unidirectional wave generation is explained as follows: each transducer finger pair is spaced one-quarter wavelength apart.
This transducer finger pair pattern is repeated at wavelength intervals to give the quadrature unidirectional interdigitated transducers, which is illustrated in Figure 2.6. A quadrature drive produces two sinusoidal signals, “Signal I” and “Signal II”, which are 90° out of phase with one another. For a device with unidirectional IDT’s a contact with the ground is necessary to hold it at 0V because “Signal I” and “Signal II” are not always symmetric about 0V. The floating fingers are used to improve device performance, also a periodic structure improves wave propagation and the floating electrodes help in keeping the wave fronts parallel.

Consider the rightward-traveling displacements in a device operating at resonance, in which “Signal I” leads “Signal II” by 90°. The horizontal expansion that begins at time \( t_0 \) under the left-most “Signal I” electrode will propagate to the right so that it is under the neighboring “Signal II” electrode at time \( t_1 \). This expansion arrives just as the voltage at the “Signal II” electrode is inducing horizontal expansion, the net result being a larger horizontal expansion. A similar thing will happen under the second “Signal I” electrode at time \( t_4 \). As this horizontal expansion passes under successive electrodes, the resulting...
expansion continues to grow in magnitude. A similar process takes place for a horizontal contraction under the left most “Signal I” electrode at time $t_2$, resulting in increasing the contraction magnitude as the contraction continues rightwards.

Now consider the leftward-traveling displacements in a device operating at resonance. The horizontal expansion that begins at $t_1$ under the left most “Signal II” electrode will propagate to the left so that at time $t_2$ it is under the neighboring “Signal I” electrode. This expansion arrives just as the voltage at the “Signal I” electrode is inducing its own horizontal contraction, resulting in the net horizontal displacements being cancelled out. Similarly, the leftward-traveling contractions and expansions are cancelled. The result is that no traveling wave is launched in a leftward direction. Figure 2.7 shows a snapshot of the tension and compression in the FPW membrane.
Figure 2.7 Pictorial view of acoustic wave generation by unidirectional IDT's in a FPW device
Chapter 3 Piezoelectricity and Lead-Zirconate-Titanate (PZT)

This chapter deals with the discussion of piezoelectricity and the deposition techniques, and the properties of PZT. The variation in the piezoelectric and ferroelectric properties of PZT with changes in different processing parameters like composition, annealing temperatures, poling, thickness of the samples etc., is discussed.

3.1 Piezoelectricity

The asymmetry in a unit cell resulting in the generation of electric dipoles due to mechanical distortion is the origin of piezoelectricity [12]. Piezoelectricity is defined as electric polarization produced by mechanical strains in crystals belonging to certain classes, the polarization being proportional to the strain and changing sign with it. This is the direct piezoelectric effect. The converse effect, a piezoelectric crystal becomes strained, when electrically polarized, by an amount proportional to the polarizing field.

3.2 Derivation of Piezoelectric Effect

The piezoelectric effect can be derived by considering the internal energy of a crystalline material at equilibrium described by the values of its state variables. For a piezoelectric material, the state variables are entropy $S$, temperature $T$, mechanical strain $\varepsilon$, mechanical stress $\sigma$, the electric polarization $P$, and the applied electric field $E$. Choosing the temperature, stress and the electric filed as independent variables, the thermodynamic potential is given by the Gibbs free energy:

$$ G = U - ST - \varepsilon \sigma - PE $$  \hspace{1cm} (3.1)
where the tension notation of $\varepsilon$, $\sigma$, $P$, $E$ is ignored and $U$ being the internal energy of the system. The differential of the above equation is:

$$dG = -SdT - \varepsilon d\sigma - PdE$$

(3.2)

Rearranging the above equation and holding the independent variables constant, we obtain the following relation:

$$-(\partial G / \partial T)_{\sigma,E} = S$$

(3.3)

$$-(\partial G / \partial E)_{T,\sigma} = P$$

(3.4)

$$-(\partial G / \partial \sigma)_{T,E} = \varepsilon$$

(3.5)

The system can also be described by expanding the energy function as a series of powers and products of the independent variables. Including only the lower order terms and neglecting the influence of temperature, the Gibbs free energy is given by:

$$G = -\frac{1}{2}S^E \sigma^2 - d\sigma E - \frac{1}{2} \chi^\sigma E$$

(3.6)

where $s$ is the elastic compliance at constant electric field, $d$ is the piezoelectric constant and $\chi$ is the dielectric susceptibility at constant stress [13].

Differentiating the above equation with respect to $E$ and combining with Equation 4.4 yields the direct piezoelectric effect:

$$-(\partial G / \partial E)_{\sigma} = P = d\sigma + \chi^E E$$

(3.7)

Differentiating Equation 3.6 with respect to mechanical stress $\sigma$ and combining with Equation 4.5 yields the converse piezoelectric effect:

$$-(\partial G / \partial \sigma)_{E} = \varepsilon = s^E \sigma + dE$$

(3.8)

We can easily verify that the piezoelectric constant $d$ is identical for both the direct and converse piezoelectric effects by comparing the second, mixed partial
derivative of the Gibbs free energy $G$ [14]. Differentiating Equation 3.7 with respect to mechanical stress $\sigma$ gives us:

$$\left(\frac{\partial^2 G}{\partial E \partial \sigma}\right) = \left(\frac{\partial P}{\partial \sigma}\right) = d$$  \hspace{1cm} (3.9)

Differentiating Equation 3.8 with respect to applied electric field $E$ gives us:

$$\left(\frac{\partial^2 G}{\partial \sigma \partial E}\right) = \left(\frac{\partial \varepsilon}{\partial E}\right) = d$$  \hspace{1cm} (3.10)

The piezoelectric constant is equivalent for both direct and converse effects since the order of differentiation is unimportant, $G$ being a state function $S$.

The notation used in this derivation is simplified and does not consider the tensor nature of the components. The electric field and polarization are actually first-order tensors and the stress and strain are second-order tensors and the piezoelectric constant is a third-order tensor. The direct and converse piezoelectric affects can be expressed in tensor notation as:

$$P_i = d_{ijk} \sigma_{jk} + \chi^\sigma \sigma_{ik} E_i$$ \hspace{1cm} (3.9)

$$\varepsilon_{jk} = s^E_{jkmn} \sigma_{mn} + d_{ijk} E_i$$ \hspace{1cm} (3.10)

The 27 independent elements of the piezoelectric tensor $d_{ijk}$ may be written as an array in the shape of a cube. The first suffix, $i$, designates the layer, while the second suffix, $j$, and third, $k$, denote the row and column of the element respectively. For example the $i^{th}$ layer can be written as a 3x3 matrix:

$$\begin{bmatrix}
  d_{i11} & d_{i12} & d_{i13} \\
  d_{i21} & d_{i22} & d_{i23} \\
  d_{i31} & d_{i32} & d_{i33}
\end{bmatrix}$$  \hspace{1cm} (3.11)

where, $i$ ranges from 1 to 3.
It is established that the mechanical stress and strain tensors $\sigma$ and $\varepsilon$ are symmetric, i.e., $\sigma_{jk} = \sigma_{kj}$ and $\varepsilon_{jk} = \varepsilon_{kj}$ [14]. It becomes necessary that the piezoelectric tensor is also symmetric with respect to $j$ and $k$ ($d_{ijk} = d_{ikj}$), reducing the number of independent elements from 27 to 18, enabling expression of the piezoelectric coefficients in the 3x6 matrix commonly used. In order to do this, the $j$ and $k$ suffixes are replaced by single suffixes in the following manner:

<table>
<thead>
<tr>
<th>Tensor Notation</th>
<th>11 22 33 23, 32 13, 31 12, 21</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix Notation</td>
<td>1 2 3 4 5 6</td>
</tr>
</tbody>
</table>

This representation reduces the matrix as:

$$
\begin{bmatrix}
  d_{i1} & \frac{1}{2}d_{i6} & \frac{1}{2}d_{i5} \\
  \frac{1}{2}d_{i6} & d_{i2} & \frac{1}{2}d_{i4} \\
  \frac{1}{2}d_{i5} & \frac{1}{2}d_{i4} & d_{i3}
\end{bmatrix}
$$

(3.12)

where the $\frac{1}{2}$’s are introduced to account for the multiplicity of the coefficients. Aligning the layers in rows and their elements in columns provides us with the simple compact 3x6 piezoelectric element matrix commonly used:

$$
\begin{bmatrix}
  d_{11} & d_{12} & d_{13} & d_{14} & d_{15} & d_{16} \\
  d_{21} & d_{22} & d_{23} & d_{24} & d_{25} & d_{26} \\
  d_{31} & d_{32} & d_{33} & d_{34} & d_{35} & d_{36}
\end{bmatrix}
$$

(4.13)

When the symmetry of a material’s crystal structure is considered, many of these coefficients are found to be simply related to zero. Even though it appears to be a second-order tensor, it is important to recall that, it is still a third-order tensor and transforms as such [14].

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The piezoelectric coefficients can be measured using the wafer flexure method; a wafer, which has the piezoelectric material deposited on it with the bottom and top electrodes defined, is placed in a special enclosure with a provision to apply pressure from one side of the wafer. When the pressure is applied, the strain produces in the film produces a potential across the film, which is measured. From the applied pressure and the generated electric potential, the piezoelectric coefficients can be calculated. In this research the piezoelectric coefficients could not be measured due to the lack of the fixture to make the measurements.

3.3 Poling

A crystal exhibiting spontaneous polarization can be visualized to be composed of negative and positive ions. In a certain temperature range, where the crystal has minimum free energy, these ions are at their equilibrium positions and the centre of the positive charge does not coincide with the centre of the negative charge. This can be visualized as an electric dipole, and the spontaneous polarization as dipole moment per unit volume, as due to an assembly of these dipoles, which point in the same direction.

Crystals, which develop an electric charge upon uniform heating, are labeled Pyroelectric. If the magnitude and direction of spontaneous polarization can be reversed by an external electric field, then such crystals are known as ferroelectrics.

Ferroelectric films possess regions with uniform polarization called ferroelectric domains. Within a domain, all the electric dipoles are aligned in the same direction. There may be many domains in a film separated by boundaries called domain walls. Adjacent domains can have their polarization vectors in anti-parallel directions or at right angles to one another. The boundaries between these domains are correspondingly known
as 180° or 90° domain walls. A single domain can be obtained by domain wall motion made possible by the application of a sufficiently high electric field, the process known as poling. Poling is very important for the application of polycrystalline ferroelectric films. Ferroelectric films do not possess any piezoelectric properties owing to the random orientations of the ferroelectric domains in the ceramics before poling. During poling, a DC electric field is applied on the ferroelectric ceramic sample to force the domains to be oriented or “poled”. While domains cannot be perfectly aligned with the field except when the grain or crystal is coincidentally oriented with its c- or a-axis in the field direction, their polarization vectors can be aligned to maximize the component resolved in the field direction. After poling, the electric field is removed and a remnant polarization and remnant strain are maintained in the sample, and the sample exhibits piezoelectricity. A simple illustration of the poling process is shown in Figure 3.1. However, a very strong field could lead to the reversal of the polarization in the domain, known as domain switching.

![Figure 3.1 Illustration of poling process of a piezoelectric material [15]](image)

### 3.4 Hysteresis Curve

A consequence of the resistance to domain switching is that polarization in a ferroelectric is hysteretic, which is another important characteristic of ferroelectrics; namely, that the polarization $P$ is a double-valued function of the applied electric field $E$,
and so is not precisely reversible with field. A ferroelectric hysteresis loop is shown in Figure 3.2. When a small electric field is applied, only a linear relationship between \( P \) and \( E \) is observed because the field is not large enough to switch any domain and the crystal will behave as a normal dielectric material (paraelectric). This case corresponds to the segment OA of the curves in Figure 3.2. As the electric strength increases, a number of negative domains, which have a polarization opposite to the direction of the field, will be switched over in the positive direction along the field, and domain orientation begins to take place. This results in a sharply rising \( P \) with increasing field \( E \), and the polarization will increase rapidly (segment AB) until all the domains are aligned in the positive direction (segment BC). This is a state of saturation in which the crystal is composed of just a single domain. As the field strength decreases, the polarization will generally decrease but does not return back to zero (at the point D in the Figure 3.2). When the field is reduced to zero, some of the domains will remain aligned in the positive direction and the film will exhibit a remnant polarization \( P_r \). The extrapolation of the linear segment BC of the curve back to the polarization axis (at the point E) represents the value of the spontaneous polarization \( P_s \). The remnant polarization \( P_r \) in a film cannot be removed until the applied field in the opposite direction reaches a certain value (at the point F). The strength of the field required to reduce the polarization \( P \) to zero is called the coercive field strength \( E_c \). Further, increasing the field in the negative direction will cause a complete alignment of the dipoles in this direction and the cycle can be completed by reversing the field direction once again.
3.5 Lead-Zirconate-Titanate (PZT)

Lead zirconate titanate (PZT) is a solid solution of lead titanate (PbTiO$_3$) and lead zirconate (PbZrO$_3$). It has the chemical composition of Pb(Zr$_x$Ti$_{1-x}$)O$_3$. PZT has a cubic perovskite crystal structure, shown in Figure 3.3(a). The piezoelectric and ferroelectric properties exhibited by PZT can be attributed to the distortion of the perovskite crystal structure. The unit cell is shown in Figure 3.3(b); in this unit cell the lead atoms (A cations) occupy the corners of the cube and Ti or Zr (B cations) occupy a position at the centre of the cube surrounded by an octahedron of oxygen anions.

![Figure 3.2 A typical P-E hysteresis loop in ferroelectrics [16]](image)

![Figure 3.3 Unit cell of PZT Perovskite [17]](image)
The excellent ferroelectric behavior of PZT is related to the mobility of the Ti/Zr (B cation) within the oxygen octahedron. There are three main distortions of the cubic perovskite crystal structure. For Ti rich compositions, having Ti concentration above 48% a ferroelectric tetragonal phase is preferred. The easy poling axis for this phase lies in the <100> family of directions. For Ti concentrations less than 48% a rhombohedral phase of the perovskite is preferred with an easy poling axis lying in the <111> family of directions. At a composition of 48% Ti and 52% Zr, a temperature independent phase (morphotropic phase boundary MPB) boundary occurs where the tetragonal and rhombohedral phases co-exist. It is in this phase that PZT exhibits the largest piezoelectric coefficients, due to the ability of the material to orient along one of the 14 easy axes of poling, 8 in the rhombohedral phase and 6 in the tetragonal phase. Increasing the zirconium concentration, an anti-ferroelectric orthorhombic phase develops. Figure 3.4 shows the phase diagram for PZT.

Figure 3.4 Phase diagram and of PZT [18]

From the diagram of the PZT solid solution system, the advantages of the system for using as piezoelectric films are evident:
1. Above the curie temperatures the symmetry is cubic, and the structure is perovskite.

2. The high curie temperatures across the whole diagram lead to stable ferroelectric states over wide, usable temperature ranges.

3. The Morphotropic phase boundary (MPB) separating rhombohedral (8 domain state) and tetragonal (6 domain state) ferroelectric domains is first-order, so there is necessarily a two phase region near this 52/48 Zr/Ti composition.

4. In the two phase region, the poling field may draw upon 14 orientation states leading to exceptional ceramic polability (Figure 3.4).

5. As the MPB is near vertical on the phase diagram, the intrinsic property enhancement in compositions chosen near to the boundary persists over a wide temperature range.

As the result of the unique structure of PZT, both its dielectric and piezoelectric properties show anomalous behavior near the MPB (Figure 3.4). There is a maximum in both the relative permittivity and the electromechanical coupling coefficient; the latter is an important parameter describing the piezoelectric properties. This feature makes PZT a practically useful piezoelectric material.

3.6 PZT Deposition Methods

The PZT thin films can be grown in different ways: sputtering deposition, sol-gel method, pulsed laser deposition, chemical vapor deposition, ion-beam sputtering, and screen-printing. The deposition methods are revised here under. Of all these sol-gel method is the most explored and widely used method in research.
3.6.1 Pulsed Laser Deposition (PLD)

Pulsed laser deposition, also called pulsed laser ablation, is a popular deposition method among researchers. It consists of material removal by bombarding the surface of the target with short energetic pulsed focused laser beam (laser ablation) this process takes place in a vacuum chamber where a gas (here oxygen) is held at constant pressure. Each pulse generally lasts a few tens of nanoseconds. The interaction at the surface of the target involves a sequence of events: surface melting, vaporization, multi photon emission, and the generation of plasma plume normal to the surface of the target. The components of the plasma plume are then collected on the substrate, forming the desired film.

Piezoelectric multi-component materials like PZT are very difficult to grow in the form of a thin film as small variations in the film stoichiometry results in the degradation of the piezoelectric and ferroelectric properties. Control over the process is required to avoid loss of volatile components (lead oxide PbO) and formation of metastable phases (pyclro-structure). PLD has some advantages with respect to other techniques. The most important being epitaxial thin films can be deposited at low substrate temperatures and with high deposition rates [19].

3.6.2 Chemical Vapor Deposition (CVD)

Chemical vapor deposition (CVD) is widely used in semiconductor manufacturing for depositing dielectric layers and metals on substrates. CVD consists of flowing gaseous precursors at elevated temperatures. The condition of the substrate surface is selected to promote a chemical reaction between the substrate and the various
precursors. The disadvantage being the use of toxic metal alkyl precursors used and the
time and effort required to properly characterize and optimize the deposition process.

3.6.3 Sputter Deposition
This technique is used widely to deposit piezoelectric thin films and has reached a
very high degree of maturity over the years. The advantages are strong adhesion to the
substrate, smooth surface, low deposition temperatures etc. The major disadvantage being
to the deposition of multi-oxide films like PZT is the control over the composition and
deposition rate.

There are several factors which affect the properties of the deposited film. They
are the power supplied, sputtering pressure, the type of gases used in the plasma,
temperature of the substrate etc. Sputtering using stoichiometric PZT composite ceramics
is expensive; the targets are prone to cracking during high power, and produced films
deficient in lead, the lead deficiency caused by the preferential resputtering of lead atoms
by the negatively charged oxygen ions. Another approach is the use of metallic targets in
a DC sputtering system which are independently powered. This method requires the
reactive sputtering in oxygen environment, or multiple metal-oxide targets can also be
used.

While the above deposition variations are primarily concerned with the altering
condition of the target and the plasma, the conditions at the substrate also have a
significant influence on the deposited films. Experiments have shown that the substrate
temperature and material have a large influence on the morphology of the deposited films.
Epitaxial films are most difficult to deposit and require a substrate material with similar
lattice parameters as PZT and substrate temperatures of above 550°C. Polycrystalline
films can be deposited at similar temperatures on any substrate. The use of certain under layers facilitates the growth in the preferential directions. A defect pyrochlore crystal structure is obtained if the films are deposited at temperatures lower than 550°C, and with no heating amorphous films are obtained.

The problem of lead loss at elevated temperatures during deposition is countered by depositing excess lead to compensate for the lead loss. Another strategy is to deposit the films at lower temperatures and annealing the deposited amorphous or pyrochlore films to nucleate the perovskite phase.

3.6.4 Sol-Gel Method

The sol-gel method is used for much of the research incorporating PZT layers due to its advantages over other techniques, such as control of composition over a large area, low initial processing temperature, high purity, and low cost. While the exact fabrication methods vary between different groups, the basic principle remains the same. Organic precursors containing the desired metals (generally lead acetate, zirconium-n-butoxide, and titanium-i-butoxide) are dissolved in specific quantities in a solvent (usually used solvents are methoxyxethanol, 2-proponol or isoproponol). These solutions are then carefully diluted to attain a specific viscosity for the application process. This solution is then filtered and used for film deposition by applying a spin-coated process. A few drops are deposited on an electrode deposited wafer and spun at 1000 – 4000 rpm. The resulting films are dried at 100°C - 150°C before another treatment at 300°C - 450°C in air to pyrolyze the remaining organic materials. A shrinkage process takes place, up to 50 – 70 % in the thickness direction: thus, for obtaining films with thickness appropriate for
applications, hundreds of nanometers up to microns, multistep deposition process is considered. The films are finally annealed at temperatures in the range of $600^\circ\text{C} – 750^\circ\text{C}$.

There are several advantages and disadvantages of depositing PZT films by this method. A major disadvantage is that the process of spinning on, pyrolyzing multiple layers for each substrate requires time and effort, making it generally unsuitable for large-scale production. The high-temperature annealing treatment is another major drawback of this technique. Finally, the precursors must be selected carefully to avoid toxicity and problems arise with residual stress in these films due to densification and out gassing during annealing.

3.7 Properties of PZT

3.7.1 Dielectric Constant and Loss Factor

The dielectric constant (also called the relative permittivity) $\varepsilon_r$, is defined as the ratio of the permittivity of the material to the permittivity of free space. The dielectric loss factor is defined as the tangent of the loss angle (tan $\delta$). It is the measure of the amount of electrical energy which is lost through conduction when a voltage is applied across the piezoelectric element.

3.7.2 Piezoelectric Coupling Coefficient

The piezoelectric coupling coefficient is defined as the ratio of the mechanical energy accumulated in response to an electrical input or vice-versa. It is also referred to the electromechanical coupling coefficient.

$$K^2 = \frac{\text{Mechanical energy converted to electrical energy}}{\text{Input mechanical energy}}$$
3.8 Influence of Structure and Composition on Physical Properties

3.8.1 Piezoelectric Properties

3.8.1.1. Influence of Orientation

The influence of orientation of the structure on the piezoelectric properties has been discussed in two fundamental papers [20, 21]. For tetragonal PZT the piezoelectric constant $d_{33[001]}$ orientation is larger than the piezoelectric constant $d_{33[111]}$ orientation. Figure 3.5 shows the piezoelectric constant $d_{33}$ in the [001] and [111] orientations for different compositions.

![Figure 3.5 Crystal orientation, and composition dependence of piezoelectric constant $d_{33}$ for PZT thin films near MPB [21, 22]](image)

However, the difference between $d_{33[001]}$ and $d_{33[111]}$ is much larger for rhombohedral PZT. It has been noted that the directions with maximum $d_{33}$ are very close to the perovskite [001] directions. Consequently, it is suggested that PZT epitaxial thin films used for sensors and actuators adopt rhombohedral compositions near the MPB with perovskite [001] orientation.

3.8.1.2 Influence of Composition

A systematic investigation of the relationship between composition and the piezoelectric constant $d_{33}$ in PZT thin films has been reported [21, 22]. It is observed that
for PZT thin films in the rhombohedral phase with varying Ti content the piezoelectric constant $d_{33}$ increase to a maximum value near the MPB. Similarly for PZT thin films in the tetragonal phase, the piezoelectric constant $d_{33}$ decreases from a maximum value at the MPB with increasing Ti content as depicted in Figure 3.5

3.8.1.3 Influence of Poling Field

In polycrystalline unoriented films the polarization under high DC electric fields is crucial because of the piezoelectric effect cannot be obtained in the absence of a polar axis. Poling of the films leads to substantial increase of their piezoelectric properties due to the dipole orientation during poling treatment. Poling at higher temperatures and for longer time also increases the piezoelectric properties. Significant improvements have also being seen with poling in a DC electric field increased in steps. The valves generally obtained are less than those of bulk ceramics, and this difference is attributed to the substrate constraints and to the restriction on the reorientation of the ferroelectric domains.

3.8.1.4 Influence of Film Thickness

It is generally observed that the increase in the thickness of the film results in higher piezoelectric coefficients. This is generally attributed to the changes in the residual stress with thickness and the presence of an interfacial layer at the electrodes whose influence on the effective properties is higher for thin films. However cracks in the film reduce their mechanical and electrical integrity.
3.8.2 Ferroelectric Properties

3.8.2.1 Influence of Composition

The ferroelectric properties show a strong dependence on composition of the PZT thin films. Thin films in the rhombohedral phase, with Ti content increasing to near the MPB, the remnant polarization values are high and low values for the coercive field, as compared to the tetragonal phase where with increasing Ti content, the remnant polarization increase with decreasing coercive field as illustrated in the Figure 3.6.

![Figure 3.6 Dependence of remnant polarization and coercive field on the composition of PZT thin films](image)

3.8.2.2 Thickness Dependence

Thickness dependence of ferroelectric properties in PZT thin films obtained by sol-gel method has being observed. The PZT films with higher thickness had better remnant polarization values compared to films with lower thickness. Also the coercive field value decrease with increasing thickness. These effects of the PZT thin films have been contributed to the greater contribution from the interfacial effects as the sample thickness decreases. As illustrated in Figure 3.7, the coercive field increases sharply at a thickness of about 1µm and the remnant polarization decreases gradually for films with thickness between 2µm and 1µm [24].

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3.8.3 Dielectric Properties

3.8.3.1 Influence of Orientation

The influence of orientation of the PZT films was shown in two papers [21, 22], the results depicted in Figure 3.8. For both the rhombohedral and tetragonal phases, the dielectric constant increases as the crystal tilting angle increasing from the spontaneous polarization direction. For rhombohedral phase the dielectric constant is maximum in the [001] direction and for films in the tetragonal phase this is much higher on the [111] direction than on the polar axis direction.

3.8.3.2 Influence of Composition

The maximum dielectric constant for PZT thin films is observed at the MPB for all orientations. The dependence has been experimentally verified in PZT films deposited
by RF-magnetron sputtering [23]. A strong dependence of composition has been obtained as depicted in Figure 3.8

3.8.3.3 Dependence on Film Thickness
Thickness dependence of dielectric properties has been found in different PZT films grown by different techniques. Kurchania and Milne [24] studied the thickness dependence on sol-gel prepared films with films of various thicknesses’ from 0.25µm to 10µm, the results plotted in Figure 3.9. The variation in the dielectric properties is due to the presence of growth stresses and thermal stresses, which increase with decreasing film thickness.

![Figure 3.9 Dependence of dielectric constant and dissipation factor on film thickness [24]](image)
Chapter 4 Fabrication and Process Flow Development

4.1 Background

The process flow development for the flexural plate wave devices presented here began with considerations of previously developed FPW fabrication methods.

FPW Fabrication: Figure 4.1 outlines the process flow previously developed for a basic FPW device for bi-directional IDT’s. Processing begins with RCA clean of high resistivity wafers (3000 ohm-cm) on which 5000Å of thermal oxide is grown. The oxide on one side of the wafer is tripped protecting the other side in Buffered Oxide Etch (BOE). The wafers are then cleaned and Spin-on-Boron is spun on the wafers which are pyrolyzed in the oven at 110°C for 20 minutes. Next, these wafers go through the high temperature boron-pre-deposition in process in the furnace to diffuse the boron. After this the wafers are deglazed in Hydro-Fluoric acid (HF) for two minutes, followed by a drive in step, a wet oxidation to grow 3000 Å of oxide. On the boron diffused side, titanium and platinum are evaporated (using AJA e-beam evaporator) and then annealed at 450-550°C in argon atmosphere. On these wafers sol-gel PZT is spun in a multistep process (to be explained in detail in another section) and the final PZT film with a thickness of ~1μm thick is annealed in oxygen atmosphere to improve the piezoelectric properties of PZT. On top of the PZT, gold is deposited with chromium used as the adhesion layer which is patterned to form the IDT electrodes. On the back side etch windows are opened lithographically and etched in BOE to etc the oxide and expose the silicon. The wafer is then places in a custom built etch jig and the Si etched from the back side in ethylene-
diamine-pyrocathecol (EDP) at 110°C for approximately 3 hours to get the FPW device with 2µm thick membranes.

**Figure 4.1 Process flow and schematic of a FPW device**

4.2 Issues in Fabrication of the FPW Devices

The various issues encountered and the resulting modifications in the process flow for the FPW device fabrication are outlined in the following sections

4.2.1 Poor Boron Etch Stop

The boron etch stop obtained by the above said diffusion process gave a very low yield in the membrane etch step, most of the membranes were etch through. The reason
for this being that the required boron concentrations of $1.0\times10^{19}$ boron atoms/cm$^2$ was not attained, Spin-on-boron being a limited source diffusion process. For this the diffusion process was changed and solid planar diffusion sources were used for the boron diffusion (Pre-dep) process. Also the time of the diffusion step was considerably increased from three hours to a seven hour process. After the diffusion process the wafers were deglazed and oxidised (drive-in step). After this step the wafers were found to have cracks on the top glass surface as shown in Figure 4.2.

![Figure 4.2 Picture of the cracks found after the drive-in process](image)

The reason for this was found out to be the formation of boric acid when the hot boron-rich glass interacts with water vapour. This reaction causes pitting on the surface as the boric acid evaporates [25]. To overcome this problem a dry oxide was grown at 700°C before the drive-in step using dry oxygen followed by a deglaze step in HF for 2 min. during this step the boron rich oxide film on the top was completely oxidising and the deglaze step completely removed the oxide and free of any surface defects. This two step boron diffusion step using planar diffusion sources resulted in good boron-etch stop layer, which resulted in a uniform smooth membranes for the FPW devices.
4.2.2 Silicon Nitride Membranes

Low stress silicon nitride was deposited on bare silicon wafers instead of the diffusion process. This change was bought about after the diffusion tube was changed and the same results were not obtained from the diffusion process as it had to be characterized for the new diffusion tube. The nitride is also a very good etch stop as the etch rate of silicon-nitride is very low in EDP. Low stress silicon nitride (1.5µm thick) was deposited followed by oxide deposition (0.5µm thick) at the clean room in Star Centre, Largo. The oxide was deposited to reduce the overall stress in the membrane stack. The membrane stack consisting of the nitride, the Ti/ Pt bottom electrode and the spun PZT thin film are all tensile in nature and hence the oxide was included in the stack due to its compressive nature. Consequently nitride and oxide of equal thickness was being tried out to offer better stress compensation to the membrane stack.

4.2.3 Bottom Electrode

4.2.3.1 Bottom Electrode/Adhesion Layer

PZT films have been deposited on a variety of substrates with variable results. Substrate selection is critical as single crystals of PZT will not grow on substrates with substantially different lattice parameters. Most MEMS and electronic applications require the PZT film to be deposited on an electrode layer. PZT films have been deposited on a wide variety of conductive films like RuO2, ITO (indium-tin oxide), TiN, Pt and Si. The bottom electrode is critical because PZT diffuses through the electrode layer inhibiting the formation of the perovskite phase, also the electrode layers get oxidised because of the repeated heat treatment the wafer has to undergo during the PZT deposition process.
Pt and RuO$_2$ electrode layers have been found to yield PZT films with the best piezoelectric properties [26].

Platinum was chosen as the bottom electrode layer because it does not get oxidised and remains conductive after the PZT deposition process. Platinum films can also be deposited with [111] preferential orientation, which is preferred because [111] planes of Pt and PZT have only 4% lattice mismatch, thus facilitating [111] oriented phase nucleation and growth.

Ti layer was used as the adhesion layer between the Pt bottom electrode and the oxide underneath because it acts as a good diffusion barrier. But the titanium was discovered to diffuse through the platinum electrode and forms a thin oxide layer. Various solutions were devised to address this problem, like oxidising the surface of the titanium layer before platinum deposition, use of TiN, TiO$_2$. TiN layer was selected as it effectively reduces the titanium diffusion through the platinum electrode reducing hillock formation.

For this research, a titanium, titanium-nitride, platinum stack as been used. The titanium film is sputtered followed by reactive sputtering of titanium-nitride in the same sputtering tool (AJA sputter tool in Star Centre, Largo) without breaking the vacuum. This was followed by sputtering of platinum. These wafers were than annealed in argon environment in the rapid thermal annealing system. Initially the films were annealed at 450 – 550°C, but later changed to a multi step anneal process where they were annealed at 700°C for 1 minute. This was done to prevent the peeling off occurring at the titanium layer during subsequent processing.
4.2.3.2 Stress in PZT/Bottom Electrode Stack

The stress changes within the bottom electrode stack was observed during the annealing step. It was found that sputtered Ti/TiN/Pt stack was initially stressed compressively due to the intrinsic stress in the deposited films. Upon annealing, the stress in the film increases. The stress increase in the system is attributed to thermal expansion differences between the platinum film and the silicon substrate. In the region between 400 – 500°C, the stress begins to decrease due to re-crystallization that removes the structural imperfections and nucleates the [200] oriented platinum grains in the film as evident from the XRD analysis shown in Figure 4.3. This is typically accompanied by stress release by the formation of hillocks at the surface of the film.

![XRD analysis of the platinum wafers annealed at different temperatures](image)

*Figure 4.3 XRD analysis of the platinum wafers annealed at different temperatures*

4.3 Sol-gel PZT Deposition

Sol-gel PZT is spun on the annealed platinum wafers in a multi-step process to get PZT films of varying thickness. The process flow diagram for spinning the PZT film is detailed in Figure 4.4.
Sol-gel PZT solution is spun on the annealed platinum wafers. The solution is spun in a two step process: 500rpm for 6 seconds followed by 2100rpm at 60 seconds. The films were then dried at 365°C for two minutes on a hot plate, placing them on the hot plate very slowly. This process of spinning results in PZT films approximately 0.1µm thick. This process is repeated 3 times followed by an anneal step in oxygen atmosphere for 1 minute to crystallize and nucleate the rhombohedral phase in the PZT film. The above process of spinning and annealing is repeated four times and results in film which is approximately 1µm thick. This multi-step process results in good films rather than a
single step process as the heat treatment results in enormous shrinkages in the film thickness direction.

![Diagram of PZT deposition process](image)

*Figure 4.5 Process flow for three step pyrolysis for PZT deposition*

The three step pyrolysis was also performed for spinning the PZT films. This was based on the fact that PZT undergoes three major volume shrinkages due to the solvent evaporation, light organic evaporation and decomposition of organics [27]. This endures complete organic removal and avoids cracking due to the above mentioned shrinkages.

Figures 4.6, 4.7, and 4.8 show the set-up for spinning PZT, the AFM-image of the PZT surface and the EDAX analysis of the PZT film deposited. The absence of platinum
in the analysis shows that the platinum bottom electrode has not diffused into the PZT layer, indicating that the bottom electrode to be intact after the repeated heat treatment (annealing process), indicating that TiN can be used as an effective barrier layer.

Figure 4.6 Setup for spinning PZT

Figure 4.7 AFM image of PZT film showing grain formation
4.4 Poling of the PZT Films

The PZT films were poled to improve their ferroelectric and piezoelectric properties. Different poling conditions were tested: poling at a constant DC bias, poling at a constant DC bias at higher temperatures, and poling in an electric field increased in steps. After which the hysteresis curves are plotted using the ferroelectric tester, Radiant Technologies RT66A.
Figure 4.10 Hysteresis curve obtained after the sample is poled [28]

Figure 4.11 SEM image of PZT film, showing good crystallization

Figure 4.9 shows the hysteresis curve for unpoled PZT samples and Figure 4.10 shows the hysteresis curve for the poled PZT sample. The remnant polarization of about
95\(\mu\text{C/cm}\) is obtained from the films. We also see a significant increase in the remnant polarization of the samples. Figure 4.11 shows the SEM image of the PZT film.

### 4.5 Modelling of FPW Device

The FPW device simulation was tried to understand the interaction between the IDT and the membrane. The solid model of the FPW was initially built in CoventorWare. Initial trials to mesh the solid model failed as the number of nodes of the meshed model was large and beyond the limitations of the system. The solid model is as shown in Figure 4.12. The model was then meshed using a different approach, using different mesh setting (each mesh element was defined depending upon individual layer geometry) for each individual layers and the use of ties and links to mechanically “tie” the layer together. The cross-sectional view of the meshed model is shown in Figure 4.13. This approach of meshing resolved the meshing issues.

![Figure 4.12 Cross section of the solid model of the FPW device built in CoventorWare](image)
Simulations on the meshed model were tried by applying a harmonic boundary condition on the IDT metal fingers, with harmonic analysis with frequencies of between 3.95MHz to 4.05MHz. Simulations of the first mode of vibration of the membrane were tried out with the above boundary conditions. These efforts were not productive as the program never converged to a solution.

The modelling of the device was also tried out using ANSYS; however the node limitations on the ANSYS University license did not result in a solution. Hence the mask design was developed based on the analytical model.
4.6 Mask Design

Eight designs were developed and used for testing the FPW device performance. The mask design used is illustrated in Figure 4.13 and the different parameters for each of the designs considered is tabulated in Table 4.1
Table 4.1 Various parameters listed for each of the eight designs used

<table>
<thead>
<tr>
<th>Design #</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
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</thead>
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<tr>
<td>Finger Width (µm)</td>
<td>20</td>
<td>40</td>
<td>40</td>
<td>40</td>
<td>40</td>
<td>20</td>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>Finger Spacing (µm)</td>
<td>20</td>
<td>20</td>
<td>40</td>
<td>20</td>
<td>40</td>
<td>20</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>Membrane Width (mm)</td>
<td>2.631</td>
<td>2.650</td>
<td>2.675</td>
<td>2.649</td>
<td>2.659</td>
<td>2.632</td>
<td>2.622</td>
<td>2.640</td>
</tr>
<tr>
<td>Membrane Length (mm)</td>
<td>7.724</td>
<td>8.107</td>
<td>7.744</td>
<td>9.712</td>
<td>10.687</td>
<td>7.540</td>
<td>9.460</td>
<td>8.850</td>
</tr>
<tr>
<td>Acoustic Distance (mm)</td>
<td>4.224</td>
<td>2.646</td>
<td>1.088</td>
<td>4.250</td>
<td>4.031</td>
<td>4.013</td>
<td>4.057</td>
<td>3.430</td>
</tr>
<tr>
<td>Resonant Frequency (MHz)</td>
<td>5</td>
<td>3.33</td>
<td>2.5</td>
<td>3.33</td>
<td>2.5</td>
<td>5</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Wavelength (µm)</td>
<td>80</td>
<td>120</td>
<td>160</td>
<td>120</td>
<td>160</td>
<td>80</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>
Chapter 5 Measurements and Results

The FPW devices fabricated were tested to find the transmission losses in the device. Two sets of measurements were performed on these devices: without a DC bias and with a DC bias applied to them. The effect of using a ground electrode was studied on the performance of the devices. Eight different designs were tested each with varying finger width, finger spacing, finger aperture, acoustic distance and different membrane dimensions. Each of the designs had 20 finger pairs both on the input side and output side.

5.1 Measurement Set-up

An arbitrary function generator was used to supply the power to the FPW device. The input signal from the function generator was split using a power splitter which splits the signals into two signals out of phase by 180°. The two signals from the power splitter were connected to the input IDT pair of the FPW using DC probes. The wafer was mounted on a probe station which had five DC probes. Two DC probes were used to actuate the device at the input end and two probes were used to sense the output voltage at the receiving IDT. The fifth probe was connected to the bottom platinum ground exposed in the corner of the wafer by PZT etching.

The input signal was a sinusoidal signal with a 0dBm input power level. The frequency was swept from 1MHz to 10 MHz and the power level from the receiving end was combined using a power combiner and fed to an Electronic Spectrum Analyzer.
(ESA). The schematic illustration is depicted in Figure 5.1, Figure 5.2 and Figure 5.3 shows the picture of the set-up with various components labelled.

Figure 5.1 Schematic of the measurement set-up

Figure 5.2 FPW characterization set-up

Figure 5.3 Picture of the FPW device under test (unshielded)
5.2 Results and Discussion

The ideal frequency response (insertion loss) from an FPW is as depicted in Figure 5.4 [29], showing the antisymmetric $A_0$ mode and the symmetric $S_0$ mode. The $A_0$ and $S_0$ modes are the only two modes that can be generated in plates having small thickness to-wavelength ratio.

The initial measurements were carried out by connecting Arbitrary Function Generators (AFG) in a phase lock loop so that both the signals are out of phase by $180^\circ$. The out-put signal from the out-put IDT pair was combined using a 0-180$^\circ$ power combiner and fed to an ESA. Due to the lack of automation software to the old ESA (HP8562A), the plots could not be exported to excel for analyzing the results. The results were plotted manually and one is shown in Figure 5.5. The plot in Figure 5.5 is not sufficient to understand the out-put characteristics of the FPW device and hence the set-up was slightly modified to automatically sweep the driving frequency from 1 MHz to 15 MHz to better understand the operation of the device. The signal from one of the AFG was fed to a power splitter and from the power splitter the signal was supplied to the input electrodes. The output was combined using a power combiner and supplied to the
ESA (Agilent E4411B). The obtained trace results were exported to a computer and plotted in Excel.

![Graph](image)

*Figure 5.5 Plot of transmission loss in dBm vs. driving frequency*

The effect of ground on the devices was tested by plotting the frequency response of the device, which were not etched. Effectively it will work as a Surface Acoustic Wave (SAW) device (thickness – wavelength ratio greater than 2). The effect of poling was also tested on the device performance. The devices were driven by applying a DC bias of 4.5V and the output plotted. The frequency plots for all the eight designs are plotted in Figures 5.6 to 5.13.
Figure 5.6 Plot of transmission loss in dBm vs. driving frequency for FPW design #1

Figure 5.7 Plot of transmission loss in dBm vs. driving frequency for FPW design #2
Figure 5.8 Plot of transmission loss in dBm vs. driving frequency for FPW design #3

Figure 5.9 Plot of transmission loss in dBm vs. driving frequency for FPW design #4
Figure 5.10 Plot of transmission loss in dBm vs. driving frequency for FPW design #5

Figure 5.11 Plot of transmission loss in dBm vs. driving frequency for FPW design #6
Figure 5.12 Plot of transmission loss in dBm vs. driving frequency for FPW design # 7

Figure 5.13 Plot of transmission loss in dBm vs. driving frequency for FPW design # 8
Eight designs were considered for the FPW device. Four of them were designed to have the same resonant frequencies with varying acoustic path, i.e., the distance between the IDT pairs.

![Graph showing transmission loss in dBm vs. driving frequency of design 2 and 4](image)

*Figure 5.14 Plot of transmission loss in dBm vs. driving frequency of design 2 and 4*

The influence of varying the acoustic path on the device performance is compared in Figure 5.14. It is observed that frequency response is observed when the acoustic path is an integral multiple of the wavelength; design two has a better response compared to design four. In addition, it is observed that the effect of ground stabilizes the response.
From Figure 5.15, it is noted that a minimum acoustic distance of 25 wavelengths is required for the FPW device; design 3 has an acoustic path that is equal to 6.8 wavelengths compared to 25.2 wavelengths for design 5.

It is also observed that the characteristic peak at the $A_0$ mode resonant frequency cannot be seen as expected. The possible reason for this is that when the FPW device under test is excited, some of the energy travels along the PZT layer to other devices on the substrates and this causes multiple reflections. The result obtained is that at the resonant frequency of each device, the reflected power will be the maximum and hence the obtained frequency plot looks like a “table” between the frequencies 2MHz to 5MHz rather than having a single peak value at the device resonant frequency.
5.3 Modified Mask Design

From the initial measurements performed on the devices two issues were found. One was the interference from the other devices in the form of reflections and secondly the mask design did not facilitate the use of RF probes to be used for calculating the S-parameters using a network analyzer. To address these two issues the masks have been redesigned with provision for the use of RF probes and also the isolation of the devices from one another by adding an etch step to etch the PZT layer in between the devices to isolate them piezoelectrically. The issue of dicing the individual devices can also be resolved by etching deep v-groves in between the devices to facilitate easy cleavage of the wafer.
Figure 5.16 Plot of the redesigned mask set isolating each device
Chapter 6 Conclusions and Future Work

The intent of this research was to develop a flexural plate wave device for use as an actuator for microfluidic applications like pumping, mixing and filtrations. The effect of various parameters in the IDT design have been studied, primarily focusing on bidirectional IDT’s. PZT sol-gel deposition process was developed and working FPW devices have been fabricated. The obtained results are summarized.

6.1 FPW Fabrication

The process flow for both depositing sol-gel PZT and the FPW device fabrication has been optimized to increase the yield. The process flow for the fabrication of the FPW device is outlined in appendix A. low stress silicon-nitride was used successfully for defining the membrane. The variation in the stress of the film during the fabrication has been minimized by the use of TiN barrier layer, a thin oxide layer to compensate the tensile stresses in the membrane and the use of different annealing conditions.

6.2 FPW Design

The bidirectional IDT electrode design has been used, to facilitate the study of the device as a sensor and a microfluidic pump at a later stage. The device performance under both a DC bias and under no bias has been measured. The influence of the ground electrode on the device performance was studied.

The FPW device modeling and simulations were carried out in CoventorWare 2004, and ANSYS 8.0. These efforts were not productive, as the programs never
converged to a solution no significant result could be obtained due to hardware and software limitations.

6.3 Future Work

The reflections from other devices on the same substrate have to be eliminated to obtain the true characteristics of each device. This can be obtained, by etching the PZT in between the FPW devices so that they are all disjoint. This can be achieved by redesigning a mask to etch PZT before the IDT’s are defined. One more problem faced during the testing was the scratching of the contact pads by the probe tips. To prevent this, the contact pads have to be made thicker to facilitate repeated testing of the devices. This has to be achieved without increasing the IDT finger thickness because doing so will load the membrane mechanically.

The device characteristics as a micropump and a sensor have to be studied. The unidirectional IDT transducer design has to be studied for use in microfluidic applications; this facilitates the use of the FPW device for filtering and mixing applications for \( \mu \)TAS applications. The FPW device-modeling needs to be carried out using other software’s like CFD, Kappa, FEMLAB or the code can be developed in MATLAB.
References


Appendices
Appendix A: Process Flow for FPW Device Fabrication

**Wafer Type**
N (100) Si, Double sided Polished, Resistivity: 3000-5000 ohm cm, Thickness: 265-285µm.

A. Step 1. RCA cleans
   1. RCA Clean (SC1 and SC2)

B. LPCVD Nitride deposition
   1. Deposit 2µm thick low-stress LPCVD nitride. (at Star Centre, Largo)
      (Used as a Membrane material and etch-stop)

C. CVD Oxide deposition
   1. Oxide deposition of 0.5µm thick (at Star Centre, Largo)

D. Bottom electrode deposition (at Star Centre Largo)
   1. Sputter Ti/TiN in AJA sputtering tool. Thickness 800Å/100Å
      2. Sputter Pt 2000Å thick

E. Pt layer annealing
   1. Rapid thermal annealing of the wafer in Ar environment.
      2. 50°C/sec ramp rate to 450°C for 1 min.
      3. 50°C/sec ramp rate to 750°C for 1 min.

F. Sol-gel PZT deposition
   1. Spin PZT sol-gel
      o Step 1: 6 sec, 500rpm
      o Step 2: 60 sec, 2100rpm.
   2. Pyrolysis hot plate 360°C, 2 min.
   3. Spin PZT sol-gel
      o Step 1: 3 sec, 500rpm
      o Step 2: 60 sec, 2000rpm.
   4. Pyrolysis hot plate 365°C, 2 min.
   5. Spin PZT sol-gel
      o Step 1: 3 sec, 500rpm
      o Step 2: 60 sec, 2000rpm.
   6. Pyrolysis hot plate 365°C, 2 min.
   7. Rapid thermal Annealing 700°C, O₂ atmosphere
      o Ramp at 50°C/sec to 450°C for 1 min.
      o Ramp at 50°C/sec to 600°C for 1 min.
      o Ramp at 50°C/sec to 700°C for 1 min.
   8. Repeat the above process for 4 runs

G. Litho for back etch window (Mask 1)
   1. Spin 3000PY on front side, 3000RPM, 60sec
Appendix A (Continued)

2. Soft Bake Hot Plate 155°C, 60secs
3. Expose 17secs (bright field mask)
4. Hard Bake Hot Plate 110°C, 60secs
5. Develop RD-6, 25secs
6. Inspect

H. Opening of the etch window.
   1. Spin AZ4620 on front side, 3000RPM, 60sec, to protect the PZT from BOE
   2. Oxide etch in 10:1 BOE for 15 min.
   3. RIE in Plasma therm at clean room in USF, Tampa
      ○ 4 steps of 10 min. etches

I. Metallization of the IDT’s and the bond pads
   1. Deposit Cr/Au 200Å/1500Å thick in the e-beam evaporator (Thin films lab ENB110).

J. IDT Mask (Front Side Alignment) Using EVG mask aligner
   1. Spin 3000PY on front side, 3000RPM, 30sec
   2. Soft Bake Hot Plate 155°C, 60secs
   3. Expose 17secs (Dark Field Mask)
   4. Hard Bake Hot Plate 110°C, 60secs
   5. Develop RD-6, 25secs
   6. Etch back Au and Cu
   7. Inspect

K. PZT Poling
   1. Etch PZT near the wafer flat using BOE until Pt surface is seen
   2. Clean the wafer with D.I. water and blow dry.
   3. Poling Voltage 15V, 30 mins using DC probes
   4. Inspect Using RT66A for Hysterisis Curve

L. Back Side Etching for Membrane
   1. EDP etching at 110 °C, 3.5 hrs. Avg. etch rate ~ 75µ/Hr. (using etch jig)
   2. Etch 2µm Membrane

M. Clean the wafer and Drying
   1. The wafer is thoroughly cleaned in Isoproponal and D.I. Water.
   2. The wafer is dried in an oven at 110°C, as blow drying the 2µ membranes is not advisable.
Appendix B: Membrane Etching (Using EDP)

B.1 EDP Mixture Preparation

<table>
<thead>
<tr>
<th></th>
<th>Single</th>
<th>Double</th>
<th>Triple</th>
</tr>
</thead>
<tbody>
<tr>
<td>DI water</td>
<td>48 ml</td>
<td>96 ml</td>
<td>144 ml</td>
</tr>
<tr>
<td>Catechol</td>
<td>48 g</td>
<td>96 g</td>
<td>144 g</td>
</tr>
<tr>
<td>Pyrazine</td>
<td>0.9 g</td>
<td>1.8 g</td>
<td>2.7 g</td>
</tr>
<tr>
<td>Ethylenediamine</td>
<td>150 ml</td>
<td>300 ml</td>
<td>450 ml</td>
</tr>
</tbody>
</table>

1. Obtain an EDP beaker, a graduated cylinder labeled “ethylenediamine”, the scale, a large plastic weigh boat, a small plastic weigh boat, and a piece of aluminum foil to cover the beaker.

2. Place an EDP waste bottle in the sink. Make sure that the hotplate is covered with two layers of clean aluminum foil.

3. Measure the DI water in the graduated cylinder and add it to the EDP beaker.

4. Weigh out the catechol in the large plastic weigh boat: add it to the beaker.
   Discard the weigh boat in the small EDP trash can. Wipe off the lip and outside of the catechol bottle before returning it to the cabinet.

5. Weigh out the pyrazine in the small plastic weigh boat: add it to the beaker.
   Discard the weigh boat in the small EDP trash can. Wipe down the outside of the pyrazine bottle before returning it to the cabinet. Wipe down the scale before returning it to the shelf.

6. Place the large graduated cylinder in the sink. Measure out the ethylenediamine and pour it in the beaker. Immediately cover the beaker with aluminum foil. Rinse the graduated cylinder with DI water: pour the water from the first two rinses into...
Appendix B (Continued)

the EDP waste bottle. Rinse the graduated cylinder three more times before returning it to the shelf. Wipe down the outside of the ethylenediamine bottle before returning it to the cabinet.

7. Place the beaker on the hotplate and insert the temperature probe through the tinfoil. Make sure that the probe is immersed but does not touch the sides or the bottom of the beaker. Set the probe temperature to 110°C. Do not allow EDP to boil.

8. After the EDP has reached the correct temperature, place the wafer to be etched in the EDP beaker. The etch rate is about 75µm per hour.

9. Post a notice with your name, date and time, and a phone number where you can be reached on the door of the hood.

B.2 Waste Handling

B.2.1 Solid Waste
All solid trash generated during an EDP etch should be deposited in the small EDP trash can. This includes paper towels, weigh boats, plastic scoopers, gloves etc.

B.2.2 Liquid Waste
EDP and/or ethylenediamine may not be poured down the drain. EDP must be disposed of in properly labeled hazardous waste bottles. Never tightly cap a bottle containing hot EDP waste or “it may explode”. Loosely place the cap on the bottle and leave it at the back of the hood until it is sufficiently cool. Then tighten the cap, wipe down the outside of the bottle, and put it in the waste chemical cabinet.
Appendix B (contd.)

B.3 Clean-up
1. When etching is finished, turn off the hot plate. Remove the temperature probe, wrap it in a wet paper towel, and set it aside.

2. Carefully pour off the EDP into the waste bottle using the funnel. Waste water from the first two rinses should also be poured into the waste bottle. Then rinse the beaker at least three more times and use a wet towel to scrub off any dried EDP residue. Scrub the inside and the outside of the beaker. Thoroughly rinse the funnel and any teflonware.

3. When rinsing your sample, as long as the water continues to turn blue-black it should be poured into the waste bottle. When it is clear it may be poured down the drain.

4. Scrub the plastic shields, including the space between the metal frame and the plastic.

5. Scrub all the surfaces of the hood with wet paper towels. Continue to clean until the paper towels no longer turn yellow.

6. When the hotplate is cool, remove and discard the tinfoil. Carefully clean the temperature probe and place them in the shelf.

B.4 Required Safety
The following items of apparel are required when doing EDP etch; long pants and shoes.

The following safety is also required: lab coat, PVC gloves, acid apron, acid gloves, safety glasses, and a face shield. It is recommended that users obtain a respirator for this procedure.