

# The Design of a FPW Resonator Using the Composite Membrane of PZT Layer and SiN<sub>x</sub> for Liquid Sensing

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## Abstract

A Flexure Plate Wave (FPW) resonator using piezoelectric thin film is proposed for liquid sensing. FPW device is suitable for liquid sensing because of less scattering of the energy from the acoustic wave to the loading liquid due to its low phase velocity. The COM theory is used to simulate the response of a two-port SAW resonator, which is extended to the design of the FPW resonator. The possible applications and the constraints for the proposed device are discussed from the theoretical expression. The comparison between the mass effect and the stiffness effect for the cases of droplet and fill loading show the application strategy in liquid sensing. The sol-gel derived lead zirconate titanates (PZT) thin film is applied on the proposed device because of the cost advantage and the high electromechanical coupling effect over other thin piezoelectric thin films. The preliminary measurement results show that the resonant frequency and the relative liquid density have a good linear correlation, and demonstrate the feasibility of the proposed device.

**Key Words:** FPW Resonator, Acoustic Sensor, Piezoelectric Film, PZT, Sol-Gel, Liquid Sensing

## 1. Introduction

Lamb wave [1] are associated with Rayleigh waves on a thin plate whose thickness is smaller than the wavelength. They can be considered as two Rayleigh waves propagating on both sides of a thin plate. Two kinds of waves can propagate through the plate independently, namely the symmetric and the antisymmetric waves. The  $A_0$  wave is the antisymmetric lamb wave with the lowest phase velocity. The low operating frequency is an attractive feature as it implies relatively inexpensive associated electronic circuit. This feature also makes the wave suitable for liquid sensing because the  $A_0$  wave does not excite compressional waves in a loading liquid and thus reduces scattering energy if its phase velocity is lower than the sound velocity of liquid.

The  $A_0$  mode lamb wave sensor is also called the Flexural Plate Wave (FPW) sensor. The excitation and the detection of acoustic waves are most readily accomplished by the use of interdigital transducers (IDTs) [2] on piezoelectric substrates. Acoustic wave sensors used to be realized on a bulk piezoelectric substrate. Piezo-

electric thin films, such as lead zirconium titanate (PZT), zinc oxide (ZnO), and aluminum nitride (AlN), have the cost advantage over crystalline materials. Among them, the electromechanical coupling effect ( $K^2$ ) of PZT is three to nine times and the dielectric constant 100 times over AlN and ZnO, which makes PZT potentially suitable for thin film acoustic sensors. However, the polycrystalline structure of PZT and the required heat treatment during the coating process complicate the realization.

The applications of lamb wave sensors to chemical and liquid sensing have raised a lot of research interests. Costello et al. [3] proposed a simple theory for the mass sensitivity of a delay-line oscillator with ZnO on silicon nitride membrane, and modeled the attenuation of plate waves in contact with viscous liquids. Laurent et al. [4] addressed the configuration design of the FPW devices using AlN and ZnO on silicon membrane, and showed that the FPW device has a large mass sensitivity compared to other acoustic devices. Weinberg et al. [5] derived the fluid-damping model for resonant FPW devices. To increase the differentiability of the resonant frequency shift, reflecting gratings are added to the lamb wave device that is first reported by Joshi [6].

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This study will discuss the design and application issues for the FPW resonator using the structure of PZT films on a silicon nitride membrane. We will derive the design of reflecting grates using the COM (Coupling of Modes) theory. The effects of the density and the viscosity of the liquid in contact on the deviation of the resonant frequency are determined to illustrate the constraints of the proposed device in liquid sensing. Finally, the experimental result is compared with the theoretical estimates.

## 2. Fabrication Procedure

The materials system of the FPW device is assumed Pt/Ti/PZT/LSMO/SiN<sub>x</sub>. The (La<sub>x</sub>Sr<sub>1-x</sub>)MnO<sub>3</sub>, LSMO, is used as a buffer layer between PZT and SiN<sub>x</sub> to enhance the piezoelectric characteristic and to avoid possible cracking of PZT. The LSMO and the PZT thin films are multi-coated by sol-gel techniques. Furnace heating of 650 °C is used to transform PZT films into polycrystalline structure. The electrodes of the IDT are patterned for the period of 40 (μm) using lift-off. Finally, the membrane cavity of the device is fabricated using KOH anisotropic etching (30 °C, 80%). The final composite membrane is consisted of 1.2 (μm) silicon nitride and PZT layer of 1.1 (μm). The size of the rectangular membrane is about 4.2 × 2.7 (mm).

## 3. Design of Reflectors

The design of the reflection gratings is derived from a two-port SAW resonator over a bulk PZT modeled using the COM theory [8]. There are three basic elements in the model: IDT, spacing, and reflector that can be described by three complex transmission matrices of [T], [D], and [G] respectively. Matrix [T] is a 3 × 3 transmission matrix for the IDTs, including both the acoustic and the electric parameters. Matrix [D] is a 2 × 2 matrix for the acoustic transmission line between IDTs and gratings. Matrix [G] is a 2 × 2 matrix for the SAW reflection gratings to describe the relationship between the acoustic transmission and reflection response.

The reflection phase,  $\theta$ , is determined by the position of standing wave at the reference plane relating the sign of reflected-to-incident surface waves entering the reflection

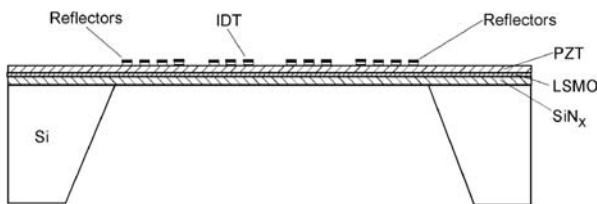


Figure 1. Schematic of FPW resonators.

gratings, which will affect the spacing design between the gratings and adjacent IDTs ( $D_2$  and  $D_6$ ). This determination of reflecting phase is difficult and often depends on the experimental for the material combination of the piezoelectric substrate and the reflecting electrodes [8]. The reflection coefficient for the PZT thin film and Pt/Ti reflection gratings is not available. However, since PZT is a strong piezoelectric, like lithium niobate, the reference phase  $\theta = 0^\circ$  is assumed for the open circuit design of reflector. The device parameters can be determined from the tradeoff between performance and device size:

- (1) The number of reflection grating is set 40 that are to form standing waves to reduce insertion loss.
- (2) The more pairs of IDT, the smaller insertion loss and the wider bandwidth. Twenty pairs of IDT are selected.
- (3) The overlap length of IDT is  $50\lambda$  for a low insertion loss and a high transmission effect, where  $\lambda$  is the acoustic wavelength.
- (4) The separation between the IDTs is  $10\lambda$ . The spacing between the gratings and adjacent IDTs ( $D_2$ ) are  $(1/8 + n/2)\lambda$  to produce a sharp resonant peak (Figure 3).

The derived reflector design will be applied to the FPW device to increase the differentiability of frequency deviation due to liquid loading.

## 4. The Sensing Mechanism of FPW Devices

The phase velocity of the  $A_0$  mode FPW propagating on a thin plate is related to the bending stiffness,  $B$ , and the mass per unit area of a homogeneous isotropic plate,  $M$ . When the device is in contact with liquid, it will introduce additional stiffness effect due to liquid weight and additional mass effect due to the agitation of the liquid. The phase velocity of the plate regime subject to a tensile stress and liquid loading can be well approximated by the simple asymptotic expression [7]:

$$v_p = \left( \frac{T_x + B}{M + \rho_F \delta_E + M_\eta} \right)^{1/2} \quad (1)$$

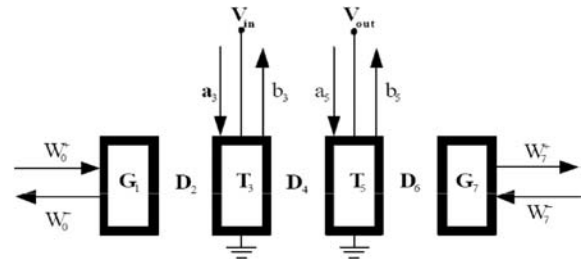
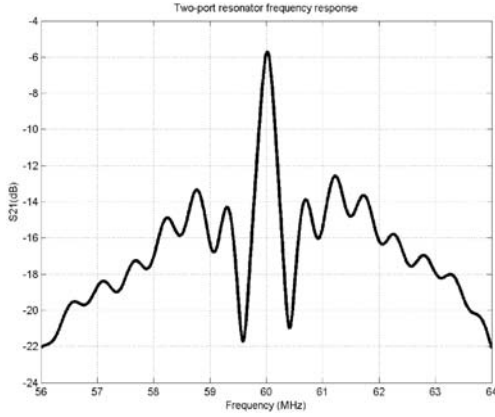


Figure 2. Representation of two-port resonator building blocks.



**Figure 3.** Two-port SAW resonator frequency response.

where  $T_x$  is the component of in-plane tension in the  $x$  direction,  
 $\rho_F \delta_E$  is the mass effect,  
 $\rho_F$  is the density of the fluid, and  
 $M_\eta$  is the viscosity effect.

$$\delta_E \approx \left( \frac{\lambda}{2\pi} \right) \quad (2)$$

if the phase velocity is much less than the speed of sound in the contact liquid.

$$M_\eta = \frac{\rho_F \delta_V}{2} \quad (3)$$

where  $\delta_V = \left( \frac{2\eta}{\omega \rho_F} \right)^{1/2}$  is the viscous decay length,

$\omega$  is the operating angular frequency, and  
 $\eta$  is the shear viscosity.

#### 4.1 The Loading Effects of Low Viscosity Liquids

When the FPW device is in contact with a low viscosity liquid, the viscosity effect of Eq. (3) is negligible compared with the mass effect, and the phase velocity will be influenced by the liquid density and the evanescent decay length in the mass effect ( $\rho_F \delta_E$ ). If the loading liquid is a small droplet on the thin plate and the contacting surface is not hydrophilic, the droplet will not spread out evenly and remain hemispherical as shown in Figure 4. Because the evanescent decay length is approximate  $\lambda/2\pi$ , not all the mass of the loading drop contributes the mass loading effect. The shape of the drop can't be accurately controlled; therefore, the change of the phase velocity usually will not be in proportional to the number of liquid droplets.

The liquid is suggested to fill up the cavity of the sensing region if the device is used to detect the liquid

density. When the liquid is thicker than the evanescent decay length, the mass-loading effect of the liquid will remain the same. The liquid weight will introduce the tensile stress in the membrane that will result in the deviation of phase velocity. The mass sensitivity and tension sensitivity of the perturbation of phase velocity are as follows:

$$\frac{\Delta v_p}{v_p} = s_m \times \rho_F + s_T \times T_x \quad (4)$$

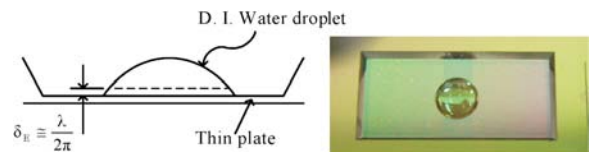
$$\text{Where } s_m = -\frac{\delta_E}{2(M + \rho_F \delta_E)}, \quad s_T = \frac{1}{2(T_x + B)}$$

The calculated phase velocity for the composite membrane in air is about  $235 \text{ (m}\cdot\text{s}^{-1})$  which is much smaller than the speed of sound in water,  $1482 \text{ (m}\cdot\text{s}^{-1})$ , to ensure less energy dissipation to the liquid in contact. The stiffness of the membrane is estimated using the composite plate theory. The calculated sensitivities in our case are  $s_m = -2.78 \text{ (m}^2\text{/N)}$  and  $s_T = 7.69 \times 10^{-5} \text{ (m/N)}$ .

Assuming 5 (mg) of water is loaded on the cavity, we can estimate the average tensile stress along the wave propagating direction from the finite element analysis. The acoustic wavelength is  $40 \text{ (}\mu\text{m)}$ . The estimated frequency deviation due to the mass loading of water is about  $-0.94 \text{ (MHz)}$  and the frequency deviation due to the tensile effect is only  $+1.24 \text{ (KHz)}$ . The tension effect due to the liquid pressure can be ignored when compared with the mass loading effect. Therefore, when the device cavity is filled up with different liquids, the change of the phase velocity will be related to the density of the liquid and thus can be used as a density sensor for low viscosity liquid.

#### 4.2 The Loading Effects for High Viscosity Liquids

From Eq. (1), we notice that the phase velocity of FPW will be influenced by fluid density ( $\rho_F$ ) and shear viscosity ( $\eta$ ) of the loading liquid. However, the respective velocity perturbations can't be differentiated because density and viscosity are coupled in the viscosity effect in Eq. (3). In another word, the liquid viscosity and density of high viscosity liquids can't be determined by the frequency deviation of the device.



**Figure 4.** The liquid droplet on the FPW device.

## 5. Experimental Results and Discussions

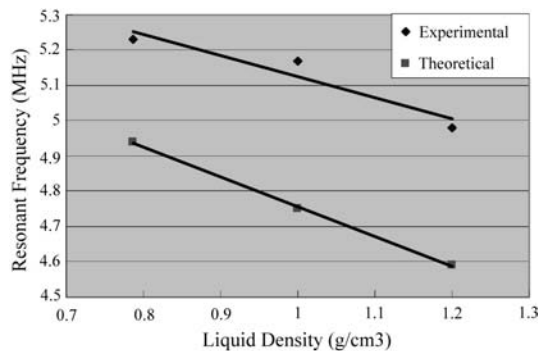
### 5.1 Density Sensing of Low Viscosity Liquids

The frequency responses of the FPW delay line and the FPW resonator are measured using a network analyzer (HP8753ES). The measured resonant frequency without liquid loading is 5.53 (MHz) for the proposed FPW resonator that is pretty close to the theoretical estimation of 5.88 (MHz). The difference might be due to the use of the bulk material properties in the theoretical estimation because the PZT film properties are not available.

The sensitivity analysis between the resonant frequency and the densities of low viscosity liquids is summarized in Figure 5. The theoretical relative density sensitivity for low viscosity liquids is  $-0.848$  (Mhz/g·cm<sup>-3</sup>). Three low viscosity liquids, DI Water (1 g/cm<sup>3</sup>), IPA (0.787 g/cm<sup>3</sup>) and saline solution (1.2 g/cm<sup>3</sup>), are applied to the resonator. The results show that the resonant frequency and the liquid density have a good linear correlation despite a static difference that may be due to the use of the bulk material properties in the theoretical estimation and the possible damping due to laminate structure.

### 5.2 The Frequency Deviation Due to Liquid Viscosity

Here, we compare two liquids, glycerol and saline solution with the same density to study the viscosity effect. From Table 1, we observe that the glycerol loading will introduce additional frequency deviation compared with the



**Figure 5.** The sensitivity analysis between the resonant frequency and the density for low viscosity liquids.

**Table 1.** The comparison between viscous and low viscosity liquids with the same density

	Theoretical		Experimental
	Frequency (MHz)	Frequency (MHz)	Insertion Loss (dB)
Saline solution	4.59	4.98	-33.38
Glycerol	4.49	4.73	-37.04

saline solution with the same density. Also, the viscosity effect of glycerol will cause more damping effect than the saline solution and increases the insertion loss.

## 6. Conclusion

This study has successfully fabricated the FPW resonator using sole-gel derived PZT thin films and compared with the modeling estimates. We have observed that the liquid loading will increase the insertion loss and decrease the resonant frequency that is consistent with theoretical prediction. The linear correlation between the resonant frequency and the liquid density of low viscosity liquids demonstrates the feasibility of density sensing. Additional frequency deviation and insertion loss are observed for high viscosity liquids, which also matches fairly with the theoretical estimation. However, the proposed device can't differentiate the velocity perturbations between liquid density and viscosity, which prevent the application of the device in the density sensing of viscous liquid via frequency deviation.

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