

# Design and Fabrication of Temperature Compensated Liquid FBAR Sensors

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**Abstract**— In this work we demonstrate a practically complete temperature compensation of the second harmonic shear mode in composite AlN/SiO<sub>2</sub> FBAR's in the temperature range 25°C to 95°C. The main advantages of this mode over the fundamental mode are its higher Q value in liquids as well as its higher frequency and hence higher resolution for sensor applications. For comparative reasons the non-compensated fundamental shear mode is also included in these studies. Both modes have been characterized when operated both in air and in pure water. Properties such Q value, electromechanical coupling, dissipation and sensitivity are studied both theoretically and experimentally.

An almost full temperature compensation of the second harmonic shear mode was observed for an oxide thickness of 1.22 μm and a typical 2 μm thick AlN resonator with 200 nm thick Al electrodes. Thus, the measured TCF in air for the non-compensated fundamental shear mode (1.25 GHz) varied between -31 and -36 ppm/°C over the above temperature range while that of the compensated second harmonic shear mode (1.32 GHz) varied between + 2 ppm/ °C and - 2 ppm/ °C over the same temperature interval.<sup>1</sup>

**Keywords;** *Temperature compensated FBAR; AlN; Second harmonic shear mode; liquid sensor*

## I. INTRODUCTION

A variety of physical, chemical and biochemical sensors based on the gravimetric principle utilizing electro acoustic devices have been around for a number of years. The principle of using acoustic wave mass sensitive transducers was initially employed for thickness measurements of thin rigid films in vacuum or in gaseous environments. The transverse shear mode resonator (TSMR) or Quartz microbalance (QCM) is by far the most frequently used device in gravimetric sensor applications due to its excellent frequency stability, high sensitivity and resolution. The commonly used AT cut QCM is advantageous due to its very stable temperature characteristics, i.e. the resonance frequency is almost independent of temperature variations. However, high frequency QCM operation requires a very thin quartz plate and the process to thin down the plate is both time-consuming as well as costly and therefore limits the practical maximum frequency to a few tens of MHz. This also sets a limit for the maximum mass

sensitivity, which according to Sauerbrey [1], is proportional to the square of the operating frequency.

A possible way to overcome this limitation and to increase the sensitivity further is to adopt the thin film acoustic wave technology originally developed for the telecom market for the fabrication of high frequency electro acoustic band pass filters, oscillators and delay lines. Commonly used piezoelectric materials are AlN and ZnO, which can be made arbitrarily thin utilizing thin film deposition processes, where reactive sputtering is a frequently employed method. These devices are characterized by an operating frequency in the GHz range, small size, low cost and good power handling capability [2]. With regard to gravimetric sensors this means orders of magnitude higher mass sensitivity, at least in theory. However, the sensor performance is ultimately characterized by its mass resolution, which depends not only on the sensitivity but also on the frequency stability of the resonator, i.e. the noise level. A QCM is very stable with a noise level below 1 Hz for a QCM operating at 5-10 MHz. The same value for a FBAR operating in the GHz range is usually much higher, which results in a mass resolution comparable with the QCM sensor. The main benefit using the FBAR technology is therefore not necessarily a substantial improvement of the sensor performance but rather to use the benefit of thin film technology for mass production of low cost miniature sensors. In this way a large number of sensors arranged in arrays, monolithically integrated with other components on a single chip, can be realized.

Physical and chemical sensor transducers based on thin film bulk resonators (FBAR) or solidly mounted resonators (SMR) have shown promising results for mass sensing in air or gas [3], [4]. Attempts have also been carried out to operate this type of resonators in liquid media for biochemical sensing [5], [6]. Normally, FBAR's based on AlN or ZnO utilize the longitudinally polarized thickness mode, which exhibits a significant acoustic leakage into the liquid resulting in a substantial degradation in Q.

Recently, (FBAR) based on c-axis inclined AlN [7] and ZnO [8] utilizing the thickness excited shear mode for operation in liquids have been reported, which show a great potential as high performance chemical and biochemical sensors since these devices retain a high Q factor even in contact with a liquid.

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Another critical issue in sensor applications is the temperature stability of the FBAR's. In comparison with AT-QCM, both AlN and ZnO are rather sensitive to temperature variations. The temperature coefficients of frequency (TCF) of AlN and ZnO are  $-25 \text{ ppm}/^\circ\text{C}$  and  $-60 \text{ ppm}/^\circ\text{C}$  respectively [9]. This negative TCF can be compensated by incorporating into the resonator an additional layer of a material that exhibits a positive TCF. Successful compensation of the fundamental longitudinal mode in SMR's using silicon dioxide as compensation layer, which exhibits a TCF of  $+85 \text{ ppm}/^\circ\text{C}$  was reported by [10].

Nevertheless, for a constant thickness of the piezoelectric film, this approach results in a substantial reduction of the resonance frequency of the fundamental thickness shear mode, thus decreasing the sensitivity of the sensor. Not to mention that the reduction in coupling is also substantial. Figure 1 shows the simulated shear resonance frequency versus SiO<sub>2</sub> thickness (using 1D NB model [11]) of a composite resonator having a 2  $\mu\text{m}$  thick AlN film sandwiched between two 200 nm thick Al electrodes. The electro-mechanical coupling coefficients ( $k_t^2$ ) of the 1<sup>st</sup> and 2<sup>nd</sup> shear modes are shown in Fig. 2. As seen the coupling of the 1<sup>st</sup> shear mode drops substantially while that of the 2<sup>nd</sup> harmonic increases significantly to reach a maximum value for a SiO<sub>2</sub> thickness of around 1.3  $\mu\text{m}$ . It is clearly evident from Figs. 1 and 2 that both the resonance frequency and the coupling coefficient are higher for the second harmonic at SiO<sub>2</sub> thicknesses above 0.7  $\mu\text{m}$ , and therefore it is of interest to study SiO<sub>2</sub>/AlN composite FBAR's, operating in the second harmonic shear mode for SiO<sub>2</sub> thicknesses in the range of 1 to 1.5  $\mu\text{m}$ .

This work reports measured and simulated results from membrane type FBAR's, based on a composite structure composed of a c-axis inclined AlN thin film and thermally grown silicon dioxide. The TCF for the first and second harmonics of the shear mode is studied. However, the main focus of the work has been to achieve zero TCF for the second harmonic shear mode. In addition, resonator properties such Q value,  $k_t^2$ , sensitivity and dissipation, both in air and in liquids are also measured. The results are compared with those obtained for non-compensated FBAR's operating at the fundamental thickness shear mode.

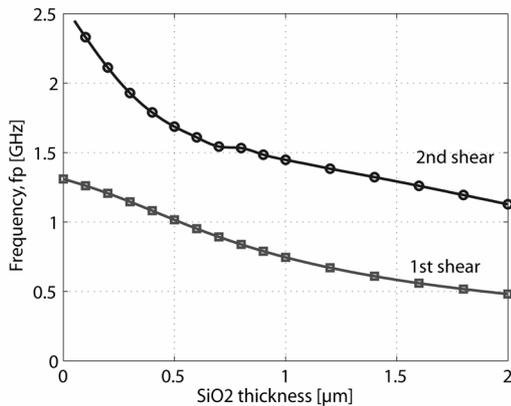


Figure 1. Simulated resonance frequency of the first and second harmonic pure shear modes versus SiO<sub>2</sub> thickness.

## II. FABRICATION

The FBAR's were fabricated in a clean room environment using standard CMOS processes and equipment. The substrates consist of double side polished silicon wafers, 300  $\mu\text{m}$  thick and 10 cm in diameter. The wafers were first thermally oxidized in a furnace at 1050  $^\circ\text{C}$  for 10 h to grow an oxide with a uniform thickness of 1.55  $\mu\text{m}$ . The wafers were then partially masked and subsequently wet etched to reduce the thickness of the exposed oxide in order to obtain a predetermined oxide thickness variation over the wafers. The oxide thickness of the different areas was determined to be 1.0, 1.22, 1.35 and 1.55  $\mu\text{m}$  respectively. Subsequently, a 200 nm thick aluminum film was sputter deposited on top of the oxide film. After patterning of the Al film, the wafer was then reinserted into its original position in the sputter process chamber for the two stage deposition of piezoelectric AlN film with a tilted c-axis process described in [12]. The resulting AlN film has a thickness of around 2  $\mu\text{m}$  while the c-axis inclination lies in an angular interval of 25 to 30 $^\circ$  (circular symmetry) over the whole wafer excluding a small area in the center where the mean tilt approaches zero. The top electrode is then deposited in the same manner as the bottom electrode and patterned. The area overlapping the bottom electrode, i.e. the active area, is 300x300  $\mu\text{m}^2$ . Finally, deep reactive ion etching (DRIE) was employed for etching the silicon substrate from the back side in order to release the composite membranes as well as to create micro-fluidic channel systems for liquid transport to the bottom resonator surface. Figure 3 shows the resonator topology and a cross section SEM micrograph of the composite layer stack. The non-compensated resonator was fabricated in an identical fashion on a non-oxidized wafer.

## III. RESULTS AND DISCUSSION

The resonators were studied by measuring the reflection coefficient in one port configuration with a Network Analyzer (HP 8720D) and Picoprobes (CCB Industries). The wafers were probed in a probe station equipped with a Thermochuck (model TP0315A, Temptronic Corporation) at temperatures in the interval 25 $^\circ\text{C}$  to 95 $^\circ\text{C}$  in steps of 10 $^\circ\text{C}$ . Typical responses in a wide band frequency interval for both types of resonators are shown in Fig. 4, where the measured S11 parameter has been

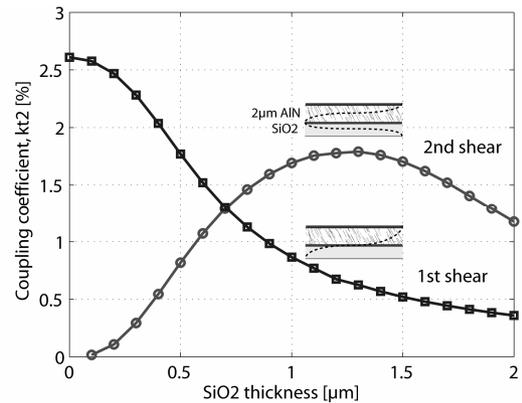


Figure 2. Calculated  $k_t^2$  of the first and second harmonic pure shear modes versus SiO<sub>2</sub> thickness.

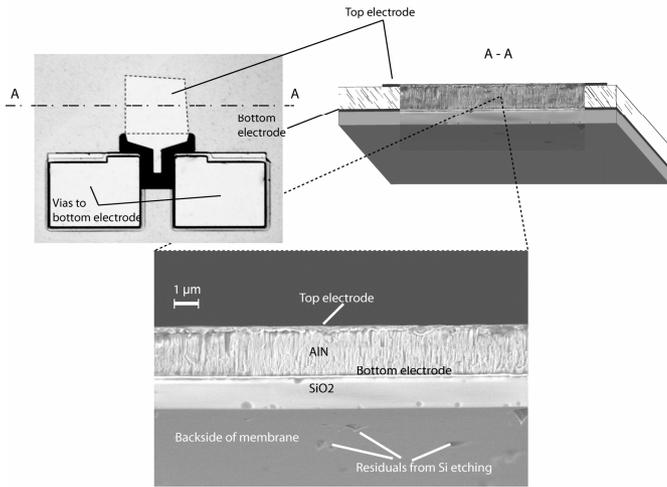


Figure 3. (Upper left) Top view of the fabricated resonator, the marked area defines the active area  $300 \times 300 \mu\text{m}^2$ . (Lower) SEM cross section image of a composite membrane.

converted to a loaded Q value. The composite resonator consists of  $1.22 \mu\text{m}$  silicon dioxide and  $2 \mu\text{m}$  AlN film, which gives a  $\text{SiO}_2/\text{AlN}$  ratio of about 0.6. Five resonances can be observed in the frequency interval 0.5 to 2.4 GHz, which are identified as the first, second and third order harmonic shear wave as well as the first and second harmonics of the longitudinal mode. For the non-compensated resonator with an AlN thickness comparable to the composite resonator, only the first shear and first longitudinal harmonics are observed at 1.25 and 2.16 GHz, respectively.

#### A. Temperature measurements

Initially, temperature measurements were performed to study the resonance frequency shift of the second harmonic shear mode for different thickness of silicon dioxide. The resonators have an AlN thickness of around  $2 \mu\text{m}$  while the  $\text{SiO}_2$  thickness in the range 1.0 to  $1.55 \mu\text{m}$ , which correspond roughly to a thickness ratio in the interval 0.5 to 0.8. The shift in series and parallel resonance frequencies as a function of temperature is shown in Fig. 5. The results show that the best compensation is achieved for the resonator composed of a

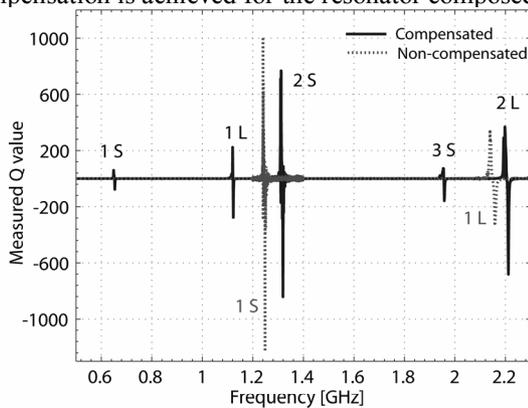


Figure 4. Measured loaded Q in air for the composite resonator with a  $\text{SiO}_2/\text{AlN}$  thickness ratio around 0.6 (continuous) and the non-compensated resonator (dotted).

$1.22 \mu\text{m}$  thick  $\text{SiO}_2$  film, where the shifts for both series and parallel resonance frequencies are small in the whole temperature range between  $25^\circ\text{C}$  to  $95^\circ\text{C}$ . For the same device, the resonance frequency increases with temperature up to around  $70^\circ\text{C}$ , where the shift exhibits a maximum, while the shift decreases for temperatures above  $70^\circ\text{C}$ . Fig. 6 is based on the same measured data fitted to a third order curve and plotted as the temperature coefficient of the parallel resonance frequency, TCFp. The TCFp has a positive value of  $2 \text{ ppm}/^\circ\text{C}$  at room temperature and decreases to  $-2 \text{ ppm}/^\circ\text{C}$  at  $95^\circ\text{C}$  with a change of sign at around  $70^\circ\text{C}$ . Full compensation at room temperature for the second harmonic shear mode can easily be achieved by tuning the thickness to be around  $1.12 \mu\text{m}$ . Thinner oxide results in under-compensation while the opposite occurs for thicknesses above this value. Figure 7 shows the measured and simulated TCFp at room temperature for the first and second harmonic shear modes. Regarding the first mode, the TCFp decreases initially to reach a minimum of  $-70 \text{ ppm}/^\circ\text{C}$  for  $0.6 \mu\text{m}$  thick  $\text{SiO}_2$  and then increases slowly to zero TCF at  $1.8 \mu\text{m}$ , at which point both the coupling coefficient and the resonance frequency of this mode have significantly deteriorated (See Figs.1 and 2). The second harmonic mode follows initially the same behavior but crosses the zero TCF at a much lower  $\text{SiO}_2$  thickness of  $1.12 \mu\text{m}$ , and then flattens out asymptotically to a value of  $6 \text{ ppm}/^\circ\text{C}$ . A very good agreement of measurements and simulations is observed.

#### B. Liquid measurements

In this study only the temperature compensated ( $1.22 \mu\text{m}$   $\text{SiO}_2$ ) second harmonic shear mode and the non-compensated fundamental mode are characterized in order to compare their behavior in liquids. When the resonators are brought in contact with pure water all longitudinal modes experience severe damping due to significant acoustic leakage into the liquid as shown in Fig. 8. The second and first harmonic shear modes for the compensated and non-compensated resonator are only partially damped and retain very high Q values of around 230 and 180, respectively. The same values in air are around 800 and 1000. The drop in Q value corresponds to a dissipation of 0.31% in the former case and 0.45% for the

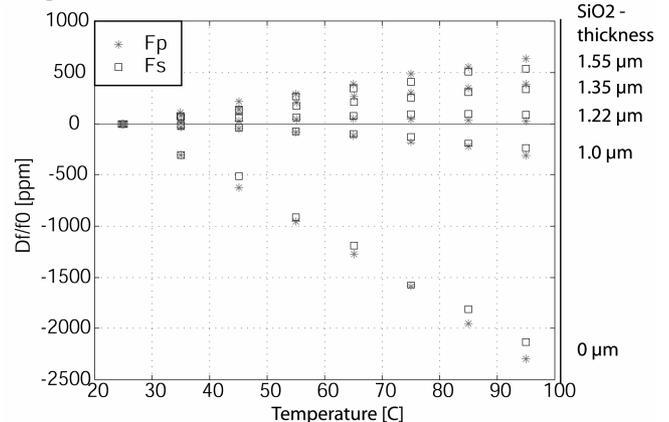


Figure 5. Measured frequency shift at temperatures ranging from 20 to  $95^\circ\text{C}$  for the second harmonic shear mode.

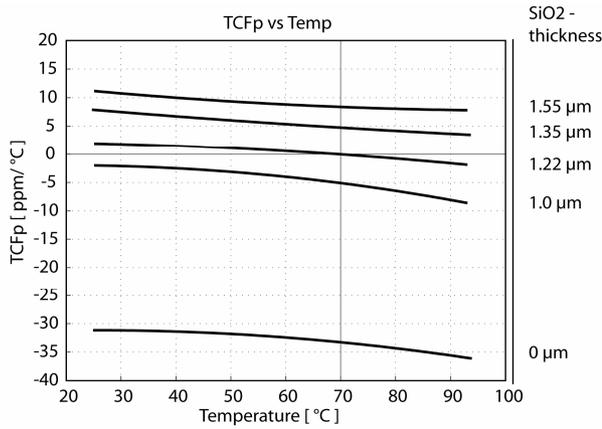


Figure 6. Measured TCFp versus temperature for different SiO<sub>2</sub> thickness. The 1.22 μm SiO<sub>2</sub> thick compensated resonator show a temperature stability ranging from +2 ppm/°C at 25 °C to -2 ppm/°C at 95 °C.

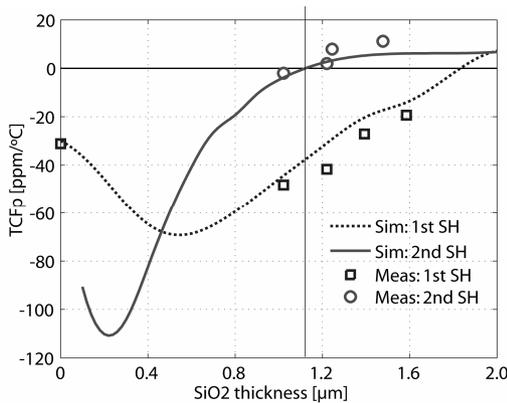


Figure 7. Measured and simulated TCFp at room temperature for the first and second harmonics of the shear mode versus SiO<sub>2</sub> thickness. A zero TCFp can be realized for the second harmonic shear with a thickness around 1.1 μm.

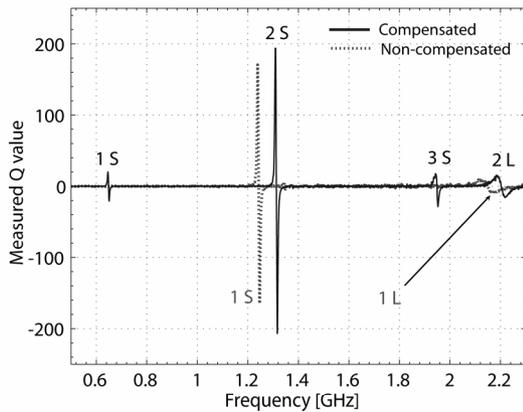


Figure 8. Measured loaded Q in pure water of the composite resonator with a SiO<sub>2</sub>/AlN thickness ratio around 0.6 (continuous) and for the non-compensated resonator (dotted).

latter, i.e. the second harmonic mode radiates less acoustic energy into the liquid compared to the fundamental mode. Further, operation in a liquid tends to lower the coupling of the fundamental shear from 2.2% to 2.0%, while for the second shear the coupling is the same for both air and liquid at a value

of 1.3%. The sensitivity to a liquid load defined as the relative change in resonance frequency is around 0.18% for both modes.

#### IV. CONCLUSION

In this work we demonstrate a temperature compensated composite shear FBAR based on inclined c-axis AlN film grown onto a layer of thermal SiO<sub>2</sub>. The resonator exhibits three important properties that make this device suitable as a high mass resolution liquid sensor, e.g. biosensor. First, this structure supports the second harmonic shear mode, which can be fully temperature compensated for a SiO<sub>2</sub>/AlN thickness ratio of around 0.55. Temperature measurements show that a TCF of the parallel resonance varying from +2 ppm/°C to -2 ppm/°C for temperatures in the range 25°C to 95°C is achieved for a device composed of 2 μm AlN and 1.22 μm SiO<sub>2</sub>. Secondly, the compensated resonator exhibits a high resonance frequency, 1.32 GHz and a coupling coefficient of 1.3%. Finally, measurements show that the second harmonic shear mode retains a very high Q value around 230 in water, which is slightly better than the fundamental mode Q value (180) for a non-compensated FBAR.

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