Analytical Modeling of AIN-Based Film Bulk Acoustic Wave Resonator for Hydrogen sulfide Gas detection Based on PiezoMUMPs.

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Abstract. Aluminium nitride (AIN) thin film bulk acoustic resonator (FBAR) sensor for hydrogen sulfide gas detection has been designed and mathematically modelled using CoventorWare and MATLAB software, respectively. The designed FBAR sensor is based on the PiezoMUMPs fabrication technology. The detection principle of the FBAR gas sensor is based on the resonant frequency changes detection due to the mass change on the top electrode of the sensor induced by the absorbed gas molecules by the nanomaterial deposited on the surface of the top electrode device. Reduced graphene oxide hybrid with copper oxide was considered as the sensitive nanomaterials and their mass loaded was evaluated in the theoretically calculation. The resonant frequency of the shear mode of the FBAR sensor has been calculated theoretically and found to be 9.4524 GHz. The effects of the gas molecules on the resonant frequency have been investigated using a mathematical equation and it shown that the increasing of the gas mass on the sensor surface will reduce the sensor resonant frequency. Furthermore, the sensitivity of the sensor was calculated to be 0.22615 Hz/fg.

Keywords: Analytical Modeling, FBAR, Gas sensing, Mass change, PiezoMUMPs.

1. Introduction
Over the last decade, there have been increased interest in developing highly sensitive chemicals and gases sensors that could help enhancing our environment and reduce the spread of the hazardous chemicals, biochemicals and gases. Micro-electro-mechanical systems (MEMS) technology has been used for developing sensors for detection of various gases in the oil and gas industries and in the surrounding environment[1]. MEMS gas sensors comprises of a micrometer device which consisted of a transducer and sensitive layer that could convert the gas information into another form of electronic signal such as frequency, current or voltage[2].

Film Bulk Acoustic Resonator (FBAR) is a promising micro-electro-mechanical system resonator for gas and chemical detection. Recently, the FBAR has received extensively attention because it has high resonant frequency, high sensitivity, and small size[3]. The FBAR electroacoustic resonator has a high fundamental working frequency at several gigahertz which provide sensors with highly enough sensitivity for gas molecular mass detection[1][4]. Compared the FBAR with the conventional
piezoelectric gas sensor such as quartz crystal microbalance (QCM), the FBAR is more advance with high frequency due to the use of the 1-2 microns piezoelectric film replacing the quartz plates in the QCM[5]. Furthermore, the FBAR can be fabricated by standard MEMS fabrication processes[6][7], therefore, the gas sensors can be produced inexpensively and combine several sensors in single chip and integrated them in an analytical circuit. For gas detection application, the FBAR is usually used as mass loading device, where a sensitive material is deposited and coated on the sensor surface to react with and absorb the targeted gas molecules[3][8]. The reaction between the targeted gas molecules and the sensitive nanomaterials or metal oxide layer which will add small mass on the top electrode layer. Theses mass changes can be observed and detected by monitoring the variation in the sensor resonant frequency. Therefore, the properties of the sensitive layer are important in the sensitivity, selectivity, stability, and reversibility of the sensor. Currently, the well-known materials that are using as coating layer including, polymers, carbon nanotubes, graphene, metal oxides and enzymes[9–13].

Graphene materials and its derivatives are considered as appropriate coating layer for hydrogen sulfide gas detection[14]. Graphene has unique sensing performance for gas detection at room temperature working condition[15]. Recently, the hybrid of graphene and metal oxide have attracted widely interest due the combination of the properties of the graphene with the metal oxides such as the high conductance, mechanical strength, and large specific surface. Moreover, reduced graphene oxide (rGO) is one of the promising graphene derivatives that can be used as platform for several kinds of gas sensors because of its high stability, easy fabrication, high carrier mobility and contained high oxygenated groups which reacted with the some targeted gases[16]. Furthermore, combing rGO with 1D nanostructured metal oxides will enhance the sensitivity of the novel sensing materials which will be able to detect the targeted gases at lowest level[14][17].

The increasing of the environmental issues and the gases leakage from the industries and oil and gas industries are threatening the human life and cause a great danger to human safety. Hydrogen sulfide is one of the flammables, toxic and extremely hazardous, colorless gas that are released during the oil and gas processes in the refineries, food processing factories, sewage, and hot springs[18–20]. The gas has smell of rotten eggs[21]. In fact, the human being cannot distinguish the H₂S gas by smell at high concentration, which is higher than 100 ppm, and in the case of expose to 320 ppm of the H₂S gas may even cause sudden cardiac death[22]. Therefore, the Occupation Safety and Health Administration (OSHA) indicated that the allowed or permissible exposure limit to H₂S is 10 ppm[23][24]. In addition, the H₂S autoignition temperature is 232 °C, and the explosion limit is 4.3%. Thus, developing a highly sensitive, selective, and stable gas sensor for H₂S detection at room temperature is extremely significant especially in the oil and gas industries.

The demand of the gas sensors that operate at room temperature is increasing rapidly because, the sensors operating at high temperature consuming high power, changing the characterizes of the nanomaterials that are using as sensitive coating layers, and the high temperature is not preferable in the flammable gas detection environment[14].

A Porous CuO nanosheet has prepared by Zhiji and co-others[25] and its H₂S sensing preparties at room temperature has been investigated. The CuO nanosheet had an average thickness of 62.5 nm and the nanomaterials has been embedded inside numerous holes with dimension ranging from 5 to 17 nm. The sensor device has presented an excellent response with good sensitivity. The sensor response and recovery time for H₂S concentration as low as 10 ppb were reported of 234, and 76 s, respectively. Furthermore, the device was reported with remarkable selectivity towards H₂S gas with small response to other gases.

Shewale et all[26], have developed a highly sensitive gas sensor for H₂S detection at room temperature. The detection coating layer was hydrothermally synthesized CuO doped with ZnO and decorated with reduced graphene oxide. The performance of the sensing layer was compared before and after doped with CuO, and the device sensitivity was remarkably enhanced by using the doped materials with Cu. This is demonstrated that the CuO has increased the reaction processes with the H₂S gas at room temperature. The response of the device was reported to increased linearly with the increase of the H₂S concentration. Furthermore, the response and the recovery of the sensor were recorded at 14 and 32 s, respectively. The others mentioned that the enhancement in the sensor sensitivity was because of both the reduced graphene oxide and the CuO.
The enhancement of the FBAR sensitivity has given extensively attention to increase the performance of the gas sensors. Zhang et al.[27], have designed and fabricated a FBAR sensor with micro holes on its top electrode for sensitivity enhancement. The sensor was designed and used for the detection of the relative humidity and ethanol. The other claimed that the micro holes on the surface of the top electrode have enhanced the sensitivity remarkably by almost 3.2 higher than the existing FBAR with complete top electrode.

The objective of the research paper is to extensively study and carry out the theoretical modeling of AIN-based FBAR sensor for hydrogen sulfide gas detection and to investigate the effect of mass loading in the top electrode of the FBAR sensor into the resonant frequency. The FBAR sensor is designed based on the PiezoMUMPs technology fabrication processes from MEMSCAP.

2. Mathematical modeling of the FBAR MEMS resonator.

Mathematically modeling was used in this work to study and investigate the changing in the resonant frequency of the FBAR MEMS when gas molecules added as mass on the top surface of the FBAR MEMS device. Figure 1, shows the model structure of the FBAR device. The dimension of the FBAR device was designed to be 500*500 µm with piezoelectrically active area which the sensitive nanomaterial will be coating and deposited on top of that layer.

![Figure 1. Schematic structure of the FBAR device](image)

As it shown in Figure 1, the active piezoelectrically active area is the area that located between the top electrode and bottom electrode only, meanwhile, the other piezoelectric materials is not consider as the active layer because it will not be excited by the applied AC voltage[5]. The FBAR resonant frequency will be reduced when gas molecular been detected. In the next section, the mathematical equations will be expressed and the detection fundamental will be explained of the FBAR sensor[12][28].

2.1. FBAR Resonant frequency

In the gravimetric FBAR sensors, the detection principle is based on the mass absorbed by the coated sensitive layer on the surface of the sensor which induced a shift in its resonant frequency. Therefore the resonant frequency will be continuously monitored[29]. In order to continuously follow the frequency variation, the FBAR system has to be embedded in a closed loop that can be either a self-oscillating loop or phase-locked loop (PLL)[30].

The bulk acoustic wave-based sensors are divided into two types depending on the way of the wave propagating in the sputtered piezoelectric layer material when an electric field is applied. They are the shear or transverse acoustic waves and longitudinal acoustic waves. The thickness of the piezoelectric material layer is also played an important role in the resonant frequency[30]. In the literature, the Quartz Crystal Microbalance (QCM) has been reported with operating frequency lower.
than a few hundred MHz which is due to the limitation of the quartz crystal layer where there is limitation in the technology to produce thin quartz layer [3]. The QCM has mass sensitivity limitation due to its relatively low operating frequency, where this limitation has been resolved by introduced and established thin film bulk acoustic wave resonator devices using MEMS fabrication technology[13][31]. The structure of the FBAR as shown in figure 1, is consisted of a very thin (0.5 µm) Aluminum Nitride (AIN) piezoelectric film sandwiched by two metallic electrodes, which are (10 µm) silicon bottom electrode, (1 µm) aluminum with (20 nm) chrome as top electrode according to the PiezoMUMPs technology processes. The FBAR device can be operating at higher resonant frequency due to the piezoelectric film low thickness, where the frequency can hit a few GHz.

The displacement $u_1$ of the piezoelectric materials AIN induced by the acoustic wave propagation inside the layer can be presented by the following equation [30]:

$$ u_1 = u_1^0 \exp \left( j \omega t - k_1 x_1 \right) $$

where $u_1^0$ is the maximal amplitude of displacement, $\omega$ is the pulsation related to the wave number, the $k_1$ is calculated as following[32]:

$$ k_1 = \frac{\omega}{V_0} $$

where $V_0$ is represented the acoustic wave phase velocity.

The acoustic wave velocity depends on the piezoelectric excitation mode, for the longitudinal acoustic wave, the acoustic wave velocity is expressed as $V_0 = V_z$ and described as the following expression [30]:

$$ V_z = \sqrt{\frac{c_{33}}{p + p_{eff} + p_{n\delta}}} \sqrt{1 + \frac{k_1^2}{1 - k_1^2}} $$

And for the transversal (shear) acoustic wave mode, the acoustic wave velocity can be expressed as $V_0 = V_{x,y}$ and it can be calculated as following expression [30]:

$$ V_{x,y} = \sqrt{\frac{c_{44}}{p + p_{eff} + p_{n\delta}}} $$

where $p$ is the mass density of the AIN piezoelectric material, $C_{33}$, $C_{44}$ are the elastic constant, and the $k_1$ is the electro-mechanical coupling factor of the bulk acoustic wave device. The values of the $C_{33}$, $C_{44}$ are 435 and 118 GPa, respectively. The electro-mechanical coupling factor can be calculated by the following expression[33][34]:

$$ k_1^2 = \frac{\varepsilon_{31}^2}{C_{11} \varepsilon_{33}} $$

where $\varepsilon_{31}^2$ is the electric field, $C_{11}$ is the elastic constant of the material, and the $\varepsilon_{33}$ is the permittivity at a constant strain. The density is defined as the measurement of mass per volume, and the average density of an objective equals its total mass divided by its total volume. Therefore, the density can be calculated as following:

$$ p = \frac{m}{V} $$

The effective density of the top electrode, nanomaterial coated on the top electrode can be expressed as following[35]:
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\[ p_{eff} = \left( \frac{t_{Al}P_{Al} + t_{Cr}P_{Cr} + t_{rGO+CuO}P_{rGO+CuO}}{t_{Al} + t_{Cr} + t_{rGO+CuO}} \right) \]  

(7)

where, \( t \), \( p \) represented thickness, density, respectively. In the case of shear acoustic wave, the FBAR resonant frequency [30] is shown in (8):

\[ f_o = \frac{V_{x,y}}{2d} \]  

(8)

where \( f_o \) is the resonant frequency, \( d \) is the piezoelectric material thickness, and \( V_{x,y} \) is the acoustic wave velocity. The key differences between the longitudinal and shear modes are depending on the c-axis angle of the crystal Columns of the deposited piezoelectric material film[5][36]. In the shear mode, the piezoelectric thin film deposited in off c-axis crystal orientation such as presented[37], where the piezoelectric material deposited with orientation of 34.5°. on the other hand, the longitudinal mode, the piezoelectric film has a crystal orientation normal to the substrate[36].

2.2. FBAR Resonator sensitivity

The FBAR resonator has various applications including the utilization of FBAR in the gravimetric measurement to be used as sensor. In the gravimetric measurement, the FBAR working principle is based on the mass change detection through monitoring the resonant frequency shift[32]. It is well-known that the resonant frequency decrease when an additional mass is added to the surface of the FBAR device. Therefore, the relationship between the frequency shift and the additional mass was developed by Sauerbrey [38] in 1959 as presented in the following equation:

\[ \Delta f = \frac{2f_o^2}{\sqrt{\mu p}} \frac{\Delta m}{A} \]  

(9)

where \( \Delta f \) is the frequency change (Hz), \( f_o \) is the resonant frequency in (Hz), \( \Delta m \) is the mass change (g), \( \mu \) is the elastic constant of the piezoelectric material (g cm\(^{-1}\) s\(^{-1}\)), \( p \) is the density of the piezoelectric material (g/cm\(^3\)), \( A \) is the piezoelectrically active area (cm\(^2\)) as shown in figure 1. Sauerbrey equation was developed for monitoring the resonant frequency of the piezoelectric material changes according to the additional mass added on the surface of the FBAR device. The deposited mass on the surface of the FBAR device is treating as an extension of the piezoelectric material thickness [5]. The additional of a uniform mass in contact with the surface of the device caused a shifted in the resonant frequency of the crystal with a mass sensitivity [4] defined as:

\[ s_m = \frac{\Delta f}{\Delta m} \]  

(10)

2.3. FBAR Resonator Quality factor

The quality factor is an important parameter for the mass detection sensors which indicates the power losses, mass resolution, and detection limit. There are several methods to determine the quality factor which affected by the quantity of the sensitive layer coated on the top surface of the device which considered as mass loaded that will cause a drop in the resonant frequency. The quality factor of the FBAR resonator can be calculated by the following expression [35]:

\[ Q_{ab} = \frac{f_x}{|f_i - f_x|} \]  

(11)

where, \( f_o \) is the resonant frequency, \( f_i, f_x \) are the frequencies at which the insertion and return wave in the piezoelectric material loss are 3 dB compared to the resonant frequency insertion and return loss [35].

3. Design of FBAR resonator based on PiezoMUMPs technology.

The FBAR device in this research so far has been designed using CoventorWare finite element analysis (FEA) software for the purpose of simulation which will be illustrated in the upcoming work. The FBAR
device has successfully designed layer by layer using the PiezoMUMPs fabrication technology from MEMSCAP[39]. The FBAR gas sensor device structure and dimensions has been presented in figure 1 and table 2, respectively. The CoventorWare software had provided us unique features for FBAR design such as 3D schematic of the device.

3.1. FBAR design concept and details
The FBAR gas sensor has been built up of various materials as shown in table 1 and Table 2. The Materials properties have been used in the CoventorWare software to be updated in the materials database. In this sensor device, AIN piezoelectric material has been used as the active material for mass sensing application. Silicon was used as substrate because it has the applicability to be integrated with electronic circuit.

**Table 1.** Properties of commonly used piezo-materials[40].

| Materials                | AIN       | ZnO       | PZT       |
|--------------------------|-----------|-----------|-----------|
| Density (g/cm^3)          | 3.255     | 5.61-5.72 | 7.6       |
| Young’s Modulus (GPa)     | 300-395   | 208       | 56-98     |
| Poisson’s Ratio           | 0.22-0.29 | 0.36      | 0.27-0.3  |
| Piezoelectric constant d_{33} (pC/N) | 3.4-6.4 | 5.9-12.4 | 60-223    |
| Effective Coupling Co-efficient k^2 (%) | 3.1-8    | 1.5-1.7  | 20-35     |
| Acoustic Velocity of longitudinal waves (m/s) | 10150-11000 | 6333 | 4500    |
| Acoustic Velocity of transverse waves (m/s) | 5500-5670   | 2700       | 3900     |
| Dielectric Constant       | 8.5-10.5  | 10.9      | 300-1300  |
| Co-efficient of thermal expansion (CTE) ×10−6 °C | 5.3       | 4-6.5     | 1.75-2    |

Furthermore, the FBAR sensor for hydrogen sulfide detection has been designed with dimension that illustrated in table 2. The thickness of each layer was built up according to the PiezoMUMPs technology features which has fixed materials and their thickness as standardized fabrication processes. The layers dimension has also presented where the performance of the FBAR device depending on the thickness and the area of the piezoelectric active layer. The FBAR performance and its resonant frequency changes with the added mass of the sensor has been investigated using MATLAB software and the frequency output will be shown in the result.

**Table 2.** FABR sensor dimension.

| Materials | Design parameters | Layer name | Layer dimension (µm) | Thickness (µm) |
|-----------|-------------------|------------|----------------------|----------------|
| Silicon   |                   | Substrate  | 500*500              | 400            |
| Thermal Oxide |             | Oxide     | 400*400              | 1              |
| SOI       |                   | SOI        | 340*340              | 10             |
| Thermal Oxide |             | Pad Oxide  | 200*200              | 0.2            |
| AIN       |                   | PZF        | 180*180              | 0.5            |
| Al+Cr     |                   | Pad Metal  | 160*160              | Al (1µm) Cr (20 nm) |
3.2. **FBAR MEMS structural based on PiezoMUMP technology**

Figure 2. shows the designed FBAR device without the silicon substrate which is not shown for maximize the view of the device.

![Figure 2. 3-D structure of the FBAR gas sensor.](image)

The bottom gray color represented the thermal oxide layer with 1 µm thickness and 400*400 µm dimension. The second layer with red color displays the SOI layer which is used as bottom electrode for the FBAR device. The SOI layer thickness is 10 µm and with 340*340 µm dimension. The third layer with yellow color represented the pad oxide which used as insulator between the top electrode and the bottom electrode with a thickness of 0.2 µm and 200*200 µm dimension. The AIN piezoelectric layer is located between the top electrode and bottom electrode with a thickness of 0.5 µm and dimension of 180*180 µm. The black color layer illustrated the top electrode of the FBAR device which consisted of 1 µm aluminum and 20 nm chrome metals.

4. Result and discussion.

The resonant frequency of the shear FBAR device has been mathematically calculated using MATLAB software to investigate the response of the sensor with the top electrode and the coated rGO hybrid with CuO nanomaterials layer. The effective density of the top electrode and hybrid layer was calculated using equation 7 and found to be 3665 kg/m³, and the acoustic velocity of the transverse wave was calculated using equation 4 and found to be 4726.2 m/s.

The resonant frequency of the shear mode FBAR was found to be 9.4524 GHz without any H2S absorbed by the sensitive nanomaterials. Table 3 presented the FBAR sensor response as a function of the mass added into the sensitive layer. The table 3 shown the actual values of the frequencies of the shear FBAR resonator at a different value of mass. The results have proven that when the mass loading in the top electrode increased the resonant frequency will decrease as it illustrated in figure 3. Furthermore, the device sensitivity has been calculated using equation 10 and found to be 0.22615 Hz/pg and 226.15 Hz/ng.

| Added mass | f (GHz)         | f(kHz)         |
|------------|-----------------|----------------|
| 0          | 9.45240918141   | 9452409.18141  |
| 1 fg       | 9.45240918141   | 9452409.18141  |
| 50 fg      | 9.45240918140   | 9452409.18140  |
| 100 fg     | 9.45240918139   | 9452409.18139  |
| 500 fg     | 9.45240918130   | 9452409.18130  |
| 800 fg     | 9.45240918123   | 9452409.18123  |
| 1 pg       | 9.45240918119   | 9452409.18119  |
| 100 pg     | 9.45240915880   | 9452409.15880  |
500 pg  |  9.45240906834 |  9452409.06834
1 ng   |  9.45240895526 |  9452408.95526
100 ng |  9.45238656645 |  9452386.56645

Figure 3. FBAR response to mass change.

5. CONCLUSION
In conclusion, in this paper, the mathematically modeling for the FBAR gas sensor for hydrogen sulfide detection has been presented. The sensor has been designed based on AIN thin film and using PiezoMUMPs fabrication technology processes. The FBAR resonant frequency was theoretically found to be 9.4524 GHz and the sensor sensitivity was found to be 0.22615 Hz/pg. It has been proven that the FBAR sensor frequency decreased with the increasing in the mass load on the top electrode of the sensor.

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