Large scale changes in overtone resonances and “resonant peak burning” of HBAR with mass loading and its potential for applications.

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This report presents the realization and characterization of a robust composite resonator i.e. high overtone bulk acoustic wave resonator (HBAR) and the changes happening to the resonance peaks once it undergoes mass loading which has got the potential for application in material characterization, communication system and sensing. Mass loading effect on a HBAR based on Ba$_x$Sr$_{1-x}$TiO$_3$ (BST) have been demonstrated by coating photoresist of various thicknesses and characterization of resonance modes present in the frequency spectrum of the resonator. Upon investigation, HBAR proves to be one of the most promising and robust systems for gravimetric sensing. Burning effect in the resonances occurs and it shifts significantly according to the amount of mass loaded (increasing thickness of the photoresist coated) on the resonator system. Some of the most important parameters like effective coupling coefficient ($k_{eff}^2$), spacing of parallel resonance frequency (SPRF) and quality factor of the resonator and its numerous modes have been investigated meticulously. The SPRF’s minima shifts by 550 MHz when the thickness of coated photoresist changes by around 335 nm. Hence, the designed resonator holds immense potential for broadband and ultrasensitive sensors. The resonator also exhibits very high $k_{eff}^2$ and Q factor and is highly dependent on the thickness of the coated layer.

Acoustic wave resonators have been used and applied in various applications ranging from military to consumer electronics. Applications in RF front-end like filters and oscillators etc. as well as sensors have been investigated by various groups$^{1-4}$. Surface acoustic wave resonators (SAW), Film Bulk Acoustic Wave Resonator (FBAR), Solidly Mounted Resonators (SMR) are some of the most popular devices in the resonator market till today and continues to have a very good projection into the future as well. Amongst the resonators, composite resonators like HBAR has been investigated and began to be applied in sensors, filters and oscillators$^{3,5-9}$. The use of ferroelectric thin films like BST, which employs induced piezoelectricity, gives an added advantage of being switchable and slightly frequency tunable with an applied bias voltage when compared to fixed frequency resonators based on piezoelectric thin films$^{10}$. The change in the value of dielectric constant of ferroelectric films like BST with an applied electric bias also gives an added advantage of studying the effect of bias (varying dielectric constant) on the various parameters of the resonator.

Various works on gravimetric sensing have been done by various groups$^{4,11-14}$. Many definitions of sensitivity have also been put forward$^4$. The most important means for sensing is by using the Quartz crystal microbalance (QCM) and the mass loading phenomenon, which make use of the change in resonant frequency of a resonator. The lesser the amount of mass that

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can be detected by the resonator, the more sensitive it is and there is always a limitation as to how much mass can be detected, and the appropriate resonator and the experimental set-up to be used for the procedure. Notable works on FBAR based gravimetric sensor has been demonstrated by using carbon nanotubes (CNT) as the replacement for both the top electrode as well as the sensing layer 12. Another is the comparison between FBAR and SMR gravimetric sensors13. Sensitivity of the resonator is the ability to give measurement data with precision and accuracy.

HBAR, a composite resonator, has a simple configuration of a thick low acoustic loss substrate along with a transducer which has an active layer sandwiched between two electrodes10. HBAR has the advantage of being easy to fabricate, is robust and multifunctional. HBAR has one of the best figures of merit (FOM) in terms of Quality factor and frequency product (Qxf). This attribute has been exploited in designing low phase noise oscillators. Other applications include material characterization and sensing.

The use of thin ferroelectric films like BST in its paraelectric state has an edge over the conventional piezoelectric films as the former becomes switchable and tunable 10. Various work have been reported in the field of acoustic wave resonators which employ thin ferroelectric films. In this work, the main motivation is to demonstrate the phenomenon of mass loading on BST based HBARs by coating polymers on the backside of the substrate. Backside coating for mass loading gives lots of flexibility in testing the system and makes the study simpler and increases its reproducibility. We also report the effect of the coating on the various aspects of the resonators performances i.e. effective coupling coefficient ($k_{eff}^2$), spacing of parallel resonance frequency (SPRF) and quality factor.

A sapphire substrate of thickness $\approx 495\mu m$ (area $1cm \times 1 \ cm$) is taken for studying their mass loading effects. Upon the substrate, platinum (Pt) is deposited using an RF sputtering system. The Pt coated substrate is then taken and coated with Ba$_{0.5}$Sr$_{0.5}$TiO$_3$ (BST5) using a pulsed laser deposition (PLD) technique. The conditions for BST5 are same as previously reported 10. Gold(Au) is coated as the top electrode and a circular patch capacitor (CPC) structure of 45 $\mu m$ radius is patterned, this makes the device fabrication possible with a single mask making it easier to fabricate. Measurements for scattering parameters (1 port S$_{11}$) are performed on the sample in the microwave frequency range of 200MHz-4GHz with bias voltage of 40V. The measurement setup involves an on-wafer probing station, bias tee, GSG probes and Vector network analyzer.

By using a spin coater, three different thickness of photoresist are coated on the backside of the resonator by using spin coater at different speeds. Table 1 specifies spin speed used and the thicknesses of the coated photoresist. The photoresist used is a positive photoresist, OiR620(Arch Chemicals, Inc.), which after spin coating is baked for 20 mins on a hot plate at a
temperature of 85°C in each of the cases. Initially, before coating, the microwave measurements are performed by switching the resonator on by applying a dc bias of 40 V. And, after spin coating for a particular spin speed, the measurement is performed for the coated sample and acetone is used to dissolve or clean the surface on the backside after measurement. These steps are repeated for the spin conditions. Initial coating for 500 rpm is performed for 5 secs for all the cases and the spin as specified in table 1 is carried out for 30 sec.

Table I. Spin speed vs thickness of the photoresist.

| No. | Spin(rpm) | Thickness(Å) |
|-----|-----------|--------------|
| 1   | 2000      | ≈13367       |
| 2   | 3000      | ≈11505       |
| 3   | 4000      | ≈9817        |

For the broadband measurement, data points of 32,000 is used. And for a narrow band measurement, i.e. to analyse the individual modes, a window of 100 MHz with 32,000 points is set starting from 500 MHz till 3 GHz. Figure 1 shows the plots for the extracted input impedances of the resonator with and without the photoresist coating on its backside. It can be clearly seen that when we compare the case of non-coated resonator and a coated resonator, there is a significant sign of disturbance in the spectrum of the resonator. This is mainly due to the change in boundary conditions in the case where photoresist is coated on the backside. The four-layer configuration of the HBAR, i.e. the two electrodes, the thin ferroelectric film and the substrate have been changed to a five-layer configuration resonator, and this in turn changes the response of the HBAR. Another interesting observation that can be made regarding the mass loading effect is the remarkable shift of the ‘burned resonance area’ towards a higher frequency upon reduction in the thickness of the photoresist coated or upon increase in the spin speed. The burned resonance area is the region in the frequency vs the impedance plot of the resonator where the intensity of the resonant peaks in the region is suppressed significantly by the introduction of an additional layer with different acoustic impedance. It is clearly evident from these results, that HBAR has the capability to be used or applied for various sensing applications. Apart from the analysis that can be performed from the impedance spectrum of the HBAR; the spacing of the parallel resonance frequency, SPRF, effective coupling coefficient, $k_{eff}^2$ and the Quality factor, $Q$ of the various modes of the spectrum are analysed.

Since the number of modes or resonant peaks are very high depending on the parameters defined mainly by the thickness of the substrate, it is important to analyse each and every mode even though it is a time-consuming technique in terms of both the measurement and the analysis of data. From figure 2-4, it can be inferred that the various aspects of measured results i.e. SPRF, $k_{eff}^2$ and the Q factor of the resonator changes drastically in the case with and without polymer coating. In figure 3, there is a gradual shift in the minima of the SPRF with increase in mass loading of the resonator. For this case two regions in
the spectrum are considered, the first is the minima occurring below 1GHz and the other is the region around 2GHz. The first case accounts to a change of around 180 MHz with a thickness difference between 2000 rpm and 4000 rpm i.e. roughly around 355 nm. And in the second region, the change in frequency is 550MHz. It can be inferred from this result that the designed HBAR can be used as a sensor for detecting materials taking advantage of the broad shift in frequencies involved. Another inference is the sensitivity or the shift in the minima which is higher in the case with higher frequency. These significant changes are all attributed to the mass loading of the resonator and the change in boundary or interface condition by the introduction of an additional layer in the system. The SPRF of the resonator which is mostly dependent on the property of the substrate like thickness, density and acoustic wave velocity changes and by properly analysing the changes, material characterization of the additional polymer layer can be performed, which is not reported in this work. The $k_{eff}^2$ of the resonator which primarily depends on the active ferroelectric thin film layer also changes accordingly with the amount of polymer coated on the resonator showing that the unique mode or the fundamental mode of the HBAR can be changed or tuned by using add on layers at the opposite end of the substrate. From the Q factor of all the cases, it is evident that the Q of all the resonators are fairly high when compared to FBARs$^{15}$, and hence each and every mode can be used for detection of minute amount of mass change in the resonating system.

It can be clearly pointed out from this study that HBAR can be easily used in mass sensing applications, filters and material characterization. The only limitation is the difficulty in analysing each and every mode present in the spectrum. Even so, the parameters characterized from the resonators gives us a very good insight on how a simple and robust structure like HBAR can be employed in some of the most precision dependent applications like mass sensors. Any one of the parameters of the HBAR i.e. the SPRF, $k_{eff}^2$ and the Q can be equally utilized to design an ultrasensitive system for applications in biosensing and other sensing purposes. Due to introduction of an additional layer in the four layered composite resonator structure, acoustic mismatch happens, and hence, SPRF, $k_{eff}^2$ and Q distribution of the system changes drastically.

The design is of a simple one mask process and since the substrate is 0.5 mm thick, handling and processing of this resonators are easier compared to many of its counterparts. An added advantage is that the functionalization need not be done on the active electrode area but rather on the backside which gives an edge over other techniques available. The SPRF, $k_{eff}^2$ and the Q factor of the resonator have been analysed and reported here in this work. The multimode nature of HBAR in itself is quite difficult because of the data to be measured and analysed are enormous but it holds great untapped utility with its applicability ranging and spanning in various multidisciplinary applications of which sensing seems to be the one.
Figure 1: Frequency spectra of HBAR with and without photoresist coatings.

Figure 2: $k_{eff}^2$ distribution for photoresist of various thicknesses.
Figure 3. SPRF distribution for various photoresist thicknesses.

Figure 4: Q factor for various photoresist thicknesses.

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