Localized and distributed mass detectors with high sensitivity based on thin-film bulk acoustic resonators

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A mass sensor based on thin-film bulk acoustic resonator, intended for biomolecular applications, is presented. The thin film is a (002) AlN membrane, sputtered over Ti/Pt on a (001) Si wafer, and released by surface micromachining of silicon. Two experiments are proposed to test the mass sensing performance of the resonators: (a) distributed loading with a MgF2 film by means of physical vapor deposition and (b) localized mass growing of a C/Pt/Ga composite using focused-ion-beam-assisted deposition, both on the top electrode. For the distributed and localized cases, the minimum detectable mass changes are $1.58 \times 10^{-8}$ g/cm² and $7 \times 10^{-15}$ g, respectively. © 2006 American Institute of Physics. [DOI: 10.1063/1.2234305]

The interest for thin-film bulk acoustic resonators (FBARs) comprises several applications. In mass sensor applications, quartz crystal microbalances (QCMs) have been a key technology. However, most recent and demanding applications—e.g., biomolecular or chemical detection—and the limited frequency range of QCM-based systems have created the need for conceiving devices with increased sensitivity. FBAR devices are able to replace QCM in such areas where higher mass sensitivity is required. Other technological approaches, such as nanoelectromechanical system (NEMS) resonators, have also proved to achieve very high sensitivity in localized-mass detection applications.

The FBAR-based mass sensor operates in the principle of mass loading, which is typically implemented by growing or depositing a thin film in one of the electrodes of the resonator. The fabrication technology determines the manner and the electrode in which the thin mass is deposited. The mass loading acts in the sensor’s frequency response, by changing its resonance frequency $f_0 = v_0/2t_0$, where $v_0$ and $t_0$ are sound velocity and thickness of the unloaded resonator, respectively. For added masses with density $\rho_m$ and thickness $t_m \ll t_0$, the loaded resonance frequency $f_m$ is evaluated in the Sauerbrey-Lostis equation as

$$f_m = \frac{1}{f_0 + 2(\rho_m t_m/\rho_0 t_0)}.$$  \hspace{1cm} (1)

In Eq. (1) the term at the right of $f_0$ corresponds to the frequency change $\Delta f$ due to added mass $\Delta m$. In this way, the frequency change relative to the unloaded resonance frequency can be solved as

$$\frac{\Delta f}{f_0} = -\frac{\rho_m t_m}{\rho_0 t_0} = \frac{\Delta m}{m_0}. \hspace{1cm} (2)$$

Equation (2) is valid if $\Delta m$ is less than 2% of the initial mass of the resonator $m_0$. For the case of distributed mass sensors, two parameters are mainly considered to evaluate the performance: Mass sensitivity (cm²/g), defined as $S_m = 1/\Sigma \rho t$, where $\rho_i$ and $t_i$ are density and thickness for each material layer in the resonator’s stack, and frequency responsivity $R_f = \Delta f/\Delta f_0$, where $\Delta f$ is the minimum detectable frequency shift. The minimum detectable mass change per unit area can be evaluated from $\Delta m = R_f S_m$. In the same way, mass responsivity per area $r_m$ (g/Hz/cm²) is calculated from $r_m = 1/(f_0 S_m)$. On the other hand, for localized-mass deposition, mass responsivity $R_m$ is the change in frequency response per unit mass change (Hz/g), but in certain cases it is more convenient to deal with the inverse responsivity (g/Hz). In this letter, the word responsivity will be used to express inverse responsivity, being the minimum detectable mass change calculated as the product $\Delta f R_m$. A typical value for a QCM operating at 40 MHz can be found in the units of ng/Hz/cm², while for a FBAR operating at 1 GHz this value reaches the units of pg/Hz/cm², i.e., 1000 better for the latter. In this work, focus is laid on the exploration of different capabilities of FBAR as mass sensors as it is the case of localized-mass detection.

The FBAR was implemented as a sandwiched (002) aluminum nitride (AlN) membrane (1 µm thick), sputtered on top of a titanium/platinum (Ti/Pt) layer (30/150 nm thick) deposited on a (001) silicon (Si) substrate, and released by surface micromachining of the silicon substrate (Fig. 1). Two

![FIG. 1. Side view of the FBAR process.](image-url)
different geometries and sizes for the FBAR electrodes were designed, being one of them a rectangular-shaped device \((50 \times 70 \, \mu m^2)\), and the second one an irregular rhomboid \((70 \times 130 \, \mu m^2)\). For this configuration, the FBAR resonates in the 2 GHz range, and its theoretical mass responsivity per area is \(4.15 \times 10^{-13} \, g/Hz/cm^2\).

Concerning the experiments to deposit mass on the FBAR, the whole surface of the top electrode of several devices was covered with a physical-vapor-deposited uniform thin film of magnesium fluoride (MgF\(_2\)), with different thicknesses—2, 5, 10, and 20 nm—in diverse samples. Taking into consideration these thicknesses, four groups of resonators were characterized. As expected, the resonance frequency shifted down several megahertz, in linear proportion to the amount of deposited mass (Fig. 2).

As a general comment, this experiment does not exceed the limits for the Sauerbrey-Lostis equation to be valid, being the ratio \(\Delta m/m_0\) between 0.06\% and 0.64\%. Hence, from the frequency shifting and deposited mass data in Fig. 2, the experimental mass responsivity per area was found to be \(5.23 \times 10^{-13} \, g/Hz/cm^2\), which is 80\% of the theoretical value for the current FBAR stack configuration. The minimum mass change that can be detected with the current measurement setup and FBAR configuration is evaluated by checking the minimum detectable frequency shift, which occurred to be \(\Delta f = 30 \, kHz\), thus obtaining a value of \(1.58 \times 10^{-6} \, g/cm^2\). The minimum frequency shifting was found from the phase of the S21 parameter. First, the phase noise is quantified from a zero-span acquisition observing the series resonance frequency (Fig. 3). As observed in Fig. 3, the maximum deviation from the mean phase value is 0.8 deg, which is divided by the phase slope—\(2.65 \times 10^{-6} \, deg/Hz\). This value is calculated from differentiation of the S21 phase and evaluated at the series resonance frequency.

In a second experiment, deposition in selected areas of a second set of resonators has been performed inside a focused-ion-beam (FIB) machine, in order to test their capabilities for localized-mass detection. For this, a platinum-containing metal organic precursor, injected in the sample’s chamber, has been decomposed by the ion beam, giving rise to the localized deposition of an amorphous compound that contains C, Pt, and Ga (65\%, 27\%, and 8\%, respectively) in the area scanned by the beam,15 whose mass density being around 4 g/cm\(^2\). In these experiments different-sized square depositions have been tested. By localized-mass deposition we must understand the deposition of a material, whose contact surface with the resonator is quite small, compared with the effective resonator’s surface. In fact, the deposited mass area for each one of the samples is always less than 0.7\% of the electrode surface. The scanning electron microscope (SEM) image of Fig. 4(a) shows a rectangular-surface mass, deposited on the center of the top Ti/Pt electrode of a rectangular FBAR, with an enlarged view in Fig. 4(b).

As in the uniform-mass case, a direct proportionality in the frequency and mass changes is observed for the localized-mass deposition (Fig. 5). For this latter case, down-shifting of resonance frequency is higher, even for smaller deposited masses. This situation suggests an increase on the mass responsivity for FBAR sensors in localized-mass applications.

Additional information concerning the mass loading are obtained in Table I, where responsivities and minimum mass detectable are compared for the cases of distributed and localized loadings, showing that the localized-loading case has figures of performance that are one order of magnitude higher. For example, the mass responsivity \(R_m\) of the FBAR—an average of \(2.38 \times 10^{-12} \, g/Hz\)—is better, compared with those of mass sensors based on NEMS resonators.5,6 Concerning the calculation of mass sensitivity, it is deduced from the product \(\Delta f/R_m\) obtaining a value of \(7.18 \times 10^{-15} \, g\). Further experiments would allow the extrac-

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**FIG. 2.** Frequency shifting vs mass loading (uniform-mass case): a linear behavior is observed for irregular rhomboid devices.

**FIG. 3.** Zero-span plot of the S21 phase response (deg) of a FBAR. Phase noise is evaluated as the maximum deviation from the mean phase value \(0.8 \, deg\). This value is divided by the slope at the resonance frequency \((2.65 \times 10^{-6} \, deg/Hz \text{ at } 2.29 \, GHz)\) to calculate the minimum detectable phase frequency shift, which is employed in evaluating the minimum detectable mass.

**FIG. 4.** (a) SEM image of a localized mass deposited on a rectangular FBAR sensor (the upper and lower edges of the FBAR are the horizontal bright lines in the image) and (b) larger magnification. A halo is observed around the mass which is caused by slight contamination during the deposition process. The images are taken tilted 52\° around a horizontal axis.
From the presented results, piezoelectric-based thin-film sensors appear as a candidate technology alternative to NEMS for very high mass sensitive applications. It has been experienced that, as only a very small fraction of the overall electrode area is loaded, the responsivity is increased between one and two orders of magnitude, compared with the uniform-deposition case. This discovery could be used to detect localized particles and allows the application of FBAR as biological mass sensor where selective spatial detection is required.

In these initial experiments, the ability of FBAR as high-responsivity, high-sensitivity localized-mass sensors has been demonstrated. Further study would allow to understand the eventual relationship between the location in which localized mass is deposited and the resonance modes of the sensor. Also, the development of improved setups for measuring the mass sensitivity is needed to evaluate the possibilities and target applications of FBAR-based mass detection.

<table>
<thead>
<tr>
<th>Case</th>
<th>Mass responsibility per area</th>
<th>Minimum detectable mass per area</th>
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<tbody>
<tr>
<td>Localized-material</td>
<td>$2.38 \times 10^{-19}$</td>
<td>$7.18 \times 10^{-15}$</td>
</tr>
<tr>
<td>Distributed-material</td>
<td>$4.71 \times 10^{-17}$</td>
<td>$5.23 \times 10^{-13}$</td>
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