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Abstract

This paper reports on the design and experimental verification of a new class of ultra-thin-film (250 nm) aluminum nitride (AlN) microelectromechanical system (MEMS) contour mode resonators (CMRs) suitable for the fabrication of ultra-sensitive gravimetric sensors. The device thickness was opportunely scaled in order to increase the mass sensitivity, while keeping a constant frequency of operation. In this first demonstration the resonance frequency of the device was set to 178 MHz and a mass sensitivity as high as 38.96 KHz·µm2/fg was attained. This device demonstrates the unique capability of the CMR-S technology to decouple resonance frequency from mass sensitivity.

Keywords

gravimetric sensors; AlN contour-mode resonators; sensitivity; ultra-thin-film AlN.

Disciplines

Acoustics, Dynamics, and Controls | Electrical and Electronics | Electro-Mechanical Systems | Electronic Devices and Semiconductor Manufacturing | Nanoscience and Nanotechnology | Nanotechnology Fabrication

Ultra-Thin-Film AlN Contour-Mode Resonators for Sensing Applications

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Abstract— **This paper reports on the design and experimental verification of a new class of ultra-thin-film (250 nm) aluminum nitride (AlN) microelectromechanical system (MEMS) contour-mode resonators (CMRs) suitable for the fabrication of ultra sensitive gravimetric sensors. The device thickness was opportunely scaled in order to increase the mass sensitivity, while keeping a constant frequency of operation. In this first demonstration the resonance frequency of the device was set to 178 MHz and a mass sensitivity as high as 38.96 KHz·μm^2/fg was attained. This device demonstrates the unique capability of the CMR technology to decouple resonance frequency from mass sensitivity.**

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I. INTRODUCTION

In recent years several efforts have been dedicated to the demonstration of ultra sensitive gravimetric sensors capable of substituting the bulky and unintegrable Quartz Crystal Microbalances (QCMs) [1] for chemical and bio sensing applications. QCMs have been successfully employed as gravimetric sensors thanks to their extremely high quality factors, which permits them to achieve limits of mass detection in the order of few nanograms. Despite this, the relatively large volume of QCMs, their low frequency of operation (only few MHz) and their inability to be directly integrated on silicon make them unattractive for the fabrication of smart, compact, portable and low cost electronic noses for multiple gas detection.

In this perspective, micro and nanoelectromechanical devices have emerged as ideal substitutes for QCMs. These new devices make excellent gravimetric sensors thanks to their reduced dimensions, higher frequency of operation and integrability with circuits. Different MEMS/NEMS resonator technologies based on electrostatic [2] or piezoelectric transduction [3] have been proposed. In particular, thin Film Bulk Acoustic Resonators (FBARs) [4,5] can provide values of mass sensitivity much higher than QCMs thanks to their reduced volume and higher frequency of operation (GHz range). Nevertheless, for FBAR, as for QCM, the operating frequency is set by the thickness of the piezoelectric layer. This precludes the capability of these devices to provide for multiple frequencies of operation (hence mass sensitivities) on the same substrate and exclude their employment for the fabrication of

multi frequency single chip sensor arrays capable of selectively detect multiple Volatile Organic Chemicals (VOCs) in a wide range of concentrations. This issue has been recently overcome by AlN Contour-Mode Resonators (CMR) [3] for which the operating frequency is set lithographically and therefore multiple mass sensitivities are possible on the same silicon substrate.

High performance AlN Contour-Mode Resonant Sensors (CMR-S) with multiple frequencies of operation on the same chip and capable of detecting multiple VOCs have been previously demonstrated by our group [6]. In this work, the unique capability of CMR-S to decouple resonance frequency from mass sensitivity is investigated. In fact, differently from conventional resonant sensors, such as QCMs or FBARs, whose operating frequency (set by the thickness of the piezoelectric layer) is intrinsically related to the sensitivity of the device to mass loading, the AlN CMR-S can use the film thickness to set the device sensitivity independently from the frequency of operation.
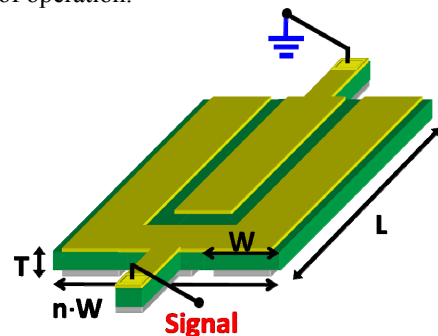

Figure 1. Schematic rapresentation of the ultra-thin film AlN CMR: a number, *n*, of subresonators (width, *W*) are mechanically coupled in order to excite a higher mode of vibration in the AlN plate (f_0 is set by *W*).

In this first demonstration the resonance frequency of the device was set to 178 MHz while the thickness of the AlN film was scaled in the nano range (250 nm thick) to achieve a value of mass sensitivity as high as 38.96 KHz·μm^2/fg (comparable to an FBAR operating at approximately 1 GHz).

The capability of CMR-S to increase the mass sensitivity by scaling the device thickness without altering their frequency of operation is extremely advantageous for their use for example in Wireless Sensor Networks (WSN), where the resonant

devices are required to operate in a specific frequency range determined by the radio links.

II. DESIGN

A CMR is composed of an AlN film sandwiched between two patterned metal electrodes (Fig. 1). When an AC votlage is applied across the thickness (T) of the device a contour-extensional mode of vibration is excited thorough the equivalent d_{31} piezoelectric coefficient of AlN. Given the equivalent mass density, ρ_{eq}, and Young's modulus, E_{eq}, of the material stack (AlN and metal electrodes) that forms the resonator [7], the center frequency, f_0, of this laterally vibrating mechanical structure, is univocally set by the period, W, of the metal electrode patterned on the AlN plate and can be approximately expressed as in Eq. (1).

$$f_0 = \frac{1}{2W}\sqrt{\frac{E_{eq}}{\rho_{eq}}} \qquad (1)$$

The other two geometrical dimensions, thickness, T, and length, L, set the equivalent electrical impedance of the resonator [3] and can be designed independently of the desired resonance frequency.

The sensitivity to mass per unit area of a CMR device loaded on its top surface can be expressed as in Eq. (2) [6].

$$S_{CMR} = -\frac{f_0^2}{\sqrt{E_{eq}\rho_{eq}}}\frac{W}{T} \qquad (2)$$

The difference (the multiplicative factor $W/2T$) between Eq. (2) and the Sauerbrey equation [8], commonly used to describe the mass sensitivity of QCMs and FBARs, is due to the fact that, for CMRs, the loading mass is not distributed where the kinetic energy is maximum (the sidewalls), but on the top surface of the device [9].

In order to explicitly express the dependence of the mass sensitivity on the device resonance frequency, f_0, and the thickness, T, it is convenient to combine Eq. (1) and Eq. (2). This leads to the compact expression of Eq. (3).

$$S_{CMR} = -\frac{f_0}{2\rho_{eq}T} \qquad (3)$$

With a similar derivation it is possible to find an expression (Eq. (4)), function of the same parameters (f_0, ρ_{eq}, T), for the mass sensitivity of devices, such as FBARs and QCMs, whose resonance frequency is set by the device thickness, T.

$$S_{FBAR} = -\frac{f_0}{\rho_{eq}T} = -\frac{f_0^2}{\sqrt{E_{eq}\rho_{eq}}} \qquad (4)$$

The substantial difference between Eq. (3) and Eq. (4) is that for CMRs the resonance frequency, f_0, and the thickness,

T, are two independent variables while for FBARs and QCMs they are intrinsically coupled and cannot be set independently from one another. This is an important and unique advantage of CMR-S. In fact, it permits to set the frequency of operation of these devices according to the specifications of the desired application and independently obtain the required value of mass sensitivity by scaling the device thickness.

In addition, for FBARs and QCMs an increase in the mass sensitivity, due to an increase in the frequency of operation, does not translate in an improvement of the overall sensor performance. In fact, the minimum detectable frequency shift is intrinsically related to the frequency stability of the device, hence to the inverse of its quality factor, Q [10]. Since FBARs and QCMs have shown a constant $f_0 \cdot Q$ product, an increase in sensitivity, due to an increase in the resonance frequency, is accompanied by a reduction in Q, hence rendering unaltered the minimum detectable mass.

On the contrary, for CMR-S, once the desired frequency of operation, f_0, has been set, the performance of the sensor can be improved by reducing the thickness, T, of the AlN layer up to the limit for which a high Q is preserved. For example, Q comparable to thicker films and in the order of 1,000 has been attained for the ultra-thin device of this work.

According to this analysis the frequency of operation of the CMR-S presented in this work was set to 178 MHz (W=20 μm) and the thickness of the AlN layer was scaled in the nano domain (T= 250 nm) in order to simultaneously improve the sensitivity and limit of detection of the resonant sensor.

In order to test the CMR as a gas sensor the top metal electrode was made out of gold, given its known ability to adsorb organic molecules in vapor phase [11].

III. FABRICATION PROCESS

A 4 mask fabrication process (Fig. 2) was employed to fabricate the devices presented in this work.

The ultra-thin (250 nm) AlN film was sputtered deposited and its quality was optimized to achieve rocking curve values as good as 2.1° (equivalent to what has been obtained in micron-size devices). The optimization was conducted in collaboration with Tegal Corporation.

The frequency setting width, W, of the sub-resonators forming the electromechanical structure (Fig. 1) was set to 20 μm (metal electrode coverage of 75%) in order to achieve a resonance frequency of approximately 178 MHz. The number, n, of mechanically coupled sub-resonators was set to 3 and the device length, L, was fixed to 200 μm.

IV. EXPERIMENTAL RESULTS

The electrical response of the fabricated ultra-thin-film AlN CMR was characterized in an RF probe station and the admittance curve measured by an Agilent® N5230A Network Analyzer after performing a short-open-load (SOL) calibration on a reference substrate.

The measured electrical response of the device was fitted to the Butterworth van Dyke (BVD) equivalent electrical circuit (Fig. 3), with the addition of a series resistance, R_s, to take into account the losses introduced by the thin and long metallization layers and vias. Both the experimental and fitted admittance curves of the device are shown in Figure 3.
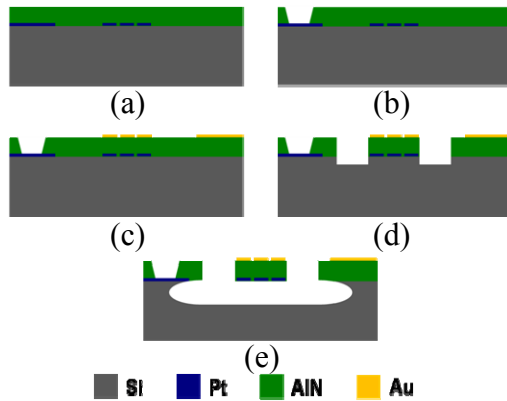

Figure 2. 4 mask fabrication process: (a) sputter deposition of Pt (50 nm) on a high resistivity Si substrate and sputter deposition of the ultra-thin (250 nm) AlN film; (b) open vias in AlN to access the bottom electrode; (c) patterning of top Au electrode (60 nm) by optical lithography and lift-off; (d) dry etching of AlN in Cl_2-based chemestry; (e). XeF_2 dry release of the AlN resonator.

The surface of the top gold electrode was cleaned using O_2 plasma before exposing the resonator to the gas adsorption process. The sensitivity to mass loading of the fabricated device was verified by exposing it to different concentrations of methanol. The steady state response of the sensor was characterized by measuring the resonator admittance curve with a network analyzer after 30s exposure to the methanol gas. Also using the same network analyzer, the transient response of the sensor was measured by exciting the resonator at a single fixed frequency (178.54 MHz) and monitoring the variation over time of its admittance magnitude due to the adsorption of methanol. The measurement setup employed for both the steady state and transient tests is described in more details in the following section.
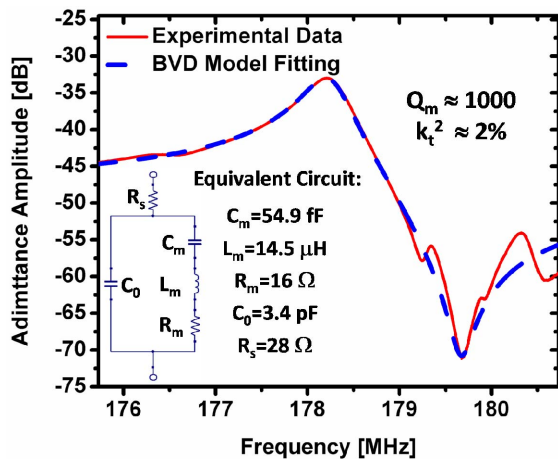

Figure 3.   Experimental and fitted (BVD model with R_s) admittance curves of the fabricated ultra-thin-film AlN CMR. The relativey high value of the resintance, R_s, is due to the high resisitvity introduced by the thin and long Pt bottom electrode. The extracted value of the device mechanical quality factor, Q_m, does not include the losses in the series resistance, R_s.

A. Measurement Setup

The die was attached to a custom designed PCB provided with on-board calibration standards and the resonator was wirebonded to a 50 Ω coplanar waveguide (CPW) line. As shown in Fig. 4, the PCB itself served as a lid for the testing chamber. The methanol vapor was generated by a bubbling method using argon as carrier. Different concentrations of methanol were produced by mixing methanol (at its saturated vapor pressure) and argon flows. The analyte-induced variation of the resonator admittance was monitored by an Agilent® N5230A network analyzer.
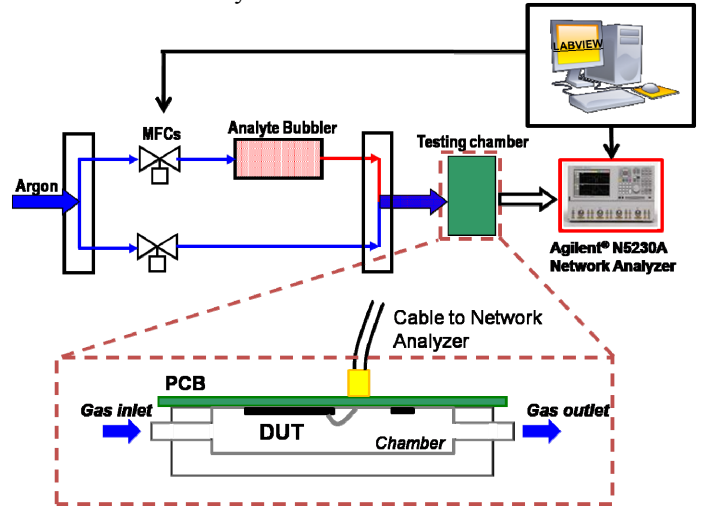

Figure 4.   Measurement setup: the main argon line is splitted in two indipendently controlled flows. Only one of them carries the analyte (saturated vapor pressure of methanol). The two flows are recombined before being injected inside the testing chamber. The total flow is always kept constant at 1,000 sccm, whereas the methanol flow is modulated by a mass flow controller and regulated to vary between 50 and 500 sccm.

B. Steady State Sensor Response

The resonator response over frequency was measured before and after 30 s exposure to methanol. Both measured curves were fitted to the BVD model in order to extract the percentage variation of motional inductance, $\Delta L/L_0$, due to the adsorption of methanol mass, Δm, on the top gold electrode. Since $\Delta m/m_0 = \Delta L/L_0$, the adsorbed mass, Δm, for a given analyte concentration can be extracted by multiplying the measured ratio by the modal mass of the device, m_o (proportional to the product of the geometrical dimensions and the mass density of the device). The accuracy of the calculated values for the equivalent mass density, ρ_{eq}, and Young's modulus, E_{eq} of the resonator using the equations in [7], was confirmed by the measurement of the device resonance frequency (178.23 MHz), which closely matches the analytical value (175.64 MHz) calculated according to Eq. (1). An error of only 1.5% was found between the experimental and analytical values of the resonance frequency and proves that a relatively accurate estimation of the device mechanical parameters was attained.

The resonance frequencies of the two extracted BVD circuits, before and after exposure to methanol, were compared and the calculated frequency shift was divided by the extracted value of adsorbed mass per unit area in order to calculate the

device mass sensitivity. The mass sensitivity per unit area was found to be 38.96 KHz·μm^2/fg. This value is in very good agreement with the one calculated analytically, 38.93 KHz·μm^2/fg, according to Eq. (3).

C. Transient Sensor Response

The transient response of the sensor was measured by exciting the resonator at a single frequency, f_c, and monitoring the variation over time of the resonator equivalent admittance amplitude. The excitation frequency, $f_c=178.54\ MHz$, was choosen to be in the inductive region of the resonator admittance curve, between the series and parallel resonances, where the slope of the magnitude of the admittance curve versus frequency is maximum (Fig. 3). In this interval of frequencies, the resonance frequency shift induced by mass loading is associated with the greatest variation in the resonator admittance amplitude. The measured transient response of the sensor exposed to different concentrations of methanol is shown in Fig. 5.
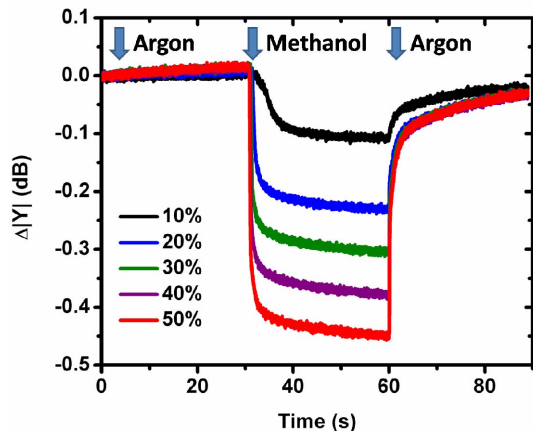

Figure 5. Transient response of the 178 MHz CMR-S exposed to different concentrations (10% to 50% vapor pressure) of methanol. The sensor reached 75% recovery in 2 s and a noise floor of approximately ±0.005 dB was recorded.

Thanks to our prior knowledge of the resonator equivalent electrical parameters (Fig. 3), it was possible to relate the measured vaiation in the magnitude of the admittance, $|\Delta Y|$, to the percentage variation in the device equivalent motional inducatnce, $\Delta L/L_0$, hence, the adsorbed mass, Δm (Fig. 6).

The minimum detected value of adsorbed mass per unit area was 51.2 ag/μm^2, which corresponded to a saturated vapor pressure of methanol of 5 % (the minimum concentration allowed by the existing measurent setup). Considering a measured noise floor of approximately ±0.005 dB (Fig. 5) and a minimum detectable admitance shift of approximetley 0.015 dB, a limit of deteciton of approximately 15.7 ag/μm^2 can be correspondigly estimated for this specific device and setup.

V. CONCLUSION

In this paper, the design, fabrication and testing of ultra-thin-film (250 nm) AlN contour-mode resonant sensors was demonstrated. The unique ability of CMR-S to increase the mass sensitivity by scaling the device thickness while keeping constant the frequency of operation was experimentally demonstrated. The sensing properties of the device were verified by exposing it to varying concentrations of methanol. A value of mass sensitivity as high as 38.96 KHz·μm^2/fg was achieved at a frequency of operation of approximately 178 MHz and values of adsorbed mass per unit area as low as 51.2 ag/μm^2 were recorded. The device analysis and the experimental data show the extraordinary potential of CMR-S for the demonstration of compact, low cost and high performance sensor platforms for multiple VOC detection.
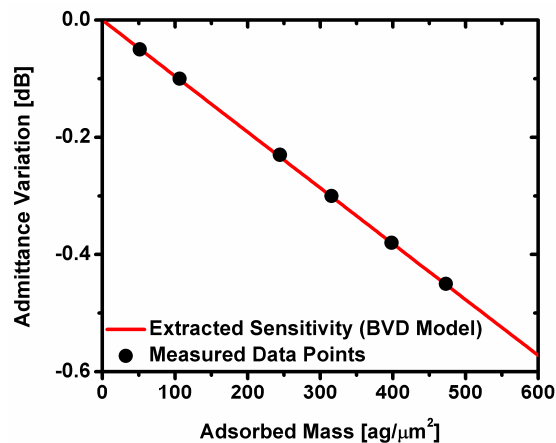

Figure 6. Sensitivity of the admittance magnitude to mass adsorption.

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