

Nanoscaled Piezoelectric Aluminum Nitride Contour-Mode Resonant Sensors

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Abstract—This paper reports on a new class of nanoscaled piezoelectric aluminum nitride contour-mode resonant sensors (CMR-S) that have been developed for the gravimetric detection of volatile organic chemicals (VOC). The use of the CMR-S and its scaling for the making of large arrays of detectors is justified in terms of the superior sensitivity and limit of detection (LOD $\sim \text{zg}/\mu\text{m}^2$) that this technology attains with respect to any other available acoustic device. The choice of a novel functionalization layer based on ss-DNA is introduced as an effective way to selectively detect multiple VOCs without altering the electromechanical characteristics of the resonator. Experimental results confirming the advantages of scaling the device dimensions and its frequency of operation in terms of improved LOD and measurement speed are presented. Furthermore, preliminary data showing selective detection of dimethyl-methylphosphonate (DMMP) and dinitrofluorene (DNF) (with LOD in the order of ppt) are reported.

I. INTRODUCTION

Acoustic resonators are highly sought as sensor transducers because of the high accuracy with which their output variable (frequency) can be measured with simple and portable electronics. The miniaturized nature and integrability with Complementary Metal Oxide Semiconductor (CMOS) electronics of NanoElectroMechanical Systems (NEMS) resonators renders them more attractive than commercially available gravimetric sensors such as Quartz Crystals Microbalances (QCM) [1, 2] and Surface Acoustic Wave (SAW) [3] devices for the manufacturing of large arrays of gas detectors.

Because of these reasons, significant efforts in the research community have been dedicated to the miniaturization of micro scale devices to attain limits of detection (LOD) for gas concentrations in the order of parts per trillion (ppt). Such low levels of LOD in a very compact form factor device will not only improve the performance of existing detectors, and provide for higher degree of safety in case of hazardous gas monitoring, but will also enable the deployment of new and portable sensors that are easily incorporated in wireless devices and sensor networks for distributed environmental or personal health monitoring.

Although scaling to smaller dimensions inevitably yields a lower device mass and higher sensitivity, these enhancements are in most cases offset by the reduced stability and power handling of the nanomechanical device, which result in a limited gain in LOD [4]. Gas sensor performance should ultimately be evaluated in terms of LOD (for gas concentrations and not absolute mass), which depends on both device sensitivity as well as signal to noise ratio. Although sensitivity is influenced exclusively by the mass and frequency of operation of the resonator, the minimum measurable frequency shift is a function of the phase noise of the acoustic oscillator and (especially if interested in real-time phenomena in the msec time frame) by the power handling and the dimensions of the mechanical device. Nanoscale beams tend to exhibit low power handling and therefore worse phase noise.

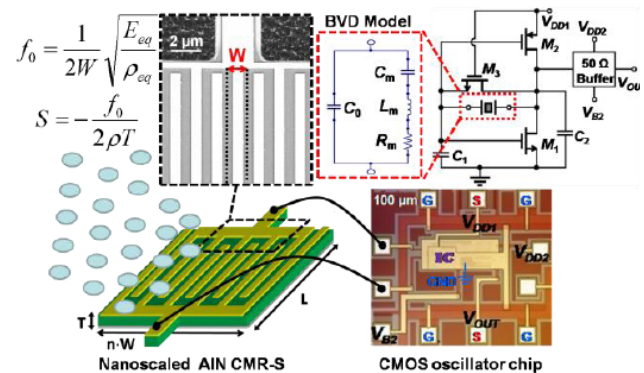


Figure 1. Schematic representation of the fabricated nanoscaled AIN CMR-S connected to a self-sustained CMOS oscillator circuit. A number, n , of subresonators (width, W) are mechanically coupled in order to excite a higher order width-extensional mode of vibration in the AIN plate (f_0 is set by W). The butterworth van dyke (BVD) model is used to describe the resonator behavior.

According to these considerations, it should be clear that sensor sensitivity and power handling should be simultaneously taken into consideration when scaling the resonator. In addition, when devising chemical sensors, the

attention should be focused on the detection of mass per unit area (*i.e.* concentration) rather than absolute mass and therefore only some of the device geometrical parameters should be scaled instead of adopting a uniform scaling strategy of all dimensions as it is done for beams. As a general guideline, optimal sensor performance is attained by synthesizing a transducer that occupies a large area and is very thin. Suspended membranes with thickness in the nanometer range are therefore desirable. Flexural plates (instead of beams) are a good compromise [5, 6], but higher frequencies and power handling are achieved in bulk mode acoustic resonators [7-10]. Laterally vibrating aluminum nitride contour-mode resonators [11-13] (Fig. 1) belong to this category and represent one of the most suitable technologies to devise high performance sensors.

This work will highlight the advantages of scaling in frequency and thickness of the contour-mode resonant sensor (CMR-S) technology in terms of enhanced sensitivity, lower LOD and faster measurement speed, and present experimental results showing that ppt limits are feasible with this solution.

Beyond the transducer, another equally important aspect in the design of VOC detectors consists in the selection of the chemically interactive material (CIM). The CIM needs to have a minimum impact on the transducer operation, maximize the selectivity between different gas species and be characterized by a short refresh time (time required to return to the measurement baseline after the adsorption-desorption process is completed). Very selective polymer layers have been identified [14-16], but each requires a different chemistry and a different method of deposition on the device surface. In the attempt of using a single chemistry and still provide a good degree of selectivity without impacting the device performance, single-stranded DNA (ss-DNA) has been evaluated as a functionalization layer for the CMR-S technology. Initial experimental results show that some degree of selectivity is possible by changing the ss-DNA sequence without impacting the performance of the individual device.

II. NANOSCALE ALN CONTOUR-MODE RESONANT SENSOR

A. AlN Contour-Mode Resonator Technology

The contour-mode resonator is formed by an AlN film sandwiched between two metal electrodes and is excited into a primarily width-extensional (in-plane) vibration by the equivalent e_{31} piezoelectric coefficient. The center frequency of the CMR is set lithographically by the width of the electrodes (Fig. 1) and multiple frequencies are attained on the same silicon chip [13]. Several of these unitary cells of width, W (known as fingers), are arrayed together and excited in an alternating fashion (two adjacent fingers are excited 180° out of phase with respect to each other) so as to form an equivalent symmetric lamb wave [17-19] in the AlN plate. The number of fingers, their length (also known as aperture of the transducer) and the film thickness are used to set the equivalent impedance of the device. Quality factors, Q , ranging between 1,000 and 3,000 in air have been demonstrated [20]. A maximum of 2.2 % has been experimentally recorded for the electromechanical coupling, k_t^2 , of the AlN resonant transducer [21]. The device is easily fabricated with CMOS-compatible manufacturing steps and

requires 3-4 masks to pattern the electrodes, the AlN active area, access the bottom electrode and release the resonator [11]. For sensing applications a top gold electrode is generally used given the simplicity with which its surface is functionalized with ss-DNA.

TABLE I. MASS PER UNIT AREA SENSITIVITY EQUATION FOR DIFFERENT MODES OF ACOUSTIC VIBRATION

Mode of Vibration	Frequency Equation	Sensitivity
Flexural	$f_0 \propto \frac{T}{L^2} \sqrt{\frac{E}{\rho}}$	$S \propto \frac{f_0}{\rho T} \propto \frac{1}{L^2}$
Shear	$f_0 \propto \frac{1}{2T} \sqrt{\frac{G}{\rho}}$	$S \propto \frac{f_0}{\rho T} \propto \frac{1}{T^2}$
Thickness Extensional	$f_0 \propto \frac{1}{2T} \sqrt{\frac{E}{\rho}}$	$S \propto \frac{f_0}{\rho T} \propto \frac{1}{T^2}$
Contour	$f_0 \propto \frac{1}{2W} \sqrt{\frac{E}{\rho}}$	$S \propto \frac{f_0}{\rho T} \propto \frac{1}{WT}$

B. AlN Contour-Mode Resonant Sensor Design

When designing a chemical sensor the key important parameters to take into account are the device sensitivity to gas concentrations and its corresponding limit of detection (without considering the effect of the CIM). According to these specifications, the adoption of the AlN CMR technology for sensing application is advantageous over several other acoustic resonators. As shown in Table I, the AlN CMR-S is the only device for which sensitivity can be set independently of frequency by varying the AlN film thickness. This feature offers a degree of flexibility in the sensor design when operation is constrained to a particular frequency dictated by the application requirements such as in wireless or radio frequency identification (RFID) components.

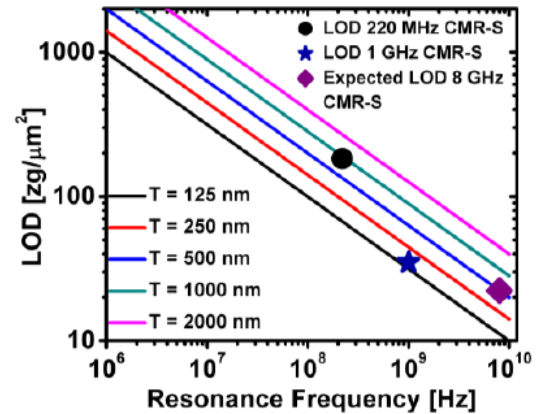


Figure 2. Limit of detection as function of the CMR-S resonance frequency and thickness. The device surface area is assumed to be constant over frequency and the input power is assumed to be the device critical power (maximum power before non-linear behavior). The measured data points follow the predicted trend (LOD for devices at 220 MHz and 1 GHz have been experimentally measured).

In addition, assuming that the thermomechanical noise of the resonator limits the oscillator phase noise (true for fast

real-time measurements in which the Allan variance has a $1/\tau$ behavior) it is possible to show that scaling the CMR-S thickness and increasing the frequency of operation yields a better LOD for a given measurement bandwidth (Fig. 2) or a faster measurement speed for a given LOD (Fig. 3). This is a consequence of the fact that the CMR power handling per unit area increases with frequency [22] and sensitivity improves by reducing the film thickness. Therefore device scaling in terms of electrode width and film thickness ultimately yields a sensor with a better LOD for gas concentrations (or mass per unit area). Functional AlN films as thin as 50 and 100 nm [23-27] have been demonstrated and are the target thickness for the making of the CMR-S. In this paper 250 nm thick devices are presented.

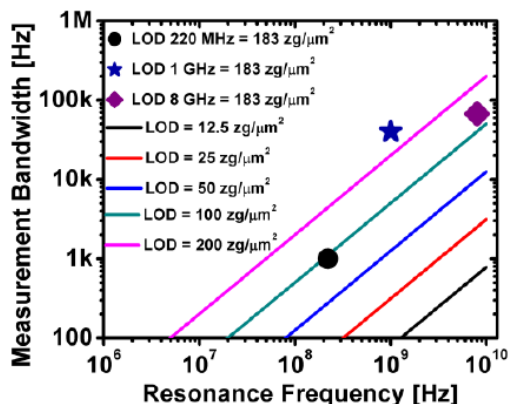


Figure 3. Measurement bandwidth (i.e. speed) as a function of the CMR-S resonance frequency for a given LOD. Note that the experimental data for the LOD (available only for the 220 MHz and 1 GHz device; predictions are made for the 8 GHz device) are in line with theoretical predictions. The device surface area is assumed to be constant over frequency and the input power is assumed to be the device critical power (maximum power before non-linear behavior).

C. AlN CMR-S Oscillator Design

An important component in the deployment of a resonant sensor consists in the electronics required to drive the acoustic sensor and generate a stable frequency signal. The simplest electronic method to measure frequency variations induced by the mass adsorption process is the insertion of the resonator into an oscillator such as a Pierce circuit (Fig. 1). The detailed design of a Pierce oscillator using AlN CMRs can be found in [21]. It is important to note that, although almost any mechanical resonator can be inserted into an oscillator loop, the designer should have the goal of minimizing power consumption and reducing phase noise. For a given CMOS technology, these two metrics are mainly affected by the resonator characteristics and in particular the k_t^2 - Q product (known as the figure of merit) of the acoustic device. It is therefore essential that this parameter is monitored when scaling the device into the nano realm. The need for a high and constant k_t^2 - Q justifies the use of AlN piezoelectric films for the transduction of the resonator, which have shown to maintain high Q and high coupling even when the film thickness is scaled to sub-micron dimensions [28]. This is another advantage of the AlN CMR-S technology over other solutions based on electrostatically-transduced devices [10],

whose coupling generally degrades with the frequency of operation.

III. CHEMICALLY INTERACTIVE MATERIAL

The CIM has an important role in the making of a VOC sensor. The CIM ultimately influences the LOD, sets the device selectivity and the response time between multiple measurements. In terms of LOD, at least, the CIM should not affect the electromechanical characteristics of the acoustic sensor so that neither the device sensitivity nor the power handling capabilities are altered. AlN CMR-S are very stiff structures and any thin CIM (of thickness less than the AlN film) will minimally impact the resonator performance. Nonetheless, the need for scaling and the use of AlN films in the 50-250 nm thickness range exclude the utilization of thick, but generally very selective, polymers. CIMs with a high affinity to specific VOCs will also help enhancing the device LOD.

In terms of selectivity, the availability of a large number of different functionalization layers is desired for the synthesis of a sensor array. It would be ideal to identify a family of CIMs that are based on the same chemistry so that the manufacturing complexity of the sensor array is drastically reduced (no need to use multiple steps or tools for the device functionalization), but still a certain degree of differentiation in the affinity to various gas species is provided. The advantage of using small size devices mainly consists in the fact that a large array (> 100) of resonators can be packed in a very small surface area, approximately 1,000 times less than what is conventionally occupied by an array of 8 QCMs [2]. A large array makes possible the utilization of redundancy as an additional parameter in setting the sensor selectivity, therefore reducing the requirements on the CIM. For these reasons, although not likely to be one of the most selective functionalizations, ss-DNA has been selected for the demonstration of AlN CMR-S for VOCs detection. Previous work in Johnson's laboratory [29] with Field Effect Transistors (FET) formed by carbon nanotubes (CNTs) functionalized by ss-DNA has shown that affinity to specific VOCs is controlled by the DNA sequence. Given the practically infinite number of DNA sequences that can be synthesized, it is feasible to hypothesize the use of ss-DNA to functionalize a large array of sensors. These different strands of DNA could also be deposited on the CMR-S simultaneously via automated printing.

The use of CNTs as adhesion layer for the attachment of two strands of ss-DNA to CMR-S has been investigated [30, 31] and has shown promising results in terms of selectivity. Currently, the direct attachment of the DNA strands to the top gold electrode of the resonator is being explored as a simpler and more manufacturable technique. In this paper, the use of thiolated single stranded DNA (HS-ss-DNA) is presented. The HS-ss-DNA sequence (Thiol - 5' CTT CTG TCT TGA TGT TTG TCA AAC 3' obtained from Invitrogen) is dispensed on the resonator by micropipetting a 2 μ L drop of 100 μ M DNA in 1 M Potassium Phosphate monobasic buffer solution. The drop is let rest for 45 minutes in a humid environment and dried in a nitrogen stream. Automated methods based on the use of small-drop size printers for covering large arrays are under development.

In terms of refresh time the CIM should quickly release the adsorbed molecules so that subsequent measurements can be rapidly taken. A large surface to volume ratio for the CIM is desirable so that gas molecules do not remain trapped in the bulk of the material. ss-DNA offers this capability since it practically forms a very thin (few nm) equivalent layer. Refresh times in the order of few 10s of seconds have been recorded [31] when the sensor is exposed to a flow of pure nitrogen. It is important to note that a fast refresh time is generally associated with a poor level of affinity to selected VOCs and a compromise between refresh speed and selectivity has to be made.

Although preliminary tests are showing that ss-DNA attached to the CNT FET devices survives and shows similar responses after a period of 1 month, the long term reliability of the selected CIM is still under investigation.

IV. EXPERIMENTAL RESULTS

A. Scaling Advantages

250 nm thick AlN CMR-S operating at frequencies ranging between 220 MHz and 1 GHz have been fabricated and inserted into a Pierce oscillator to monitor their short frequency stability and ultimate experimental LOD without any functionalization layer. Fig. 4 compares the noise equivalent minimum detectable mass per unit area for different measurement times (extracted from the measured Allan deviation and sensor sensitivity) for a 220 and a 905 MHz oscillator and experimentally confirms that operation at higher frequencies effectively improves the device LOD as predicted by the design equations. The oscillator core consumes an equivalent power in the order of 0.5 mW for the 220 MHz device and 2 mW for the 905 MHz one. Although the two devices have slightly different area (the 220 MHz is $200 \times 45 \mu\text{m}$, whereas the 905 MHz is $80 \times 60 \mu\text{m}$) and Q (1,400 for the 220 MHz and 760 for the 905 MHz), the effective improvement in LOD can be explained by the increased sensitivity and power handling of the resonant device. It is important to note that such devices exhibit unprecedented LOD in the order of few 100s of $\text{zg}/\mu\text{m}^2$ even without a functionalization layer.

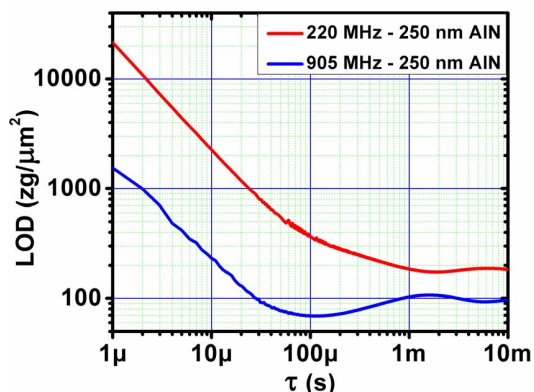


Figure 4. Comparison of minimum detectable mass per unit area (noise equivalent) for a 220 and 905 MHz oscillator for different measurement integration times. Note that a lower LOD is achieved with faster measurements at higher frequencies.

Similarly, the experimental data confirm that the measurement speed can be increased for a given LOD by raising the device frequency of operation. Depending on the application, continuous and rapid monitoring of the events might be required. In addition, a fast measurement time is desired if a large array of resonators is addressed in a time-multiplexed fashion. In order for each resonator to monitor the same event, the data acquisition for each device should occur at a speed of at least 100-1,000 times faster than the measured event so that the entire array can be sampled within the duration of the event.

B. Volatile Organic Chemical Detection

In order to devise a deployable sensor array, the top Au surface of a 220 MHz CMR-S was functionalized by a thiolated ss-DNA to test its impact on the acoustic device performance and screen its use for the selective detection of multiple VOCs.

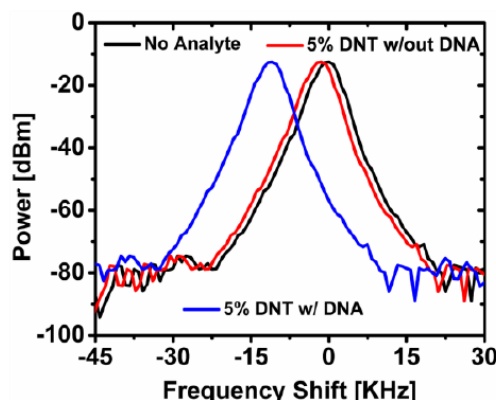


Figure 5. Sensor response to 5% DNT vapor pressure before and after DNA functionalization.

As shown in Fig. 5 the addition of the ss-DNA does not alter the oscillator response (same output power and phase noise), but positively impacts the device performance by significantly enhancing the sensitivity of the non-functionalized CMR-S to DNT.

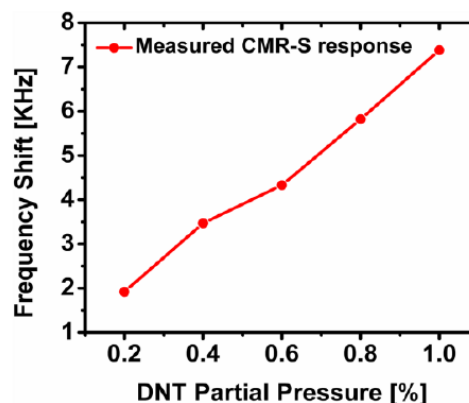


Figure 6. Response of the nanoscaled 220 MHz CMR-S oscillator for different concentrations of DNT ranging between 0.2% and 1% of the saturated vapor pressure.

A 6 fold increment in the device sensitivity is recorded and is a sign of the improved adsorption affinity between the selected strand of DNA and DNT. A test of the same ss-DNA with DMMP (a nerve agent simulant) has yielded an even higher level of enhancement (about 10 X) with respect to DNT. This is an indication that some level of selectivity is achievable and a large ss-DNA library could be created for the detection of multiple VOCs. Currently, there is no way to a priori predict the affinity of a DNA strand to a specific gas and such library will have to be built by means of experimental trials.

The 220 MHz CMR-S was also capable of detecting the minimum concentration of DNT that could be produced with the currently available testing setup. As shown in Fig. 6 the CMR-S measures a shift of 2 KHz, which corresponds to an equivalent concentration of DNT of 1.5 part per billion (ppb). Measurement of the Allan deviation for the same functionalized CMR-S oscillator shows that a noise equivalent concentration of 15 ppt of DNT can be detected, effectively setting the LOD for this device well into the parts per trillion. These data clearly show that the AlN CMR-S technology is a very promising solution for the implementation of sensor arrays with very low LOD.

V. CONCLUSION

The aluminum nitride piezoelectric CMR-S has been presented as a viable technology for the making of large arrays of VOC detectors. Scaling of device thickness and frequency of operation are shown to be the two most important parameters a designer can act on in order to improve the device LOD and measurement speed. These predictions have been experimentally validated and the CMR-Ss have shown LOD in the order of $\text{zg}/\mu\text{m}^2$ even without a functionalization layer.

The use of ss-DNA as a CIM to devise a selective array of sensors has been explained in terms of limited impact of the DNA strand on the acoustic resonator, enhanced LOD, fast refresh time, and simple access to a chemistry with different gas affinity. Preliminary experimental results on devices functionalized directly by HS-ss-DNA on the resonator top Au electrode have shown up to 10 times enhancement in the device sensitivity and some degree of selectivity between DMMP and DNT.

Current work is focused on further miniaturizing the resonator technology in order to improve the sensor LOD and power consumption. At the same time, the demonstration of large arrays of AlN CMR-S functionalized by several different DNA strands and integrated with multiplexed oscillators is being pursued.

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