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System on chip mass sensor based on polysilicon cantilevers arrays for multiple detection

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Abstract

In this work, we present an on chip microelectromechanical system based on an array of cantilevers for mass detection. The sensor transducer, using polysilicon as structural layer, has been integrated monolithically with the CMOS circuitry. Arrays of four and eight resonant cantilevers excited and detected electrically through the integrated circuit have been fabricated. Submicrometric dimensions of the integrated cantilevers allow obtaining a mass sensitivity of 28 Hz/fg being the expected mass resolution in vacuum in the femtogram range. In this paper, we present the MEMS fabrication process, some examples of the integrated cantilevers arrays and the electrical test of the on chip MEMS system to characterize the performance of the system for mass sensing purposes.

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1. Introduction

For biochemical and medical applications and, in general, for nanoscience and nanotechnology applications there is a strong demand for very sensitive mass sensors, being the target a sensor for single molecule detection. The desired mass resolution for very few or even single molecule detection, has to be below the attogram range (for instance, the haemoglobin protein mass is only 0.109 ag and constitutes a medium size biomolecule). One of the strong candidates as a transducer for this kind of sensors is the one based on Micro/Nano-electromechanical systems (MEMS/NEMS) [1]: very small resonating structures with dimensions below the micrometer and high resonance frequencies. These portable sensors formed by a MEMS/NEMS with high mass resolution allow on line measurements, providing faster and accurate in situ registration. With all these characteristics, these sensors improve the complex detections obtained by traditional chemical analyses in laboratories [2,3]. Additionally, one of the advantages of these MEMS/NEMS devices is their capability for being fully integrated [4]. These on chip transducers based on MEMS/NEMS devices, fabricated using standard microelectronic technologies and integrated monolithically with the CMOS conditioning circuitry, allow parallel fabrication, small and portable devices and high sensitivity, reducing at the same time the overall cost and providing more versatility to the system. For all these reasons, a major effort is being taken on the application of MEMS/NEMS to develop sensors for high mass resolution detection.

The actual main commercial mass sensors are the quartzcrystal-microbalances (QCM), which are based upon the acoustic vibratory modes of quartz crystals. The mass sensitivity achieved by QCM are in the range of ng/cm² [5], detecting the frequency shift when an added mass is deposited over the

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surface electrode. The QCM provides very high resolution for a distributed mass measurements: monolayers of deposited materials and studies for very thin films depositions or reactions have been done [6,7], but the final mass resolution is below the nanogram. Recently, different transducers based on micro or nano-electromechanical devices are being reported for higher mass resolutions. Micro-cantilevers as transducers for mass or chemical sensing have been widely used for different groups and very high mass resolutions and promising applications have been obtained [8-17]. Resonant cantilevers operating as microbalances, in which a frequency shift is detected as a consequence of the mass change, have been used obtaining mass sensitivities in the Hz/ag range and mass resolutions in the sub-picogram or femtogram range [8-13]. Also static cantilevers in which a deflection is produced due to the surface stress derived from the adhesion of the target molecules onto the specific functionalized sensor surface, has been reported with very high achieved resolutions [14]. Other kind of transducers not based on cantilevers, have also been reported as very sensitive sensors for biological applications. For instance, Nicu et al. [18] present a matrix of micromachined piezoelectric membranes achieving mass sensitivities above Hz/pg operating the system in liquid. The last examples we want to mention as a very promising mass transducers, are based on the use of carbon nanotubes (CBN) [19,20]. Due to the mechanical properties of the CBNs, very high mass sensitivities (in the range of zeptogram) can be achieved [19]. Experimental evidence of mass measuring with a CBN is reported in ref. [19]. In this reference, a deposited mass below the fg is measured using a SEM microscope for the evaluation of the oscillation amplitude of the nanotube, which complicates the experimental set-up. Other detection techniques for CBN transducers based on electrical actuation and detection has been also reported [20], opening new possibilities for the application of the CBN for more practical applications. Nevertheless, the difficulty in the CBN manipulation complicates the fabrication process of the transducer; making very challenging the development of a final sensor. For the moment, the most sensitive systems with mass sensitivity better than Hz/ag has been reported by Ekinci et al. [15] and by Ilic et al. [16]. In ref. [15], using a mechanical transducer based on a resonating clamped-clamped cantilever they are able to achieve a mass resolution of attograms. In the case of [16] using resonating cantilevers with a paddle configuration, a minimum resolvable mass below the attogram is reported. From these last references, it seems that the cantilever structure for the transducer is widely accepted as one of the best transducer for high mass resolution measurements and sensory applications.

In this paper, we propose a microelectromechanical system (MEMS) for mass detection based on an array of resonating cantilevers able to work under ambient or vacuum conditions and fully integrated onto a CMOS circuit. Implementing an array of cantilevers, multiple sensing or redundancies for the same measurement are possible increasing the versatility of the mass sensor. For instance, an array with different length cantilevers will allow sensors with different mass sensitivities or on the contrary, having an array of exactly equal cantilevers will

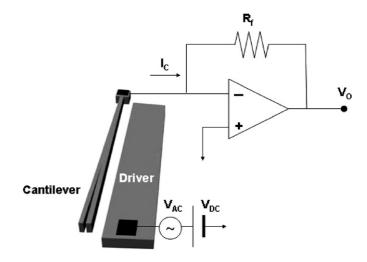


Fig. 1. Schematic draw of the sensor system. Resonance of the cantilever is achieved by electrical excitation through a polarization driver. Resonance is measured by detection of the displacement current between cantilever and driver.

provide sensors with redundancy or differential measurements (one cantilever used as reference and the others used as sense electrodes).

In the system not only a more versatile sensor will be implemented but also very high mass sensitivity will be provided. In this case, an array of cantilevers working in vacuum can be applied advantageously for the detection of small flux of atoms or molecules, mapping the spatial distribution of molecules or atoms in evaporation systems (metal, molecular, MBE). In our array of cantilevers, we have spatially distributed cantilevers with an individual high mass resolution, which can facilitate the mapping of the beam.

The designed sensor is formed by an array of four and eight submicron scale laterally oscillating cantilevers, which are excited and detected electrostatically. Mass detection is based on monitoring the change on the resonance frequency for each cantilever independently. The electrical excitation of each cantilever up to resonance and the electrical detection of the resonance allow system on chip integration, as we have already demonstrated in previous works [12,13]. Lateral cantilever oscillation is achieved by means of electrostatic excitation through a polarization driver. Resonance is measured by detection of the displacement current between the cantilever and a read-out electrode, which can be the same excitation driver or another driver placed close and parallel to the cantilever (Fig. 1). In order to detect an electrical signal a dc bias voltage must be applied between the cantilever and driver. The microresonators are fabricated on polysilicon by a CMOS compatible post-process after the front-end CMOS circuit fabrication. For demonstration purposes, we have used the CMOS-CNM25 2P, 2M technology (2.5 µm with two polysilicon layers and two metal layers [21]).

In the next sections, we present the design and working principle of the device, the fabrication process of the transducers and the results of this fabrication process. Finally, the electrical characterization of the devices and a discussion about the achievable mass resolution is provided.

2. Transducer characteristics and sensor design

As we have already explained in Section 1, cantilevers are one of the best transducers, due to their simplicity, their mechanical characteristics and dynamic behaviour. Cantilevers mechanical behaviour has been deeply studied and it is clearly determined [22,23].

The working principle of resonating cantilevers as mass sensors comes from the dependence of the resonance frequency of the lever with the mass. Considering a mass-spring model for the cantilever movement ($f_0 = 1/2\pi\sqrt{k/m_{\text{eff}}}$), the mass responsivity or mass sensitivity can be expressed as

$$\Re = \frac{\delta f_0}{\delta m_{\rm eff}} = -\frac{f_0}{2m_{\rm eff}} \tag{1}$$

where f_0 is the resonance frequency of the fundamental on plane mode for the cantilever, $m_{\rm eff}$ the effective mass of the cantilever and k is the elastic constant of the cantilever. For this derivation, we are assuming that the deposited mass will only produce a shift on the frequency and will not affect the spring constant. From Eq. (1), the minimum detectable mass (defined as the mass resolution) will depend on the minimum resonance frequency shift measurable on the system and can be expressed by Eq. (2).

$$\delta m = \Re^{-1} \delta f \tag{2}$$

Taking into account the characteristics of the material that forms the cantilever (*E*, Young Modulus and ρ , mass density) and the cantilever dimensions (*t* thickness, *l* length and *w* width), for spring constant and resonance frequency evaluation (Eqs. (3) and (4)), the mass resolution (Eq. (2)) can be expressed as Eq. (5).

$$k = \frac{E}{4} \frac{w^3}{l^3} t \quad (N/m) \tag{3}$$

$$f_0 = \frac{1}{2\pi} \sqrt{\frac{E}{\rho}} \frac{w}{l^2} \quad (\text{Hz})$$
(4)

$$\delta m = -\pi \rho \sqrt{\frac{\rho}{E}} l^3 t \,\delta f \quad (\text{kg}) \tag{5}$$

From Eq. (5), it is clearly deduced, that in order to increase the mass resolution, shorter and thinner cantilevers has to be implemented. On the other hand, shorter cantilevers produce higher resonance frequencies (Eq. (4)), which can be a problem for the specific CMOS on chip read-out circuitry. In the same sense, thinner cantilevers will be very difficult to detect, if electrical excitation and detection is to be used. As a trade-off between these considerations small width cantilevers are required. In our designs and to obtain mass sensitivities in the order of Hz/ag and resonance frequencies in the MHz range, cantilevers having widths smaller than 1 μ m and lengths between 40 and 50 μ m will be defined. Note that in our transducers, the thickness, *t*, of the cantilever is not a design parameter because we are using a polysilicon layer available in the used CMOS technology (Section 3). The thickness of this polysilicon layer is 600 nm.

In order to integrate several cantilevers minimizing area and allowing the CMOS integration, an efficient methodology for the management of the array has to be established. This management of the array for independent control of the cantilevers can be chosen between simultaneously or sequentially cantilevers movement characterization. Both schemes have been implemented depending on the number of cantilevers per array.

The first array implementation is composed by a system with a multiplexing CMOS circuit that allows the individual selection of each cantilever. This configuration is schematized in Fig. 2a, and detailed in Fig. 2c. With this configuration arrays of eight cantilevers are implemented. This configuration allows sequential measurements of each cantilever of the array.

The other possibility is the implementation of several readout circuits, which will allow the simultaneous read-out of several cantilevers. Due to the capacitive detection of the cantilever movement, parasitic capacitance has to be minimized during the design. If several circuits are implemented, distance between cantilever pad and circuit input has to be constant. This assumption is not easy to implement for more than two circuits. Additionally, the use of a read-out circuit for each cantilever becomes unfeasible due to the large dimensions of the circuitry compared with the mechanical structure for an array with multiple cantilevers. For this reason, only arrays of four cantilevers have been implemented connected to two independent read-out circuits. This implementation allows simultaneous measurement of two cantilevers, increasing the accuracy of the device if off-chip differential measurements are done. Fig. 2b shows a schematic draw of a four cantilever array connected to two read-out circuits. In this configuration, the parasitic current can be reduced with the bias polarization scheme shown in Fig. 2d.

The motional current of each cantilever is detected by the integrated read-out circuit. In this work, we have used a transimpedance amplifier based on an operational amplifier with a resistive feedback (as shown in Fig. 1), unlike our last works in which a voltage detection scheme were used [12].

For circuit design, a linear $R_sL_sC_s//C_p$ model of the cantilever-driver system has been used [24]. This model has permitted the simulation of the frequency response of the readout circuit. Adjusting mechanical parameters of the resonator, as Young Modulus, for a polysilicon cantilever with a length of 40 µm, width of 1 µm and a thickness of 600 nm, considering a *Q*-factor of 50; the expected values for the current peak and the resonance frequency of the cantilever are around 20 nA and 900 kHz, respectively.

The main advantage of the transimpedance amplifiers is the fact that the effect of the parasitic capacitance (Cpa, in Fig. 3) is negligible by virtually grounding it through the operational amplifier.

Another requirement for this *I/V* amplifier is to provide enough gain to amplify the low current at the resonance frequency. So, a large feedback resistance is necessary. There are two problems to integrate this large resistance: (i) the area and (ii) the fact that large values of Cpa along with a high value of feedback resistance cause instabilities on the circuit. In addition, any external resistance must be avoided, in order not to increase the overall stray capacitance.

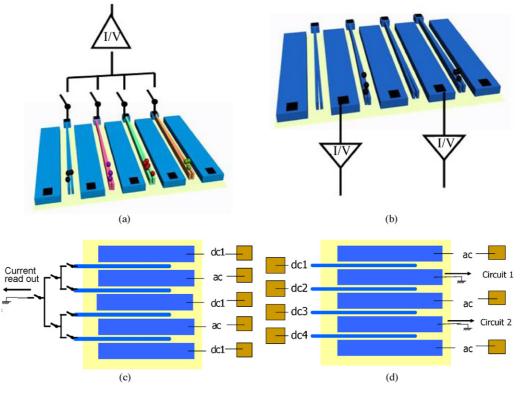


Fig. 2. Schematic drawing of a four cantilever array showing the different possibilities for the management of the cantilevers in the array. (a) Scheme for multiplexing the cantilevers in time. Only one read-out circuitry is needed. (b) Scheme for the simultaneous characterization of two cantilevers. Two read-out circuits are needed. (c) Bias polarization (dc for current detection and ac for cantilever movement excitation) for the multiplexing scheme. (d) Bias polarization for the simultaneous cantilever reading. In this case, the parasitic current due to the ac excitation is diminished.

Fig. 3 shows the design for the transimpedance amplifier. It is based on a T-configuration feedback with a shunt capacitance, C. The transfer function between the output voltage, V_{out} , and the current, i, at the transimpedance amplifier input, in Laplace domain, is given in Eq. (6).

$$\frac{V_{\text{out}}}{i} = \left(R_1 + \frac{R_2}{1 + R_2 Cs}\right) \left(1 + \frac{R_3}{R_4}\right) \tag{6}$$

With this configuration an acceptable gain of almost 2×10^6 V/A is obtained without having to integrate high values of resistance (higher integrated resistance is $175 \text{ k}\Omega$). The function of the shunt capacitance, *C*, is to compensate the effect of the input stray capacitance, Cpa, on the circuit stability. In Fig. 4, the

layouts for the four and eight cantilevers arrays with its read-out and management circuitry are shown.

3. Sensor fabrication process and results

The mechanical transducers are defined as a post CMOS process in specific areas defined during CMOS fabrication. These areas are formed by polysilicon rectangles connected to the circuit by metal pads on top of a silicon oxide layer 1 µm thick. The polysilicon layer with a thickness of 600 nm is the first polysilicon layer of the technology (CMOS CNM25 technology [21]). This layer forms the plate of the CMOS capacitors and will be the structural layer for the cantilevers and drivers. The silicon

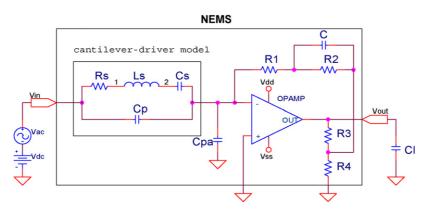


Fig. 3. Schematic diagram of the transimpedance amplifier along with the transducer electrical model.

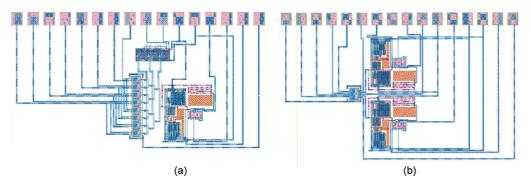


Fig. 4. Layout of the sensors (a) eight cantilevers array with multiplexing system with independent control on each cantilever for sequential detection of the cantilevers array and (b) four cantilever array connected to two circuits that allow simultaneous detection of two cantilevers.

oxide layer used as the field oxide of the MOS transistors is used as the sacrificial layer to release the cantilever. The structures definition is performed by UV lithography. This technique has a limited resolution, but allows a parallel fabrication, reducing the costs. Sensor resolution can be increased by using other lithography methods also compatible with the technology as we have already shown [12,13,25,26].

Fig. 5 shows a schematic view of all the processes after CMOS. During CMOS process the transducer fabrication region is protected with the second polysilicon layer of the technology. In Fig. 5a, a cross section of the fabrication area after CMOS is shown. First step is to remove the protection layer by dry reactive ion etching (Fig. 5b). Fig. 5c–e schematize the UV lithography for the definition of the cantilever-driver structures. The pattern is transferred to the substrate using reactive ion etching (Fig. 5f). After the pattern transfer, the remaining resist is removed (Fig. 5g). The structure is finally released by a wet etching of the silicon oxide, which requires a new photolithographic process for the CMOS protection (Fig. 5h and i).

In the mask definition conservative dimensions have been implemented. Also, due that photolithography is performed on a CMOS processed wafer, highest resolution of the technology is not assured, for these reasons minimum defined width has been 1 μ m. In the eight cantilevers arrays, cantilevers of 40 μ m length and 1 μ m width have been defined. Fig. 6 shows optical images of the transducers integrated monolithically with the CMOS transimpedance amplifier. The multiplexing system, formed by a set of switches in ladder configuration controlled by a digital module is shown also in the figure. A detail of the switches and transducers can be observed in the zoom in of the image.

To analyze precisely the cantilever dimensions scanning electron microscope (SEM) micrographs have been done. Electron radiation can degrade the CMOS circuit electric characteristics. For this reason, in most of the cantilevers, characterization with SEM to determine exactly the dimensions has not been done. Fig. 7 shows an SEM image of one cantilever of the previous array. The final width for the cantilever has been reduced to 700 nm, which is due to the underetching during the reactive

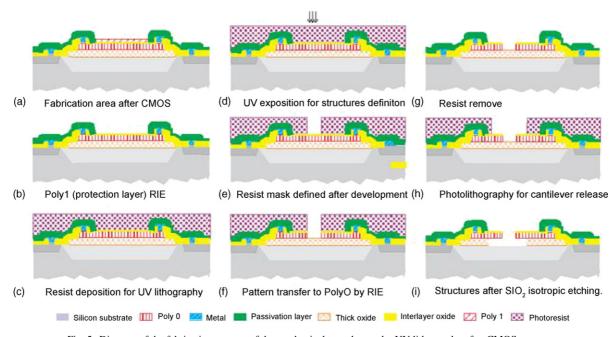


Fig. 5. Diagram of the fabrication process of the mechanical transducers, by UV lithography after CMOS process.

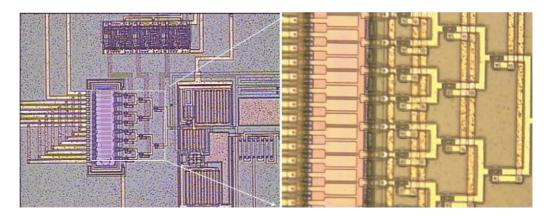


Fig. 6. Optical images of a sensor formed by eight cantilevers array integrated monolithically with the CMOS multiplexing and read-out circuit, right side zoom in of the mechanical transducer.

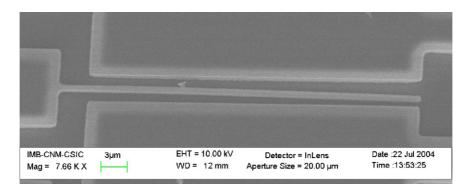


Fig. 7. SEM image of one polysilicon cantilever after the full fabrication process. The free extreme of the cantilever is stuck to the substrate in this case.

dry etching used to transfer the pattern to the polysilicon. The thickness of the polysilicon layer is around 600 nm. One detail that can be observed on this image is that in this case the free end of the cantilever is stuck to the substrate. Sticking problems are relatively common in free standing MEMS, which can be solved using freeze-drying techniques during the releasing of the structures [26] or using specific anti-sticking layers recovering the cantilever surface [27,28]. In our case, in which long and thin cantilevers are made, the problem appears sometimes after wet etching of the sacrificial silicon dioxide layer and sometimes during functional operation of the sensor. For this reason, the anti-sticking layer recovering would be a solution. In this work and as we are using the sensor as a prototype we have not implemented this solution. In fact, we have been using an atomic force microscope (AFM) tip for releasing the cantilevers when it was required as a rapid method for individual cases [29].

For shorter cantilevers, the sticking problem is less important as has been demonstrated in previous works [13,26], in which the freeze-drying technique was enough. Some authors [30] are considering the modification of the resonator shape to avoid sticking problems, which could be also considered in further implementations of the device.

Fig. 8 shows optical images of a four cantilevers array with simultaneous reading using two read-out circuits. In this case, the read-out is performed by the drivers and the cantilever selection is performed by applying or not the ac voltage for its electrostatic excitation according with the scheme shown in Fig. 3c.

4. Electrical characterization of the sensor

To characterize the integrated MEMS system a full electrical test of the on chip resonant cantilevers has been performed.

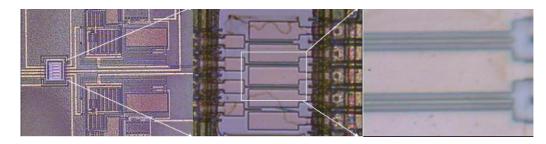


Fig. 8. Optical images of a four cantilevers array connected to two read-out circuit. From left to right zoom in on the mechanical transducers. Cantilevers are 50 μ m long and 1.1 μ m wide.

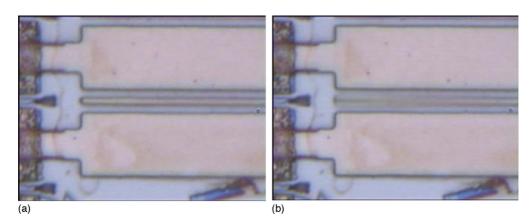


Fig. 9. Optical images of a cantilever 50 µm long, 600 nm thick and 1.1 µm wide (a) out of resonance and (b) at resonance.

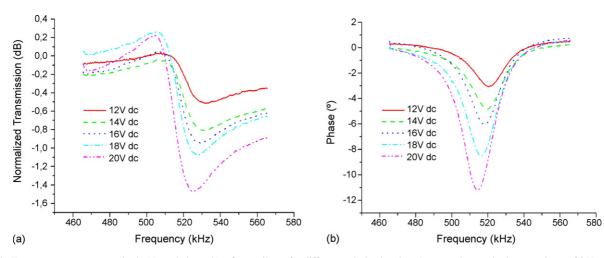


Fig. 10. Frequency response magnitude (a), and phase (b), of a cantilever for different polarization dc voltages and an excitation ac voltage of 3 V peak-to-peak.

The resonant behaviour of the mechanical cantilever has been analyzed as a function of excitation voltages and for different ambient conditions (measurements in air and vacuum).

Electrical characterization of the system has been done using a probe station, several power sources and a network analyzer. Due to the relatively low resonance frequency of the devices (between 350 and 600 kHz, depending on the dimensions) and relatively large dimensions of the cantilevers, it has been possible to observe optically the resonance. First step on the characterization has been to observe optically the resonance. Fig. 9 shows an optical image of the cantilever 50 μ m long, 1.1 μ m wide and 600 nm thick (a) out of the resonance and (b) at resonance.

Applying different polarizations voltages and an ac excitation voltage of 3 V peak to peak, the system frequency response for a single cantilever has been obtained as it is shown in Fig. 10. From this figure, it can be seen the resonance peak and the antiresonance peak due to the parasitic capacitance intrinsic to the system (electrostatic excitation and detection).

From these first measurements, it can be obtained the natural resonance frequency of the cantilever. In particular, from the quadratic dependence of the resonance frequency with the effective applied voltage, as it is shown in Fig. 11, the fundamental resonance frequency at 0 bias voltage is calculated, giving a value of $f_0 = 519.6$ kHz. According to this experimental value,

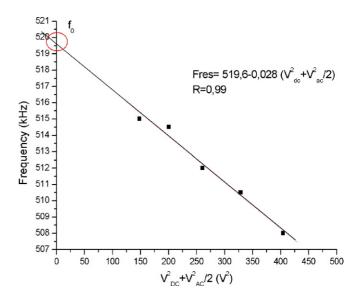


Fig. 11. Experimental dependence of the cantilever resonance frequency with the applied effective voltage. Electrostatic spring softening explains the decrease on the resonance frequency for higher voltages.

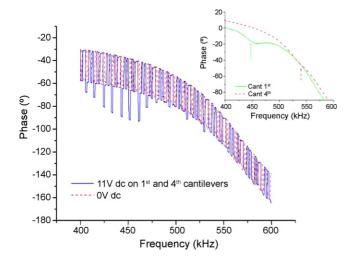


Fig. 12. Frequency response (phase) of the multiplexing of 6 cantilevers of an array of 8. Two of them are resonating, at 450 and 510 kHz, as it can be seen, the other 4 cantilevers has not been exited. In the inset, frequency response for the same resonating cantilevers without multiplexing is shown.

the Young modulus, *E*, for this polysilicon cantilever can be determined (E = 128.3 GPa) which is in the order of the ones reported in the literature [4].

Characterization has been done independently for eight and four cantilever arrays. Eight cantilevers array corresponds to the multiplexed devices. In this case, the objective is to use the sensor for multiple and sequential detection. By multiplexing in time several cantilevers of the array, and extracting afterwards the single dependence of each, sequential multiple detection in very short time can be achieved. Fig. 12 shows the frequency response (in phase) of the on chip MEMS system, when six cantilevers of the array are being multiplexed in time during the frequency sweep. In this case, cantilevers first and fourth multiplexing positions are polarized; cantilever fourth is shorter than cantilever first, so its resonance frequency is higher. In the figure, variation in phase corresponds to the resonance of these two cantilevers, when applying dc voltage. In the inset, the frequency response of these two cantilevers without any multiplexing is shown to prove the system performance.

Frequency response of two cantilevers on a four cantilevers array has been successfully measured simultaneously. Fig. 13 shows the resonance response of two cantilevers excited and detected simultaneously on the same array. In that case, the dc and ac applied voltages were 18 and 9 V peak to peak, respectively. Resonance frequency is at 501 kHz for cantilever 2 (#1) and at 512 kHz for cantilever 3 (#2). The electrical measurement was performed in air conditions. Although dimensions of cantilevers from design and polarization conditions are the same, process mismatch can be the responsible of the 11 kHz difference on the resonant frequency. The mass sensitivity of each of the four-components array cantilevers can be calculated from Eq. (5), obtaining a value of 14 Hz/fg.

The dependence of the Q-factor on the environmental conditions has been studied due to its strong influence on the minimum detectable mass (mass resolution). From Eq. (2), it is easily observed that the mass resolution of the sensor system will

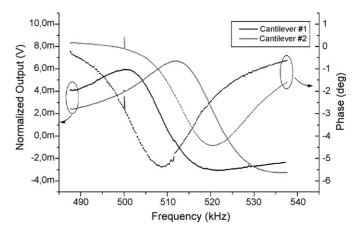


Fig. 13. Frequency response (magnitude and phase) obtained at the output of two independent CMOS readout circuits, corresponding to two resonant cantilevers from a 4-cantilevers array.

depend on the minimum detectable shift of the resonance frequency. Assuming that the resonant system behaviour can be approximated to a second-order linear system around the resonance, the variation in phase of the frequency response will be higher than in magnitude (see, for instance, Fig. 13). Thus, the minimum detectable resonance frequency shift is evaluated through the noise of phase signal measured at constant excitation frequency and the variation of the phase with frequency. For a second-order mechanical system the sensitivity of the phase with frequency will linearly depend on the Q-factor of the system $(\delta \phi / \delta f \propto Q / f_{res})$, consequently high quality factor cantilevers with low resonance frequencies will provide the best electrical transducing characteristics (minimum detectable frequency shift). Note that as we have already mentioned the mass responsitivity of the transducer increases for higher resonance frequencies (Eq. (1)).

In order to determine the expected mass resolution of the on chip mass system, both the experimental Q-factor for the transducer and the phase noise of the sensor system have been studied. An analysis of the quality factor for these on chip cantilever resonators has been done for different pressures in a vacuum chamber. In Fig. 14, the dependence of the quality factor on the pressure for a cantilever 40 μm long, 0.7 μm wide and 600 nm thick of an eight cantilevers array, with a natural resonance frequency of 440 kHz is shown. The Q-factor extracted from the electrical responses reaches a maximum of 25,000 at a pressure of 0.5 mbar with polarization voltages of $V_{DC} = 0.5$ V and $V_{\text{AC-PP}} = 180 \text{ mV}$. Note that the *Q*-factor increases for higher dc voltage polarizations and also for lower pressure as it is expected. Note also that in order to avoid the cantilever collapsing into the drivers; it is necessary to decrease the bias voltage at lower pressures. The low Q-factor value found in air is similar to the ones reported on other silicon cantilevers not integrated in CMOS and may come from squeeze film damping effect [31].

From stability measurements of the system in vacuum, the minimum detectable frequency change can be estimated. These stability measurements will take into account both the intrinsic noise due to the thermomechanical noise of the cantilever and the extrinsic noise due to the electrical noise from the CMOS circuit

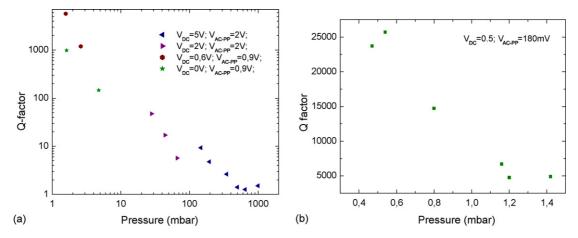


Fig. 14. Quality factor dependence on the pressure. Note the different dc and ac applied voltages for measurements at different pressures.

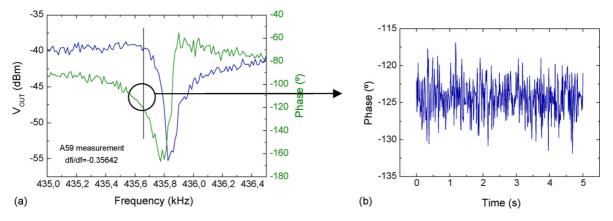


Fig. 15. (a) Frequency response (magnitude in blue and phase in green) for a resonant cantilever at a pressure of 1 mbar. (b) Variation of the phase with time at a constant frequency.

and the experimental set-up. The intrinsic frequency shift due to the thermomechanical noise can be theoretically estimated taking into account the thermal energy at room temperature, $k_{\rm B}T$, the driving energy due to the movement of the cantilever (*x*_c represents the cantilever displacement), $E_{\rm C} = m_{\rm eff} 4\pi^2 f_0^2 x_{\rm c}^2$, and the frequency bandwidth of the measurement system, BW, [23]: $\delta f^2 = (k_B T/E_C)(f_0 B W/Q)$ which gives 0.67 Hz assuming a cantilever displacement of 250 nm, a 1 kHz bandwidth and the experimental Q-factor of 1380. Fig. 15 shows the frequency response of the cantilever at a pressure of 1 mbar, and with $V_{\text{DC}} = 5 \text{ V}$ and $V_{\text{AC-PP}} = 180 \text{ mV}$. From these measurements, the resonance frequency and the maximum phase slope (assuming a linear dependence around this resonance frequency), can be derived ($f_{res} = 435 \text{ kHz}$ and $d\phi/df = -0.36^{\circ} \text{ Hz}^{-1}$, respectively). Acquiring the evolution of the phase with time at this series resonance frequency (Fig. 15b), the maximum variation of phase (phase noise) is found to be 16° . From these values, it is easily derived this minimum detectable frequency change (45 Hz). Consequently and according with the estimated mass sensitivity (28 Hz/fg), derived from Eq. (5), the mass resolution for this on chip MEMS sensor will be smaller than 1.6 fg. From the minimum detectable frequency shift due to the cantilever thermomechanical noise, the intrinsic mass resolution would be in the 30 ag range at this pressure. We can conclude that in vacuum operation a mass sensor with multiple sensing capabilities and a mass resolution down to 100 ag has been fabricated as a full CMOS-MEMS system.

5. Conclusions

Monolithic integrated multiple mass sensor based on cantilevers arrays have been designed, fabricated and characterized. Arrays of four and eight cantilevers with on chip multiplexing system have been fabricated. Sequential measurements on each cantilever of the array have been performed for the eight cantilever configuration. Simultaneous electrical characterization of two cantilevers of the same array has been performed, allowing off-chip differential measurements for the four cantilevers configuration.

The high *Q*-factor found in vacuum, allows better resolution in the detection of the resonance frequency enhancing the minimum detectable mass for the fully integrated MEMS system. The on chip MEMS sensor presented allows a mass sensitivity higher than 28 Hz/fg and a mass resolution smaller than 2 fg. The achieved mass sensitivity, the monolithic integration of all the system (transducer and read-out circuitry) and the cantilever array configuration of the sensor makes the presented MEMS sensor an improvement in relation with present state of the art mass sensors.

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References

- K.L. Ekinci, M.L. Roukes, Nanoelectromechanical systems, Rev. Sci. Inst. 76 (2005) 061101-1–061101-12.
- [2] E. Ursula, Spichiger-Keller, Chemical Sensors and Biosensors for Medical and Biological Applications, Wiley-VCH, 1998.
- [3] O. Brand, H. Baltes, Micromachined Resonant Sensors. An Overview, Sensors, vol. 4, Wiley-VCH, 1998.
- [4] H. Baltes, O. Brand, G.K. Fedder, C. Hierold, J.G. Korvink, O. Tabata, CMOS-MEMS, vol. 2, Willey-VSCH, 2005.
- [5] See, for instance, the commercial QCM provided by QCM Research, www.qcmresearch.com, or Qsense, www.qsense.com.
- [6] L. Bouzidi, S.S. Narine, K.G. Stefanov, A.J. Slavin, High-stability quartzcrystal microbalance for investigations in surface science, Rev. Sci. Instrum. 74 (2003) 3039–3044.
- [7] S.S. Narine, A.J. Slavin, Use of the quartz crystal microbalance to measure the mass of submonolayer deposits: measuring the stoichiometry of surface oxides, J. Vac. Sci. Technol. A 16 (1998) 1857–1862.
- [8] T. Ono, X. Li, H. Miyashita, M. Esashi, Mass sensing of adsorbed molecules in sub-picogram sample with ultrathin silicon resonator, Rev. Sci. Instrum. 74 (3) (2003) 1240–1243.
- [9] N.V. Lavrik, P.G. Datskos, Femtogram mass detection using photothermally actuated nanomechanical resonators, Appl. Phys. Lett. 82 (2003) 2697–2699.
- [10] S. Dohn, R. Sandberg, W. Svendsen, A. Boisen, Enhanced functionality of cantilever based mass sensors using higher modes, Appl. Phys. Lett. 86 (2005) 233501–233504.
- [11] F.M. Battiston, J.-P. Ramseyer, H.P. Lang, M.K. Baller, Ch. Gerber, J.K. Gimzewski, W. Meyer, H.-J. Güntherodt, A chemical sensor based on a microfabricated cantilever array with simultaneous resonance-frequency and bending readout, Sens. Actuators B 77 (2001) 122–131.
- [12] V. Jaume, G. Abadal, J. Teva, M. Villarroya Gaudó, A. Uranga, X. Borrisé, F. Campabadal, J. Esteve, E. Figueras Costa, F. Pérez-Murano, Z.J. Davis, E. Forsén, A. Boisen, N. Barniol, Design, fabrication and characterization of a sub-microelectromechanical resonator with monolithically integrated CMOS readout circuit, J. Microelectromech. Syst. 14 (3) (2005) 508–519.
- [13] E. Forsén, G. Abadal, S. Ghatnekar-Nilsson, J. Teva, J. Verd, R. Sandberg, W. Svendsen, F. Perez-Murano, J. Esteve, E. Figueras, F. Campabadal, L. Montelius, N. Barniol, A. Bojsen, Ultrasensitive mass sensor fully integrated with complementary metal-oxide-semiconductor circuitry, Appl. Phys. Lett. 87 (2005) 043507-1–043507-3.
- [14] J. Fritz, M.K. Baller, H.P. Lang, H. Rothuizen, P. Vettiger, E. Meyer, H.-J. Guntherodt, Ch. Gerber, J.K. Gimzewski, Translating biomolecular recognition into nanomechanics, Science 288 (2000) 316–318.
- [15] K.L. Ekinci, X.M.H. Huang, M.L. Roukes, Ultrasensitive nanoelectromechanical mass detection, Appl. Phys. Lett. 84 (22) (2004) 4469–4471.
- [16] B. Ilic, H.G. Craighead, S. Krylov, W. Senaratne, C. Ober, P. Neuzil, Attogram detection using nanoelectromechanical oscillators, J. Appl. Phys. 95 (2004) 3694–3703.
- [17] B. Rogers, L. Manning, M. Jones, T. Sulchek, K. Murray, B. Beneschott, J.D. Adams, Z. Hu, T. Thundat, H. Cavazos, S.C. Minne, Mercury vapour detection with a self-sensing resonanting piezoelectric cantilever, Rev. Sci. Inst. 74 (11) (2003) 4899–4901.
- [18] L. Nicu, M. Guirardel, F. Chambosse, P. Rougerie, S. Hinh, E. Trevisiol, J.-M. Francois, J.-Pi. Majoral, A.-M. Caminade, E. Cattan, C. Bergaud, Resonanting piezoelectric membranes for microelec-

tromechanilly based bioassay: detection of streptavidin-gold nanoparticles interaction with biotinylated DNA, Sens. Actuators B 110 (1) (2005) 125–136.

- [19] M. Nishio, S. Sawaya, S. Akitaa, Y. Nakayama, Carbon nanotube oscillators toward zeptogram detection, Appl. Phys. Lett. 86 (2005) 133111-1–133111-3.
- [20] V. Sazonova, Y. Yaish, H. Üstünel, D. Roundy, T.A. Arias, L. McEuen Paul, A tunable carbon nanotube electromechanical oscillator, Nature 431 (2004) 284–287.
- [21] CNM-CMOS 2-poly 2-metal 2.5 µm technology, http://www.cnm.es.
- [22] D. Sarid, Scanning Force Microscopy. With Applications to Electric, Magnetic and Atomic Forces, Oxford University Press, 1994.
- [23] K.L. Ekinci, Y.T. Yang, M.L. Roukes, Ultimate limits to inertial mass sensing based upon nanoelecromechanical systems, J. Appl. Phys. 95 (2004) 2682–2689.
- [24] G. Abadal, Z.J. Davis, B. Helbo, X. Borrise, R. Ruiz, A. Boisen, F. Campabadal, J. Esteve, E. Figueras, F. Pérez-Murano, N. Barniol, Electromechanical model of a resonanting nano-cantilever based sensor for high resolution and high sensitivity mass detection, Nanotechnology 12 (2001) 100–104.
- [25] G. Rius, M. Villarroya, J. Verd, J. Teva, G. Abadal, E. Figueras, J. Esteve, N. Barniol, F. Pérez-Murano, Patterning of nanometer-scale cantilevers integrated in CMOS by e-bean lithography, in: Proceedings of second Nanospain Workshop, 2005.
- [26] S. Ghatnekar-Nilsson, E. Forsen, G. Abadal, J. Verd, F. Campabadal, F. Pérez-Murano, J. Esteve, N. Barniol, A. Boisen, L. Montelius, Resonators with integrated CMOS circuitry for mass sensing applications, fabricated by electron beam lithography, Nanotechnology 16 (2005) 98–102.
- [27] B.H. Kim, T.D. Chung, C.H. Oh, K. Chun, A new organic modifier for anti-stiction, J. Microelectron. Syst. 10 (1) (2001) 33–40.
- [28] W.R. Ashurst, C. Yau, C. Carraro, R. Maboudian, M.T. Dugger, Dichlorodimethylsilane as anti-stiction monolayer for MEMS: a comparation to the octdecyltricholosilane self-assembled monolayer, J. Microelectron. Syst. 10 (1) (2001) 41–49.
- [29] G. Abadal, Z.J. Davis, X. Borrisé, O. Hansen, A. Boisen, N. Barniol, F. Pérez-Murano, F. Serra, Atomic force microscope characterization of a resonating nanocantilever, Ultramicroscopy 97 (2003) 127–133.
- [30] R. Legtenberg, J. Gilbert, S.D. Senturia, M. Elwenspoek, Electrostatic curved electrode actuators, J. Microelectron. Syst. 6 (3) (1997) 257– 265.
- [31] Z.J. Davis, G. Abadal, B. Helbo, O. Hansen, F. Campabadal, F. Perez-Murano, J. Esteve, E. Figueras, J. Verd, N. Barniol, A. Boisen, Monolithic integration of mass sensing nano-cantilevers with CMOS circuitry, Sens Actuators, A 105 (2003) 311–319.

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