DTU Library

# Bulk disk resonator based ultrasensitive mass sensor

**Cagliani, Alberto; Davis, Zachary James**

*Published in:*
Sensors, 2009 IEEE

*Link to article, DOI:*
10.1109/ICSENS.2009.5398403

*Publication date:*
2009

*Document Version*
Publisher's PDF, also known as Version of record

# Bulk Disk Resonator Based Ultrasensitive Mass Sensor

Alberto Cagliani and Zachary J. Davis
Department of Micro-and Nanotechnology, DTU-Nanotech
Danish Technical Univeristy, DTU
Kgs. Lyngby, Denmark
Alberto.Cagliani@nanotech.dtu.dk

***Abstract*—In the framework of developing an innovative label-free sensor for multiarrayed biodetecion applications, we present a novel bulk resonator based mass sensor. The sensor is a polysilicon disk which shows a Q-factor of 6400 in air at 68.8 MHz, resulting in mass resolutions down in the femtogram range. The sensor has been characterized in terms of sensitivity both for distributed mass detection, performing six consecutive depositions of e-beam evaporated Au, and localized mass detection, depositing approximately 7.5 pg of Pt/Ga/C three times consecutively with a Focused Ion Beam system. The sensor has an extremely high distributed mass to frequency shift sensitivity of 60104 Hzcm$^2$/µg and shows a localized mass to frequency sensitivity up to 4405 Hz/pg with a localized mass resolution down to 15 fg. The device has been fabricated with a new microfabrication process that uses only two photolitographic steps in order to minimize cost and complexity.**

## I. INTRODUCTION

In recent years the possibility of integrating a mass sensor with picogram resolution directly into the CMOS process have been investigated. The commercially available mass sensors that can reach this resolution are the quartz crystal microbalance (QCM). The working principle is based on monitoring the frequency shift due to the mass loading on the surface. However the QCM can not be scaled down and the novel micro-QCM technology uses materials which are not easily integrated into the CMOS process [1]. That is why several MEMS-resonator based mass sensors have been demonstrated recently [2][3][4]. The working principle is similar to that of QCM one while the actuation of the resonator and the transduction of the output signal are often not piezoelectric but instead capacitive, optical or magnetic. Moreover the MEMS resonator based mass sensors show sensitivities and mass resolutions much higher than QCM [2][3][4]. A technique that can be readily integrated is the capacitive transduction and actuation. The presented mass sensor is based on a bulk disk resonator resonating at 68.8 MHz in Wine-Glass mode and fabricated as polysilicon disk of 60 µm in diameter electrostatically driven in resonance. The electrostatic output signal is due to the change of the capacitance between the disk and the output electrode.

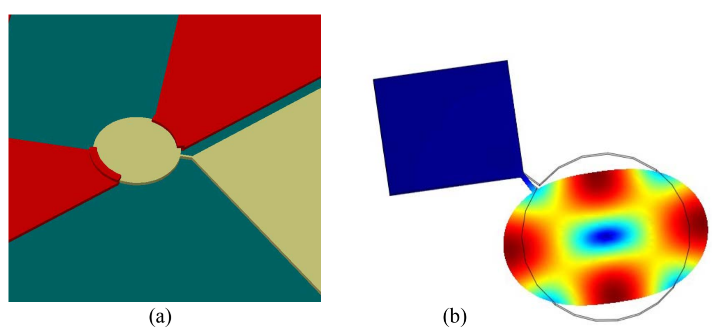
The microfabrication processes of bulk resonators presented so far use at least three photolithographic steps to shape the resonator and the electrodes around it [2] [5]. The device presented here is based on only two photolithographic steps and only two etching steps.

## II. DESIGN

The device is composed of a resonating disk of 60 µm in diameter anchored at one of four quasi-nodal points and by two transduction electrodes aligned on the same axis. The disk is driven in resonance at 68.8 MHz in so called Wine-Glass mode illustrated in Fig. 1. The actuation of the disk and the transduction of the output signal are both capacitive and, due to the fact that the electrodes are placed on the same axis, a 180 degrees phase shift is introduced between the input and the output signal. Only one anchor is used in order to reduce the losses due to the anchoring. The air gap between the disk and the electrodes is designed to be 120 nm.

## III. MICROFABRICATION

The starting point of the microfabrication process is a poly-SOI wafer, where the phosphorus doped polysilicon layer is 3 µm thick, the PECVD $SiO_2$ is 5 µm thick and the thermal silicon dioxide is 500 nm (Fig. 2a).



(a) (b)

Figure 1. (a) Layout of the bulk disk resonator: in red the transduction electrodes and in yellow the disk and its anchor; (b) Wine-Glass mode shape which shows 4 quasi-nodal points.

At first the disk and anchor are defined by an anisotropic plasma etching containing $CF_4$, $SF_6$ and $O_2$ (Fig. 2b). After that a 120 nm TEOS $SiO_2$ sacrificial layer is deposited (Fig. 2c) in order to create the air gap between the electrodes and the disk. The second layer of 3 μm of LPCVD polysilicon is then deposited and after a doping process in phosphorus atmosphere the electrodes are defined with the same anisotropic etching used in the previous step (Fig. 2d and Fig. 2e). The releasing is performed in 40% HF (Fig. 2f). In Fig. 3 the complete device is shown. The microfabrication process is completed with only two photolithographic steps and only two etching steps. The simplicity of the process reduces the device fabrication time, therefore the device cost, and also limits problems due to the misalignment of the masks and non-uniformity of the etching steps. The drawback of this approach is that no specific solutions to minimize the feed trough parasitic capacitance are used. If the signal is heavily buried in a feed trough background the estimation of the Q-factor and of the motional resistance is difficult, but there are techniques available to overcome this problem without changing the design of the chip and the microfabrication process [6].

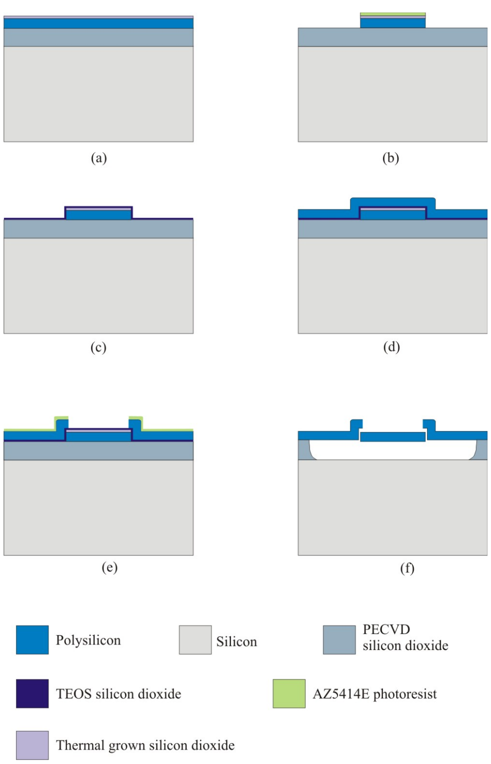



Figure 2. (a) Poly-SOI wafer: phosphorous doped polysilicon 3 μm thick, PECVD silicon dioxide 5 μm and thermal silicon dioxide 500 nm; (b) the first photoresist mask is used for etching the disk and the anchor; (c) a layer of 120 nm of TEOS silicon dioxide is deposited; (d) a second layer of phosphorous doped LPCVD polysilicon is deposited; (e) the second photoresist mask for etching the electrodes; (c) final device after the releasing in 40% HF.

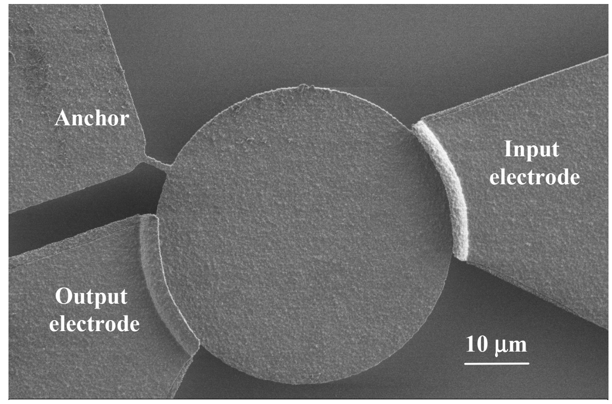



Figure. 3 SEM micrograph of the complete bulk disk resonator with one lateral anchor and two electrodes.

## IV. MEASUREMENTS

In order to characterize the performances of the bulk disk resonator as a mass sensor for distributed and localized mass detection two experiments have been performed. In the first one we deposited 2 nm of Au six times consecutively measuring the frequency shifts; in the second one approximately 22.5pg of a Pt/Ga/C composite have been deposited on a specific point of the surface. Considering the mechanical model for a harmonic resonator the relation between the frequency shift and the added mass can be expressed by

$$f_0 + \delta f = \frac{1}{2\pi}\sqrt{\frac{k_{re}}{m_{re} + \delta m}}, \qquad (1)$$

where $f_0$ is the resonant frequency, $\delta f$ is the frequency shift $k_{re}$ is the effective spring constant, $m_{re}$ is the effective mass and $\delta m$ is the added mass. If the added mass $\delta m$ can be considered much smaller than $m_{re}$ and $k_{re}$ does not change adding mass, the previous expression can be expanded in series and the linear term can be written as

$$\delta f = -\frac{f_0}{2m_{re}}\delta m. \qquad (2)$$

The slope of this linear relation defines the sensitivity of the device as

$$\Re = \frac{f_0}{2m_{re}}. \qquad (3)$$

The sensitivity of a 60 μm in diameter of a polysilicon disk (2.62 μm thick) vibrating in wine glass mode has been estimated calculating, by a FEM simulation, the effective mass and the resonance frequency for the Wine-Glass mode. The estimated sensitivity is 4464 Hz/pg with $m_{re}$ = 7.47 ng and $f_0$ =68.8 MHz.

### A. *Distributed mass sensitivity*

Six consecutive depositions of 2 nm of e-beam evaporated Au have been performed and the frequency shifts after every deposition have been recorded. In Fig. 4 three transmission curves of the device are shown, while in Fig. 5 the complete calibration curve can be seen. The Q-factor of the resonator remains constant at 6400 after every deposition. The sensitivity for distributed mass detection can be calculated with a linear fitting of the first three data points, leading to a value of 60104 Hzcm$^2$/μg and dividing by the area 2100 Hz/pg. Only the first three data have been taken into account because the assumption of linearity is not any longer valid if the total added mass is not much smaller than $m_{re}$. Since the mass is evaporated all over the disk the mode shape factor $M/m_{re}$= 2.204 (mass of the disk divided by the effective mass) has to be taken in to account in order to compare the experimental distributed mass sensitivity with the result of (3). Therefore the distributed mass sensitivity can be written as

$$\Re_d = \frac{\Re \cdot m_{re}}{M}. \tag{4}$$

This expression gives a value of 2025 Hz/pg in agreement with the experiments within the experimental error.

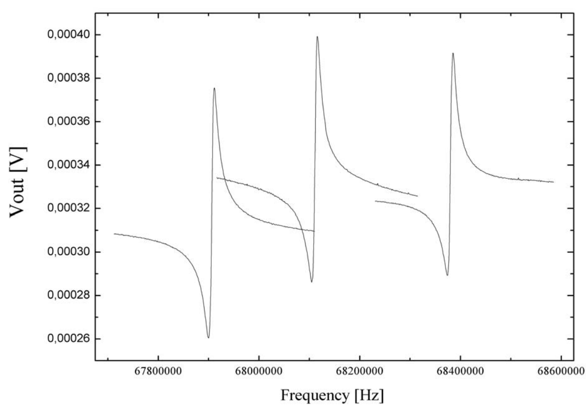



Figure 4. Ouput curves measured in air; 2 nm of gold have been deposited each time and the frequency shift due to the mass loading is shown.

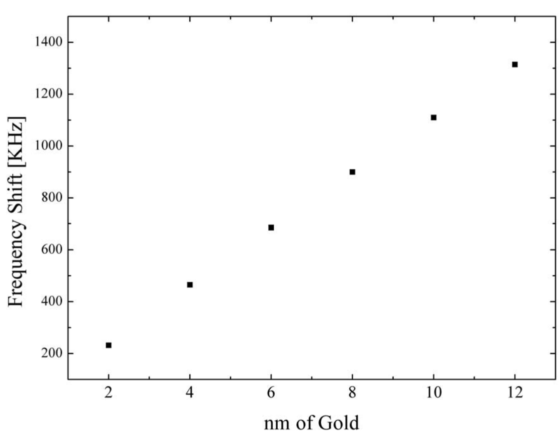



Figure 5. Calibration data reporting the six frequency shifts of the resonance frequency

## B. Localized mass sensitivity

Three consecutively depositions of a Pt/Ga/C composite have been performed inside a FEI Quanta-3D Focused Ion Beam system in order to measure the localized mass sensitivity. Differently from previous experiments the mass has been concentrated in a small area at the edge of the disk where the displacement is at maximum according to the mode shape in Figure1. Every deposited mass is a rectangle of dimensions 1.1 μm x 1.1 μm x 0.5 μm and after every deposition the frequency shifts were measured resulting in 33 kHz, 31 kHz and 38 kHz. In Fig. 6 the three added mass can be seen. The total added mass has been calculated matching the FEM simulated frequency shift with the measured frequency shift average using the density of the composite as a parameter, while the geometrical parameters have been precisely measured with a SEM. The density of the composite is unknown because it depends on the deposition parameters and specific surface properties but the range of variability can be estimated from [7]. The estimated value for the density of the composite is 12.4 g/cm$^3$ and the total added mass is 22.5 [pg] split in three depositions. In Fig. 7 two of the three frequency shifts can been seen. The estimated sensitivity is 4405 Hz/pg which is in good agreement the theoretical value from (2). Since we deposited the added mass on one of the maximum displacement points the calculated local sensitivity is maximized. Clearly the local sensitivity depends from where the tiny mass is deposited on the disk and can vary from zero to 4405 Hz/pg.

## C. Mass resolution

In order to estimate the mass resolution of the sensor the frequency noise at resonance frequency has been estimated by

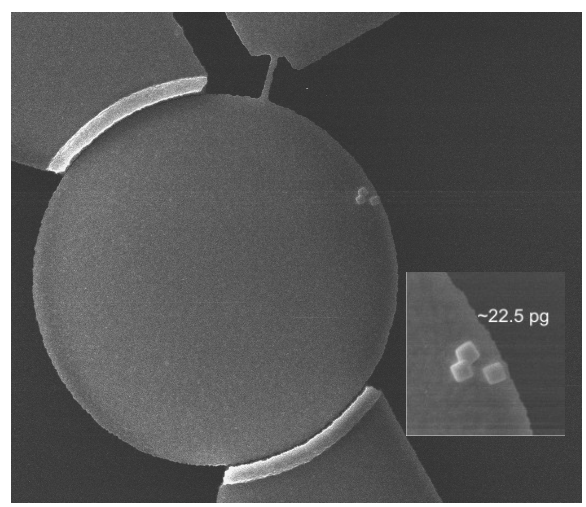
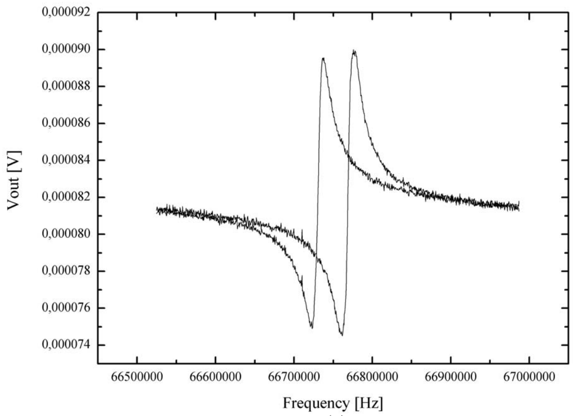
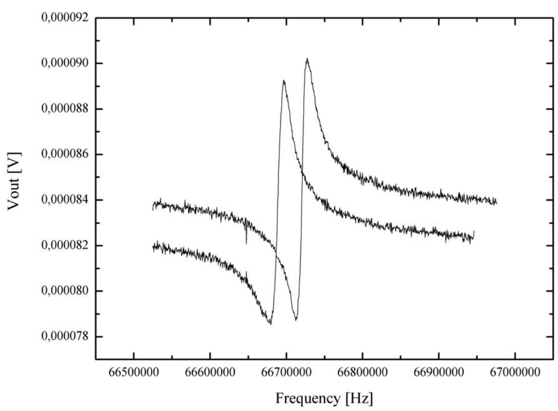



Figure 6. Approximately 22.5 pg of Pt/C/Ga composite have been deposited at the edge of the disk by FIB deposition .




(a)




(b)

Figure 7. (a) Frequency shift of 38 kHz after the second deposition; (b) Frequency shift of 31 kHz after the third deposition.

$$\Delta f = \left( \left. \frac{d\vartheta}{df} \right|_{f_0} \right)^{-1} \Delta\vartheta, \qquad (5)$$

where $\Delta\vartheta$ is the rms phase noise at resonance and $\left. \frac{d\vartheta}{df} \right|_{f_0}$ is the slope of the phase at resonance. In Fig. 8 a zero span acquisition at $f_0$ is shown and the rms value of the signal is 0.07 degrees. Multiplying the slope by the rms value a noise level for the sensor of 64 Hz is found. This leads to a localized mass resolution of 15 fg and a distributed mass resolution of 1065 pg/cm$^2$ corresponding to 6 mÅ of gold with a density of 19.3 g/cm$^3$.

## IV. Discussion and Conclusions

The possibility of using a bulk disk resonator at 68.8 MHz as ultrasensitive mass sensor has been proved and the sensor has been calibrated for distributed mass detection. The sensor shows a distributed mass sensitivity of 60104 Hzcm$^2$/μg and shows a localized sensitivity up to 4405 Hz/pg. The estimated mass resolution is respectively 1065 pg/cm$^2$ and 15 fg. The behavior of the sensor for thicker layer of gold has

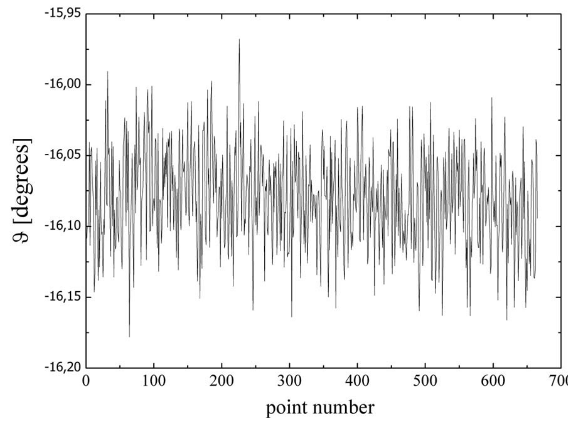



Figure 8 Zero span phase data at the resonance frequency of the device.

to be investigated in order to define the limit value at which the Q-factor significantly decreases. The map of the local sensitivity has to be completed measuring at different positions over the disk. We expect that the local sensitivity is proportional to the local displacement of the disk.

## References

[1] P. Kao, A. Patwardhan, D. Allara, S. Tadigadapa, J. Strutwolf, D. Arrigan, "Performance of micromachined quertz gravimetric sensors upon electrochemical adsorption of monolayers", IEEE Sensors 2008.

[2] J.E.-Y. Lee, B. Bahreyni, Y. Zhu and A.A. Seshia, "Ultrasensitive mass balance based on bulk acoustic mode single-crystal silicon resonator", Applied Physics Letters **91**, 2007, 234103.

[3] K.L. Ekinci, X.M. H. Huang, and M. L. Roukes, "Ultrasensitive nanoelectromechanical mass detection", Applied Physics Letters **84**, 22, 2004.

[4] D. Paci, F. Pieri, P. Toscano, A. Nannini, "A CMOS-compatible magnetically actuated resonator for mass sensing applications", Sensors and Actuators B, 129, 2008.

[5] J.R. Clark, W. T. Hsu, A. Abdelmneum, C.T.-C. Nhuyen, "High-Q UHF Micromechanical Radial-Contour Mode Disk Resonators" Journal of Micromechanical system **14**, 6, 2005.

[6] J. E. Y. Lee, Y. Zhu and A. A. Seshia, " A bulk acoustic mode single crystal silicon microresonator with a high-quality factor", Journal of Micromechanics And Microengineering, **18**, 2008, 064001.

[7] A. Villa, F. Hernadez-Rmirez, J. Rodrigues, O. Casals, A. Romano-Rodriguez, J.R. Morante and M. Abid, "Fabrication of metallic contacts to nanometre-sized materials using a focused ion beam (FIB)", Material Science and Engineering C, 26, 2006.