Microcantilever: a chemical resonant sensor

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MICROCANTILEVER: A CHEMICAL RESONANT SENSOR

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Abstract

Silicon micromachined cantilevers can be used as chemical resonant sensor by adding a sensitive layer on the device structure. In this paper, the process technology, based on the use of SOI wafer, and the sensitive layer deposition are described. Using four different geometrical microcantilevers, the frequency dependence to mass modification is measured and allows to predict gas sensor response.

Key Words: microcantilever, sensitive coating, resonant frequency, mass effect

I. INTRODUCTION

Micromechanical freestanding microstructures, and microcantilevers in particular, are gaining interest as new applications for gas detection. The sorption of specific species by a sensitive layer deposited on silicon microcantilevers modifies the mechanical properties of the structure. In fact, perturbations of the sensitive coating properties induce frequency drift (Fig. 1), thus making chemical detection possible [1, 2]. This physical effect is used to realize a chemical sensor and the sensitive coating is chosen for its high affinities with molecules to detect.

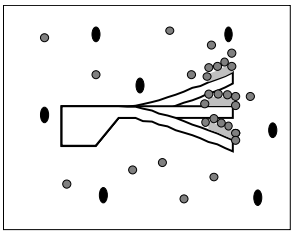


Fig. 1. Resonant frequency variation

In this paper, experimental measurements performed with microcantilevers having different geometries are presented. In fact, the measurement of the mass dependence of the resonant frequency allows to predict the gas sensor response, especially the gas concentration sensitivity.

II. MICROCANTILEVER DEVICES

II.1. PROCESS FABRICATION

The process to achieve the microstructures has been done at ESIEE group [3] using silicon technologies. Five mask levels (Fig. 2) were necessary for the fabrication of silicon microcantilevers: the first two are used to define strain gauges, another one is dedicated to electric contacts and the last two are required to etch both the top and bottom silicon sides by DRIE. In order to chose accurately structures thicknesses, n-type SOI (Silicon On Insulator) wafers have been used. The oxide thickness separating SOI to silicon is about 0.5µm and acts as stop layer for DRIE etching. First, a 4500Å silicon oxide is deposited by wet oxidation, used later as a protect layer when boron diffusion is set up on silicon. This step is followed by a photolithography which permits to define strain gauges areas. After cleaning, the structure is doped with boron implantation at 975°C during 20min and annealed inducing the boron diffusion on silicon substrate (a). The second mask draws contact strain gauges aperture, then the oxide is etched with HF 10% (b). At this stage, 1µm aluminum thick is deposited by pulverization followed by the third photolithography which specifies contact pads and coils for magnetic actuation (c). Aluminum etching is later realized in wet. Using the fourth mask, the next step consists on etching the top side of the SOI by DRIE. Before the DRIE, considering the weak SOI thickness, a photoresist has to be deposited as protective layer, then the passivation oxide and the bottom side oxide are removed (d). The bottom side etching requires to protect the top side with thick resin, then a 5000Å aluminum layer, deposited by pulverization, is used on the back side as protective layer. The last photolithography needs a bottom side alignment in order to achieve the complete etching of the wafer by DRIE. The total substrate

thickness (525µm) is etched up to the oxide. Finally, the last and most important step is the release of the freestanding microstructures obtained by wet etching (BHF 10%) (e). At last, the cheap dicing is realized before the cleaning so that the resin protects the structures.

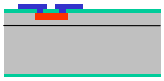
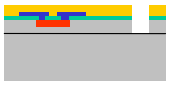
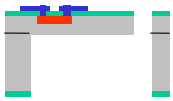
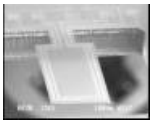
steps	*diagrams*	*pictures*
(a)		
(b)		
(c)		
(d)		
(e)		

Fig. 2. Technological steps

II.2 COATING MATERIAL

Among a wide range of polymeric coating materials, partial selectivity and sorption properties can be investigated. Sensor sensitivities and selectivities are controlled by tailoring the chemical and physical properties of the coating material to maximize particular solubility interactions. The coating selection is made through a systematic variation of the coating material solubility properties, so that the sensor is selective for a different balance of solubility interactions [4]. According to our application, in order to detect humidity and organic volatile compounds (alcohols, toluene, n-octane…) the microsensors are coated with a slightly polar poly(etherurethane) PEUT film. This polymer, received from Thermedics Inc. (Tecoflex EG-80A), presents a high porosity and its physical data are summarized in Table 1.

Table 1. Physical Data

	Flexural modulus	**Mass density**
PEUT	**0.971 psi**	**1040 kg/m^3**

This polymer is deposited on one side of the cantilever by "spray coating" using a time controller, to obtain a reproducible layer (Fig. 3). Before spraying, the PEUT is dissolved in methylene dichloride (25mg/ml) and is continuously agitated to maintain a better homogenisation. Then, the solution is sprayed onto the cleaned devices with an airbrush system (Dosage 2000, model Valmemate™ 7040) using pure nitrogen as carrier gas and a shadow mask.

Fig. 3. Coating system

III. MASS EFFECT MEASUREMENTS

III.1. MEASUREMENT PROCEDURE

Targeting a full compact microsystem, the excitation of the cantilever in the resonant mode is performed by using piezoelectric material or a magnetic field. The structure oscillations are detected with the strain gauges etched on the device surface. Then, the microcantilever is inserted in the retroaction loop of an amplifier to constitute an oscillator (Fig. 4) and, according to the Barkhausen conditions, the resonant frequency is monitored. Using this electronic circuit, shifts in the frequency are observed when the structure mass changes, e.g by addition of a sensitive coating or later by the sorption of vapor into surface coating.

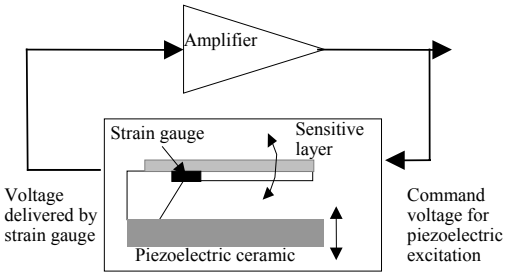


Fig. 4. Block diagram of the electrical oscillator with piezoelectric excitation and piezoresistive measurement

III.2. EXPERIMENTAL RESULTS

The measurements have been realised with four structures labelled **IC5** , **IC8** , **IB5** , **IB6** (Fig. 5 and Table 2) [5].

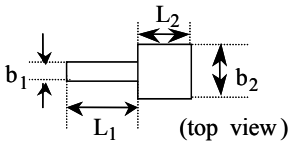


Fig. 5. Microcantilever geometry

Table 2. Device dimensions

	L_1	b_1	b_2	L_2
IC5	1mm	200µm	1mm	1mm
IC8	1mm	200µm	2mm	2mm
IB5	500µm	200µm	1mm	1mm
IB6	500µm	200µm	2mm	2mm

Hence, the experiments consist in recording the frequency shift of each structures in oscillator configuration (Fig. 6). Ten sprays of dissolved polymer (duration: 1s) have been made with a polymer stabilisation waiting between each spray.

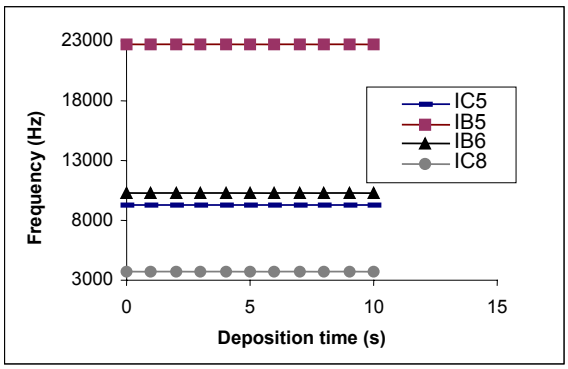


Fig. 6. Resonant frequency versus deposition time

The variation of the resonant frequency with the deposition time is due to the cantilever mass sensitivity which will be determined in the following section.

IV. GAS SENSOR RESPONSE

IV.1 MASS SENSITIVITY EVALUATION

The microstructure resonant frequency can be expressed by the classical expression:

$$f = \frac{1}{2\pi}\sqrt{\frac{k}{0.24m}}$$

with m the microstructure mass and k the microstructure stiffness.

For a cantilever composed of two different materials, the mass and stiffness expressions are:

$$m = (\rho_1 h_1 + \rho_2 h_2)\Sigma$$

and

$$k = \frac{\alpha\Sigma}{L}\left(\frac{\hat{E}_1 h_1^3}{12} + \frac{\hat{E}_2 h_2^3}{12} + \frac{h_1 h_2 \hat{E}_1 \hat{E}_2 (h_1 + h_2)^2}{4(h_1\hat{E}_1 + h_2\hat{E}_2)}\right)$$

with α a constant depending on the shape of the microcantilever, Σ the upper-surface of the microcantilever, L the length, h_1 and h_2 the thicknesses of the two materials, ρ_1 and ρ_2 the mass densities, $\hat{E}_1 = E_1/(1-\nu_1^2)$ and $\hat{E}_2 = E_2/(1-\nu_2^2)$ with E_1 and E_2 the Young's moduli and ν_1 and ν_2 the Poisson's coefficients. During the sensitive coating deposit, the structure stiffness can be considered constant and only the mass is modified [6]. Consequently, the frequency expression f with coating is linked to the frequency without coating, f_0, as follows:

$$f = f_0 \frac{1}{\sqrt{1 + \frac{\rho_2 h_2}{\rho_1 h_1}}}$$

Using this expression, the polymer thickness is extracted from experimental frequency shift (Fig. 6) and is plotted versus time deposition (Fig. 7). These results, obtained with different structures measurements, allow to characterise the spray coating (the PEUT thickness increases linearly with the deposition time in a reproducible way) and to determine the mass sensitivities for each microcantilevers: S_m (Table 3).

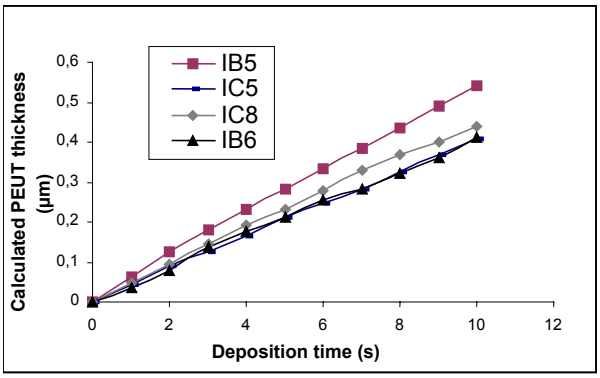


Fig. 7. PEUT thickness versus time deposition of each structure

Table 3. Mass sensitivities

IC5	S_m=0.025 Hz/ng
IC8	S_m=0.003 Hz/ng
IB5	S_m=0.06 Hz/ng
IB6	S_m=0.008Hz/ng

IV.2. STRUCTURES COMPARISON

Then, using the partition coefficient K (relative to the sensitive coating and gas) and the measured mass sensitivities (Δf/Δm), (Table 3) the gas concentration sensitivity is estimated [5]:

$$S_{Cg}^{f} = \Delta f / \Delta C_g = K\, h_2\, \Sigma\, (\Delta f/\Delta m)$$

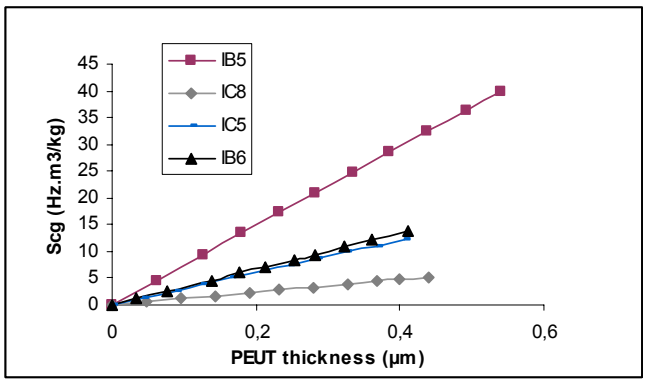


Fig. 8. Gas concentration sensitivity versus PEUT thickness

Both experimental results (Fig. 8) and theoretical model show a sensitivity S_{Cg}^{f} proportional to resonant frequency f_0 and polymer thickness h_2. However, the oscillator noise usually increases with the frequency and consequently the limit of detection could be less performing for high resonant frequency system. Moreover, large PEUT thickness induces important diffusion time (sorption/desorption) and so a long sensor response time. Consequently, the choice of resonant frequency and polymer thickness for gas detection will result from comprises concerning sensitivity, response time and limit of detection which is directly linked to the oscillator noise.

V. CONCLUSION

SOI microcantilevers with integrated excitation (piezoelectric, electromagnetic) and piezoresistive measurement have been realized. A spray coating system has also been developed. Then, the first measurements of resonant frequency variation show the coating thickness reproducibility and allow to calculate the mass sensitivity. Thanks to this experimental results, the gas sensor response has been studied by estimating the gas concentration sensitivity. Actually, a gas line test bench is in process which will permit to compare the results to gas measurements.

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