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Sensor Application Using Longitudinal Mode of Screen-Printed PZT Cantilever

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Abstract

High gas sensitivity, expected when using the unusual longitudinal vibration mode of PZT cantilever for such application, is based on its higher resonant frequency when compared to transversal bending mode. For this purpose, a new PZT cantilever structure has been fabricated with thick-film technology based on the sacrificial layer method. This structure is well adapted to generate the 31's longitudinal vibration. Studies of the composition and microstructure of Au/PZT/Au beam show the harmlessness of the process. Advantage of the 31's longitudinal mode for gas detection is confirmed by the values of resonant frequency and good quality factor for a 8x2x0.08 mm³ PZT based cantilever. Indeed, taking a 0.1Hz frequency resolution for the second 31 mode, the limit of detection of 6 ppm toluene is reached with a 44µm PEUT coating.

Keywords: Cantilever, Gas Detection, Self-Actuated piezoelectric, Thick-film

1. Introduction

Until now, MEMS based on silicon micromachining are well suited for chemical sensor applications and more precisely in case of cantilever structure. To perform detection of chemical or biological compounds, specific-sensitive layer is prior deposited on the cantilever to trap the different species. This phenomenon, which affects the mass of cantilever, induces both bending rigidity and surface stress changes. These mechanical modifications find expression in static or in dynamic modes respectively by curvature or resonance alterations [1]. Generally, resonating cantilevers are activated with transversal mode and give satisfactorily results with piezoelectric layer driver [2]. Well known sensing studies with these modes are performed with a bilayer cantilever structure of this previous type fabricated with silicon IC technology. However, better sensitivity might be obtained using another resonating mode at higher frequency. Unlike classical modes like transversal bending resonating mode, the called 31's longitudinal mode works at higher frequencies. We thus expect better sensitivity and lower viscous and piezoelectric losses without reducing both the cantilever size and the gas interaction surface [3].

In order to generate 31 longitudinal mode, fabrication of a new PZT cantilever structure with high symmetric geometry is proposed using thick-film processing as an alternative to silicon micromachining. Indeed, previous work on free-standing resonating microstructures based on screen-printed PZT, confirms the potentiality of thick-film processing for cantilever fabrication [4].

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2. Design and fabrication

2.1. Fabrication of the self-actuated cantilever

Contrary to silicon cantilever, self-actuating piezoelectric microcantilever shown in Fig. 1 does not need any added piezoresistance or optical device for detection. It consists of multi-layered Au/PZT/Au partially released from alumina substrate. Free-standing structures of $8 \times 2 \times 0.08 \text{ mm}^3$ are achieved with screen-printing technology. After a prior deposition and polymerization of the sacrificial layer made of epoxy-based strontium carbonate on an alumina substrate (Fig. 1a), bottom electrode, PZT layer and upper electrode are successively screen-printed and dried 20min at 120°C (Fig. 1b). Commercial gold 8836 ESL paste is chosen for electrode.

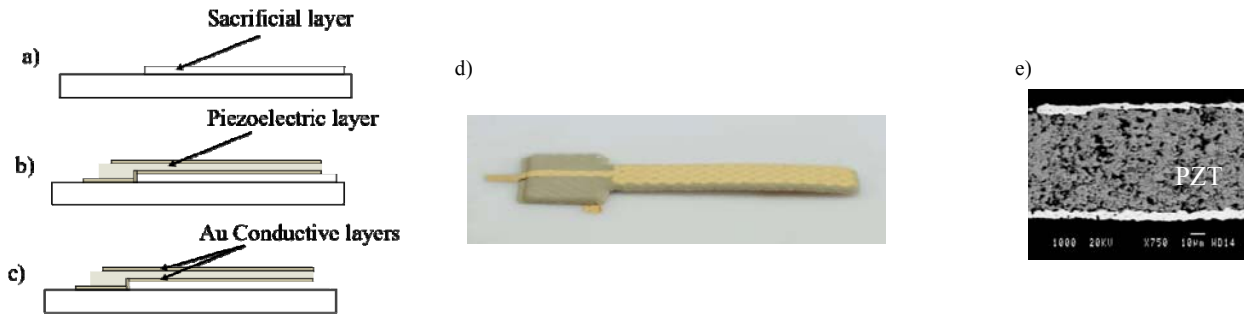


Fig. 1. Fabrication and flow chart of micromechanical PZT cantilever for self-actuating/sensing (a) thick-film deposition of sacrificial layer; (b) multi-stage screen-printing Au/PZT/Au; (c) sacrificial layer elimination with H_3PO_4 ; (d) photo of piezoelectric cantilever; and (e) SEM picture of multi-layered structure Au/PZT/Au.

The piezoelectric paste is prepared from piezoelectric PZ26 powder (Ferroperm) and 7%-w lead borosilicate glass frit blended with ESL 400 organic vehicle [4]. The samples are fired 15 min at 850°C in air. Finally, dissolution of sacrificial layer is performed in the 0.9 mole.l^{-1} of H_3PO_4 aqueous solution. PZT cantilevers shown on Fig.1.d are then poled at 550K with an electric field of 1 kVmm^{-1} . From the SEM microstructure of the porous PZT layer, a 70% compacity has been estimated with an image calculation (Fig.1.e). Thickness of $65 \mu\text{m}$ and $8 \mu\text{m}$ are respectively measured for PZT layer and gold electrodes.

2.2. Analyze of transversal bending and 31's longitudinal modes

Optical measurements with a vibrometer MSA Polytec (Fig.2.a) and electrical characterization with impedancemeter HP 4194A (Fig.2.b) on PZT cantilever show the three first transversal bending modes and three first 31's longitudinal modes respectively. In the framework of full piezoelectric cantilever structure, it follows the analytical expressions of resonant frequency of 31's longitudinal (eq. 1) and transversal bending (eq. 2) modes

$$f_{31}^{(n)} = \frac{\lambda_{31}^{(n)}}{2\pi} \sqrt{\frac{k}{m}} = \frac{1}{2\pi} \left(\frac{\lambda_{31}^{(n)}}{L} \right) \sqrt{\frac{E_{PZT} h_{PZT} + 2E_{Au} h_{Au}}{\rho_{PZT} h_{PZT} + 2\rho_{Au} h_{Au}}} \quad \text{and} \quad \lambda_{31}^{(n)} = (2n+1) \frac{\pi}{2}, n = 0 \dots \quad (1)$$

$$f_{\text{Bending}}^{(n)} = \frac{1}{2\pi} \left(\frac{\lambda_{\text{Bending}}^{(n)}}{L} \right)^2 \sqrt{\frac{h_{PZT}^3 E_{PZT} + (6h_{PZT}^2 h_{Au} + 12h_{PZT} h_{Au}^2 + 8h_{Au}^3) E_{Au}}{12(\rho_{PZT} h_{PZT} + 2\rho_{Au} h_{Au})}} \quad (2)$$

where k , m , ρ , L , h , and E are respectively the stiffness, mass, density, length thickness, and Young moduli. $\lambda_{31}^{(n)}$ and $\lambda_{\text{Bending}}^{(n)}$ (1.875, 4.69...) are the dimensionless n th-mode eigenvalues for 31 and transversal bending modes.

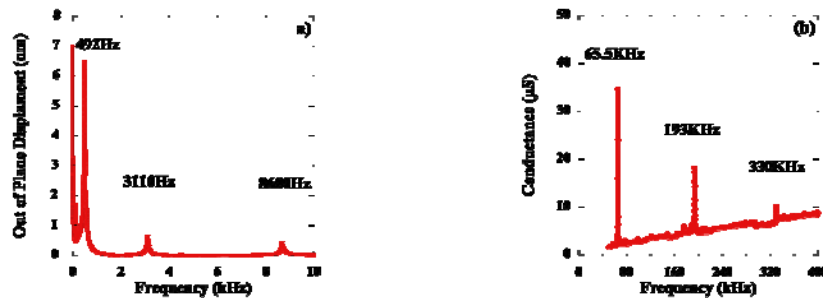


Fig. 2 : Measurements of (a) the three first bending modes with a vibrometer MSA500 Polytec and (b) the three first longitudinal modes with an impedancemeter HP 4194A.

Multi-layered symmetry doesn't favor piezoelectric actuation of bending modes whose presence are only observed with optical measurements even for displacements as low as few nanometers. However, 31's modes of piezoelectric cantilever easily recordable by impedance analyzes, show their ability for actuating/sensing with low power consumption.

Based on the estimated density ($\rho_{\text{PZT}} \sim 5.4 \text{ g.cm}^{-3}$) of PZT layer and the gold parameters (ρ, E), thickness and Young's modulus of PZT layer calculated with equation (1) and (2) (table. 1) are in agreement with SEM picture observations and thus verify analytical expression of 31's modes.

Table. 1: Parameters of PZT self-actuated microactuator

| Material | Mass density, ρ [kg.m ⁻³] | Thickness, h [μm] | Young's modulus, E [GPa] |
|----------|--|---------------------|----------------------------|
| Au | 19 300 | 8 | 70 |
| PZT | 5400 | 64.6 | 26.7 |

3. Mass influence and toluene detection

To evaluate the mass sensitivity of screen-printed PZT cantilevers, studies of toluene absorption on PEUT polymer film are undertaken. Before gas detection, the polymer influence on the resonant properties is achieved with both the first and the second 31's longitudinal modes.

3.1. Influence of polymer deposit on first and second 31's longitudinal modes

Successive PEUT polymer layers are sprayed and modify the mass and rigidity of the microcantilever. Polymer thicknesses $h_{\text{pol1}}=4\mu\text{m}$, $h_{\text{pol2}}=13\mu\text{m}$, $h_{\text{pol3}}=24\mu\text{m}$ and $h_{\text{pol4}}=44\mu\text{m}$ have been calibrated using silicon cantilever. Considering the mass addition δm and the stiffness δk of PEUT polymer, resonant frequency is given by equation 3:

$$f_{31}^{(n)} = \frac{\lambda_{31}^{(n)}}{2\pi} \sqrt{\frac{k_{\text{cantilever}} + \delta k}{m + \delta m}} \quad (3)$$

Figure 3-a and Figure 3-b represent the influence of the four polymer thicknesses h_{pol1} , h_{pol2} , h_{pol3} and h_{pol4} on the conductance of PZT cantilever at different frequencies. According to the analytic expression 3, a better mass sensitivity for the second mode is observed. In addition, better quality factor 250 of second mode compared to 110 for the first mode might give lower noise on electrical measurements.

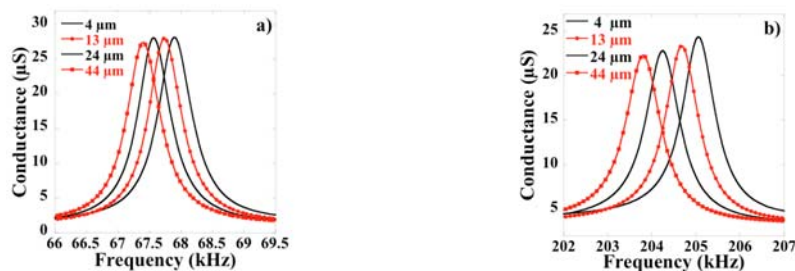


Fig. 3 : Evolution of admittance's real part during PEUT deposit for (a) First 31's longitudinal mode and for (b) Second 31's longitudinal mode

3.2. Toluene detection

Based on the above results, the mass sensitivity of the cantilever can be used for gas detection. Detection measurements under $100\text{ml}\cdot\text{min}^{-1}$ were performed with four gas concentrations of toluene diluted in nitrogen. The mass increase due to the absorption of toluene by the PEUT layer h_{pol4} (partition coefficient of 1000) leads to linear variation of the frequency shifts for the first and second longitudinal 31 modes (Fig.4). As observed in figure4, better sensitivity obtained with the second mode compared to those of the first mode, is in agreement with previous results obtained with PEUT polymer.

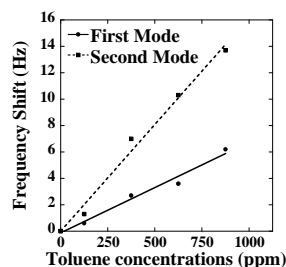


Fig. 4: Measurement of resonance shift of first and second resonances for different concentrations of toluene

In other respects, using the estimated minimum measurable frequency shift of 0.1 Hz, the limit of detection of 15 ppm for toluene has been calculated for the first longitudinal mode and 6 ppm for second one.

4. Conclusion

Electrical and optical measurements of the bending and the 31 longitudinal modes, expected for the symmetric geometry, demonstrate the feasibility of screen-printed PZT cantilever. Unlike silicon cantilevers excited by piezoelectric material, the actuating/sensing possibility of this new microsystem is effective with the longitudinal 31's modes working at higher frequencies than bending modes. The best mass sensitivity of PEUT polymer, obtained with the second longitudinal mode, is confirmed for toluene detection. Finally, the good results obtained with 31 longitudinal modes of screen-printed PZT cantilever for gas detection are attractive for measurement in liquid media.

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