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Abstract:

The use of microcantilevers as (bio)chemical sensors usually involves the application of a chemically sensitive layer. The coated device operates either in a static bending regime or in a dynamic flexural mode. While some of these coated devices may be operated successfully in both the static and the dynamic modes, others may suffer from certain shortcomings depending on the type of coating, the medium of operation and the sensing application. Such shortcomings include lack of selectivity and reversibility of the sensitive coating and a reduced quality factor due to the surrounding medium. In particular, the performance of microcantilevers excited in their standard out-of-plane dynamic mode drastically decreases in viscous liquid media. Moreover, the responses of coated cantilevers operating in the static bending mode are often difficult to interpret. To resolve these performance issues, the following emerging unconventional uses of microcantilevers are reviewed in this paper: (1) dynamic-mode operation without using a sensitive coating, (2) the use of in-plane vibration modes (both flexural and longitudinal) in liquid media, and (3) incorporation of viscoelastic effects in the coatings in the static mode of operation. The advantages and drawbacks of these atypical uses of microcantilevers for chemical sensing in gas and liquid environments are discussed.

Keywords: Cantilever, Chemical detection, Resonant sensor, In-plane mode, Viscoelasticity, Static mode.

1. Introduction

In recent years, interest in microcantilever-based chemical and bio-chemical sensing systems has risen due to their projected high sensitivity [1], [2], [3], [4] and [5]. The large ratio of surface area to

volume makes the microcantilever extremely sensitive to surface processes. For (bio)chemical detection, the microcantilever is usually coated with a (bio)chemically sensitive layer that aims to selectively sorb the analyte or molecule of interest. The sorbed substances can then be detected by monitoring either the resonant frequency shift (dynamic mode) or the quasi-static deflection (static mode). A simple way to explain the basic principle of such sensors is to say that in the case of dynamic mode operation, the change in mass associated with the sorption of analytes into the sensitive coating causes a shift in resonant frequency, which may be correlated to the ambient concentration of the target substance, while for static-mode operation, the sorption of analyte causes a cantilever deflection that is induced by the tendency of the sensitive coating to expand (or contract) upon analyte sorption (modification of strain and stress in the coating). However, the behavior of such cantilevers may actually be much more complicated as illustrated in Fig. 1.

As illustrated in Fig. 1 and as noted in the previous paragraph, the predominant effect in the dynamic mode is the mass modification, while in the static mode the stress and strain modification; however, secondary effects may include static deflection due to sorbed mass and frequency shifts due to analyte-induced coating stress/strain. Another effect which is not necessarily negligible is the effect of the state of the surrounding medium on the coating sorption properties (e.g., fluid temperature on polymer partition coefficient) or on the static or dynamic response of the microcantilever (e.g., "thermal drift" in the output signal due to Young's modulus temperature dependence).

While such cantilever-based sensors generally operate relatively well, they have shortcomings depending on the type of coating, the medium of operation and the application. The main performance issues can be summarized as follows:

- *Drawbacks due to the sensitive coating:* In addition to being subjected to environmental effects including temperature, humidity and aging effects, most chemically sensitive coatings, often polymers, only show partial selectivity to various analytes. Ideal selectivity is often achieved at the expense of reversibility. Moreover, the limited long-term

stability of the viscoelastic coatings and the resulting aging affect the reliability of the sensor.

- *Drawbacks of the commonly used flexural out-of-plane dynamic mode in liquid media:* Dynamically excited microcantilevers are well suited for operation in low-viscosity media such as air. Their usefulness as a sensing platform is limited when operating in viscous liquid media. This is due to the large decrease in the device's quality factor caused by the additional viscous losses in the fluid. While the viscous damping reduces the quality factor the effective displaced fluid mass causes a significant decrease in the resonant frequency. The result is a decrease in the device sensitivity as well as an increase in the system's susceptibility to frequency noise, thus raising the sensor's detection limit. It is noted that the sensitivity of the sensors in the dynamic mode increases with the resonant frequency, whereas the quality factor Q of the resonance mode influences the sensor resolution.
- *Drawbacks due to the interpretation of the static-mode responses:* In static-mode operation, polymer-coated microcantilevers may exhibit either a monotonic transient response or a response exhibiting an overshoot [6]. As the stresses in the cantilever coated with a viscoelastic material may depend on time, the coated cantilever's response (bending or deflection) will therefore exhibit more complex behaviors. Such complexities in the sensor response make it difficult to easily interpret the measurements, analyze and predict the sensor's response, and perform signal processing for rapid and error-free detection of chemical analytes.

In the present paper, we will review unconventional uses of microcantilevers as chemical sensors to solve some of the performance issues and avoid or minimize the associated drawbacks. To eliminate problems associated with the sensitive coatings, an alternative method to detect and quantify chemical species without the use of a sensitive coating will be presented (Section 2). To resolve the drawbacks associated with the commonly used out-of-plane flexural dynamic mode in viscous liquid media, the characteristics of alternative vibrating modes, such as the in-plane (flexural and longitudinal)

vibrating modes, will be described (Section 3). Finally, an interpretation of the static-mode cantilever response taking into account the viscoelasticity of the sensitive coating will be introduced in Section 4.

Notation

The cantilever geometry is shown in Fig. 2a and the actuation and deflection in Fig. 2b.

The geometry of the cantilever is defined by the width b , thickness h , and length L . Coordinate x is measured along the beam length. The actuation force F and the resulting deflection w occur at the angular frequency ω . The properties of the cantilever material are the Young's modulus E and the beam mass density ρ . The fluid (gas or liquid) properties are defined as ρ_f , the fluid mass density, and η , the fluid viscosity.

2. Uncoated microcantilever-based chemical sensors operating in dynamic mode

2.1. Basic equations for dynamic mode operation

When a microcantilever vibrates in a viscous fluid (gas or liquid), the fluid offers resistance to the motion. The force per unit length, F_{fluid} , which is the consequence of all normal and tangential stresses (hydrodynamic pressure and viscous shear) exerted by the fluid on all the surfaces of the cantilever, can be written in the frequency domain (underlined notation) as [7]:

$$\underline{F}_{\text{fluid}} = -j\omega \underline{g}_1(\omega, x) \underline{w}(\omega, x) + \omega^2 \underline{g}_2(\omega, x) \underline{w}(\omega, x) \quad (1)$$

where x is the longitudinal coordinate, ω is the radial frequency of the vibration, $\underline{w}(\omega, x)$ is the microcantilever deflection, and \underline{g}_1 and \underline{g}_2 are functions specific to the fluid properties and the microcantilever cross-sectional geometry. They may be interpreted, respectively, as the distributed damping coefficient of the fluid and the distributed effective fluid mass. In general, these quantities may be x -dependent; however, in what follows they will be assumed uniform in

x as this assumption has been shown to be sufficiently accurate for modeling microcantilever flexural vibrations in viscous fluids (e.g., [8]).

The fluid effects (viscous and inertial) influence the dynamic response of the beam, in particular, the resonant frequency f_r and the quality factor associated with viscous losses, Q_{visc} , as [8] and [9]:

$$f_r = f_{0,\text{vac}} \frac{1}{\sqrt{1 + L \underline{g}_2 / m}} \sqrt{1 - \frac{1}{2Q^2}} \quad (2)$$

$$Q_{\text{visc}} = \frac{2\pi \sqrt{1 + L \underline{g}_2 / m}}{L \underline{g}_1 / m} f_{0,\text{vac}} \quad (3)$$

where $f_{0,\text{vac}}$ is the undamped natural frequency of the microcantilever in vacuum, Q is the total quality factor of the cantilever/fluid system (incorporating all losses), m is the microcantilever mass and L is the microcantilever length.

The above equations illustrate the fact that in the general case of dynamic mode operation of a microcantilever, the resonant frequency depends on the fluid properties via the terms \underline{g}_1 and \underline{g}_2 . Such dependency can be used, in some cases, to characterize the fluid, thus eliminating the need for a chemically sensitive coating.

2.2. Gas detection in binary mixture

Using the previous equations, analytical expressions for the relative frequency shift due to small changes in both mass density and viscosity of the fluid can be obtained. In the case of different binary mixtures (He/N₂, CO₂/N₂ and H₂/N₂), it has been shown that the predominant effect for the change in the microcantilever resonant

frequency is due to the gas mass density variation. The sensitivity to mass density can be approximated by [10]

$$\frac{df_r}{d\rho_f} = -\frac{a_1\pi}{8} \frac{b}{\rho h} f_r \quad (4)$$

From Eq. (4) one may conclude that the relative frequency shift due to the mass density variation of the gas increases with the ratio of the microcantilever width to the microcantilever thickness. Examples for the detection of hydrogen in nitrogen and carbon dioxide in nitrogen using silicon cantilevers with electromagnetic actuation and piezoresistive measurements are presented in Fig. 3. The silicon cantilever has a length of 2 mm, a width of 400 μm and a thickness of 15 μm . The resonance frequency of the uncoated sensor is approximately 5 kHz.

As predicted by Eq. (4), due to the fact that hydrogen is lighter than nitrogen, the resonant frequency increases with the hydrogen concentration, whereas it decreases in the case of carbon dioxide detection because this gas is heavier than nitrogen.

In order to estimate the limit of detection with this cantilever in the case of hydrogen detection in nitrogen, measurements with smaller hydrogen concentrations have been conducted (Fig. 4). It can be seen that, without any signal processing, the noise in the resonant frequency measurement is approximately 2 mHz and that the detection of 200 ppm of hydrogen is possible.

Compared to classical resonant microcantilever-based chemical sensors with sensitive coatings, the uncoated microsensors exhibit shorter response times because there is no analyte sorption into the coating affecting the sensor response. Moreover, the absence of the sensitive coating leads to a more reliable and reversible behavior because there is no significant absorption and desorption phenomenon. The above results indicate that uncoated microsensors may serve as viable devices for the detection of specific concentrations of one gas in a binary mixture. The sensitivity and resolution of such

sensors will be larger for those cases in which the difference between the mass densities of the two gases is higher. The major drawback of such sensors is that there is no intrinsic selectivity; thus, these sensors can only be used for specific applications such as when monitoring environments in which it is known that only one gas concentration can vary while the relative concentrations of the other gases remain fixed.

3. In-plane vibration modes in liquid media

From Eq. (2), it can be seen that the decrease of the resonant frequency when the microcantilever is immersed in a fluid is essentially due to the mass effect $\underline{g_2}$; this effect can be minimized if the term $\underline{g_2}$ is small in liquid. On the other hand, the strong decrease in the quality factor in liquid is due to the stronger influence of the viscous term $\underline{g_1}$ compared to the influence of the displaced fluid mass (term due to $\underline{g_2}$) in Eq. (3).

In order to limit the decrease of both the resonant frequency and the quality factor upon immersion in liquid media, alternative vibration modes that essentially shear the surrounding fluid rather than exerting normal stress on it have been studied and tested. In particular, two in-plane vibration modes (lateral flexural mode and elongation mode) (Fig. 5) have been theoretically studied [11] and [12] in order to better understand the "intuitive" advantages offered by the in-plane mode of microcantilever-based sensors over their out-of-plane counterparts, as well as to quantify these advantages. Example analysis and measurements of the resonant frequency and quality factor associated with these two unconventional modes are presented in what follows.

3.1. In-plane (lateral) flexural mode

Exciting the microcantilever in the in-plane flexural vibration mode (or lateral vibration) rather than the out-of-plane flexural mode would reduce the amount of fluid resistance (combined effects of fluid-related inertial and viscous forces), which could potentially improve the sensitivity and limit of detection of microcantilever-based chemical sensors. The characteristics of the in-plane flexural mode can be

obtained by evaluating the hydrodynamic forces (\underline{g}_1 and \underline{g}_2) acting on a laterally vibrating microcantilever.

A first modeling approach consists of considering that the displacement of the fluid is the same as in the case of an infinite vibrating plane. Then the hydrodynamic force acts only on the two larger surfaces of the cantilever and the expressions of \underline{g}_1 and \underline{g}_2 are [13] and [14]:

$$\underline{g}_1 = \underline{g}_2 \omega \quad \text{and} \quad \underline{g}_2 = b \sqrt{\frac{2\eta\rho_f}{\omega}} \quad (5)$$

Modeling the fluid resistance using Eq. (5) corresponds to the limiting case of high Reynolds's number (high frequency), i.e., $Re \rightarrow \infty$, and thin microcantilevers vibrating in the lateral flexural mode, i.e., cross-sectional aspect ratio $h/b \rightarrow 0$. This type of fluid resistance is described as "Stokes resistance" and leads to simple analytical expressions for the quality factor due to viscous fluid losses and the relative frequency change due to immersion in a viscous fluid [13] and [14].

A more complete modeling approach consists of taking into account both the shear forces acting on the two larger surfaces and the pressure forces acting on the two other surfaces (Fig. 6). Each force is composed of two terms: one proportional to velocity and one proportional to acceleration. Then it is also theoretically possible to extract the two terms \underline{g}_1 and \underline{g}_2 . But due to edge effects, as shown in Fig. 6, each force is not uniformly distributed. Recently, finite-element results combined with semi-analytical models [11] and [15] have appeared in the literature to provide more accuracy results for the hydrodynamic force. These studies show that \underline{g}_1 and \underline{g}_2 are functions of both Reynolds number (dimensionless frequency involving fluid properties) and cantilever aspect ratio (h/b). Obtaining the hydrodynamic forces allows for the resonant frequency and Q-factor to be investigated as functions of both beam geometry and medium properties [11]. Trends in these characteristics can be used to optimize device geometry and maximize the frequency stability in sensing applications.

Several cantilevers with widths of 45, 60, 75 and 90 μm , lengths of 200, 400, 600, 800, 1000 μm and silicon thicknesses of 8 and 12 μm , corresponding to in-plane resonant frequencies up to about 3 MHz, have been characterized in air and water. The modeling of the fluid interaction terms, \underline{g}_1 and \underline{g}_2 , according to Eq. (5) for the lateral mode result in the resonant frequency being proportional to b/L_2 (where b is the cantilever width and L is the cantilever length), the relative resonant frequency shift from air to water being proportional to $L/(h\sqrt{b})$ and the quality factor (in water) being proportional to $h\sqrt{b}/L$ [13] and [14]. The results of a simple, closed-form analytical expression for Q gives excellent quantitative agreement for the quality factor in water for aspect ratios up to $h/b = 0.2$ (see, e.g., Fig. 7a) and a reasonable upper-bound estimate for thicker beams [13] and [14]. In addition, the analytical formula for resonant frequency in fluid [13] matches the trend exhibited by measurements made on the different cantilever geometries when operated in water (Fig. 7b). While the simple theory predicts qualitatively certain aspects of the behavior of the relative frequency drop from air to water (see, e.g., the slope in Fig. 7c), the quantitative results for the relative change in frequency significantly underestimate the data due to the neglected pressure effect on the smaller faces (i.e., the beam thickness effect). Therefore, the analytical result for the fluid-induced relative frequency change [13] should only be used as a general guide for the first-order dependency of relative frequency drop on beam geometry. (An extension of the analytical formula for % frequency change that accounts for the pressure effect has been achieved recently and will be submitted for publication in the near future.) Other interesting features of the experimental data include (1) for the first lateral flexural mode the microcantilevers exhibit quality factors in air up to 4300 and up to 67 in water (which usually does not exceed 10–20 in water for out-of-plane modes); (2) the resonant frequencies of the in-plane mode are only lowered by 5–10% in water compared to the values in air, whereas this reduction is approximately 50% for the out-of-plane modes. These two characteristics of the lateral mode operation are promising for future detection in liquid media.

For the microcantilevers designed and fabricated for operation in the lateral flexural vibration mode, thermal excitation and piezoresistive detection, both based on the use of diffused resistors,

have been chosen as actuating and sensing mechanisms, respectively. The design of the actuation resistors and integrated Wheatstone bridge allow a more efficient excitation of the in-plane flexural mode than the more classical out-of-plane flexural mode [16]. It is noted that, for example, at a given driving voltage, the free-end vibration amplitude, measured using a Polytec MSA-500 laser vibrometer, is 118 nm at the first transverse flexural resonant frequency whereas it is 750 nm for the first lateral flexural resonant frequency.

For dynamically driven microcantilever chemical sensors, the limit of detection is usually defined as the analyte mass or concentration corresponding to a frequency shift equal to three times the frequency noise of the system measurement, (or is defined as three times the ratio of the device/system frequency noise to the chemical sensitivity). It is difficult to directly measure and compare the LOD because of the system noise for various transducers. However, when operating in an oscillator configuration, normalizing the chemical (mass) sensitivity, the limit of detection is proportional to $3f_r/(S_m Q)$ [17]. Using this definition with the values of the operating frequencies, together with the values of the quality factor and the mass sensitivities S_m , one can assess the LOD for each operational mode. For example, when vibrating the microcantilever in the lateral direction compared to the transverse direction, the quality factor (Q) is larger due to the decreased viscous damping and the ratio of the resonant frequency to the chemical sensitivity (f_r/S_m) is smaller due to the decreased effective mass of the system. Furthermore, due to the increased resonant frequency and decreased effective mass of the beam/fluid system (compared with transverse mode operation), the estimated mass sensitivity of a laterally excited microcantilever is found to be much larger. For example, the mass sensitivity of a $200 \times 60 \times 5.8 \mu\text{m}^3$ cantilever is found to be roughly two orders of magnitude larger in water (4.65 Hz/pg vs. 0.0456 Hz/pg) (and even better in more viscous solutions) when laterally excited compared to transversely excited. In an oscillator configuration, the reduced frequency noise (due to the increased quality factor) and improved mass sensitivity are expected to yield much lower limits of detection. These improvements in device characteristics indicate that microcantilevers excited laterally are more suited for operating in media of high viscosities compared to microcantilevers excited transversely, and thus will result in a lower minimum detectable mass.

Based on measurements made in liquid media with the cantilevers presented in this section with various analytes, estimated LODs on the order of 100 ppb were determined, which represents roughly an order of magnitude improvement in LOD compared to values reported for out-of-plane cantilevers in water [18].

3.2. In-plane longitudinal mode

For the same reason as noted in the previous section, the longitudinal (axial) mode may be of potential interest for detection in liquid media. To assess the characteristics of this mode, self-actuated resonant-microcantilevers based on a thick-film piezoelectric layer associated with two electrodes have been processed and tested. Screen-printed microcantilevers comprising Au/PZT/Au layers are partially released from an alumina substrate (Fig. 8) using a sacrificial layer process [19].

Using an impedance analyzer (HP4194A), frequency spectra have been measured for three different cantilever geometries (piezoelectric layers of $10 \times 2 \times 0.075 \text{ mm}^3$, $8 \times 2 \times 0.075 \text{ mm}^3$ and $6 \times 1 \times 0.075 \text{ mm}^3$ coated with 5–10 μm thick symmetrical electrodes) in air and in various other fluids with viscosities ranging from 1.55 to 300 cP. Even though the quality factor in air is relatively low (probably due to internal mechanical losses and piezoelectric losses), ranging from 130 to 300, the quality factors in liquid are significantly higher compared to those of classical out-of-plane modes; the measured total quality factors for the three cantilever geometries range from 70 to 107, 41 to 51, and 20 to 22 for the 1.55, 20, and 300 cP fluids, respectively. The decrease of the resonant frequency of the first longitudinal mode from air to dodecane (1.55 cP) is only 1% and from air to 300 cP silicone oil is a mere 2.8%.

As was the case with lateral-mode silicon cantilevers having an integrated scheme for actuation and measurement, these thick-film piezoelectric cantilevers actuated in the longitudinal mode and utilizing impedance measurement are promising in terms of sensitivity and limit of detection for (bio)chemical detection in liquid media.

4. Static-mode cantilever response: influence of the viscoelasticity of polymer coatings

A static-mode, polymer-coated microcantilever undergoes a quasi-static deformation when analyte sorption causes either a change in the surface free energy (adsorption) or coating expansion (absorption). In either case, it is necessary to have an accurate model of the beam's deflection in order to design devices for high sensitivity and to analyze or predict the sensor's response. Unlike the elastic case, the stresses in the cantilever coated with a viscoelastic material will depend on time even if the absorbed analyte concentration remains constant. The coated cantilever's transient response will therefore exhibit more complex behaviors [20]. The transient response in the static mode can be of various shapes when polymeric sensitive coatings are used, which include monotonic response and response with an overshoot. An analytical solution for predicting deflection and stress in a cantilever with viscoelastic coating when the coating is subjected to an exponential eigenstrain history (i.e., sorption-induced swelling strain) has been developed (Fig. 9) which provides an explanation for the different shapes observed in the transient responses [21].

As shown in Fig. 9, the model considers that the viscoelastic material is characterized by its biaxial relaxation modulus which is defined by three parameters: the initial biaxial modulus M_0 , the steady-state biaxial modulus M_∞ and the relaxation constant time τ_R . The time constant of the swelling strain is denoted by τ_S and corresponds to the diffusion time of the analyte into the coating. Using this notation, the analytical expression of the cantilever deflection allows one to obtain an "overshoot criterion" for determining if an overshoot will occur in the quasi-static beam response. Overshoot is predicted (Fig. 10) to occur if

$$\frac{\tau_R}{\tau_S} \frac{M_0}{M_\infty} > 1 \quad (6)$$

The validation of this analytical model and its limitations has been made using finite element modeling [22].

Based on the results of the analytical model, an optimization of the choice of the mechanical viscoelastic properties of the polymer coating (instantaneous and asymptotic biaxial moduli and corresponding relaxation time) and of the coating thickness (determining the time constant of the chemical sorption into the coating and then of the eigenstrain) can be envisaged in order to have larger transient deflection and an overshoot whose characteristics could possibly be used to perform more rapid chemical detection, possibly eliminating the need to wait for the steady state to be achieved. The reduction in detection time could be especially large in the case of thick coatings and/or polymeric coatings that exhibit slow creep/relaxation.

5. Conclusion

The limitations of classical microcantilever-based microsensors are essentially due to the low selectivity and reliability of the sensitive coating, the limitation of the dynamic mode in liquid media, and the difficulty in interpreting complex static-mode responses. In the present paper, some emerging alternative uses of microcantilevers as chemical sensors have been presented. The feasibility of these unconventional uses has been confirmed by physical analytical modeling, in addition to experimental observations and/or numerical validation. These unconventional uses have some advantages (shorter response time, better reliability when sensitive coatings are not used, higher quality factor and resonant frequency in liquid for in-plane resonant modes relative to the classical out-of-plane mode, and higher signal and shorter response time utilizing the overshoot phenomenon in the static mode). However, these advantages are accompanied by some drawbacks, including lack of selectivity when sensitive coatings are not employed and the difficulty in determining the viscoelastic properties of polymer coatings for static-mode applications. The preliminary results are promising, but further measurements and modeling must be performed in order to fully realize the benefits that these unconventional uses may offer for some applications.

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List of Figure Captions

- Fig. 1.** Principle of microcantilever-based sensors for chemical detection: dynamic and static modes.
- Fig. 2.** a) cantilever geometry, b) Example of actuation force and deflection for an out-of-plane flexural mode.
- Fig. 3.** Example of (a) detection of hydrogen in nitrogen and (b) carbon dioxide in nitrogen with an uncoated 2000 x 400 x 15 μm^3 silicon cantilever
- Fig. 4.** Example of detection of small concentration of hydrogen in nitrogen with an uncoated 2000 x 400 x 15 μm^3 silicon cantilever
- Fig. 5.** Out-of-plane and in-plane modes. a) transverse mode (out-of-plane) b) lateral bending mode (in-plane), c) longitudinal (elongation) mode (in-plane)
- Fig. 6.** Hydrodynamic forces acting on the cantilever in lateral bending mode. (Only lateral forces indicated.)
- Fig. 7.** Results of analytical model compared with experimental results for microcantilevers in lateral (in-plane) flexure [13]: (a) quality factor in water, (b) resonant frequency in water, (c) relative frequency decrease from air to water.
- Fig. 8.** Example of screen printed piezoelectric cantilever designed for axial-mode operation [12]
- Fig. 9.** Modeling of the static mode. a) Cantilever profile, b) material properties: biaxial moduli (biaxial stress corresponding to a unit step-

function biaxial strain) of viscoelastic coating (pink curve) and elastic base material (blue curve) [18].

Fig. 10. Cantilever deflection obtained with the analytical model

List of Figures

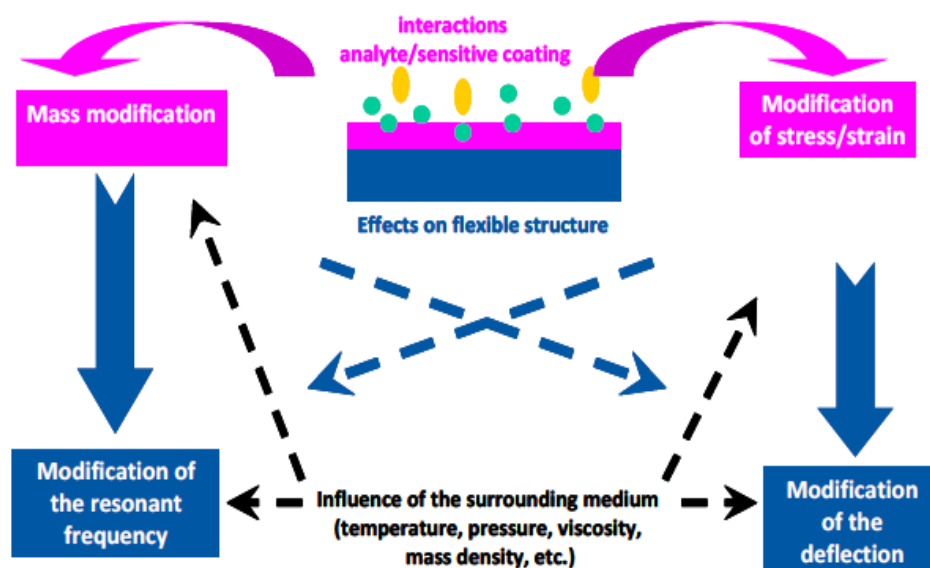


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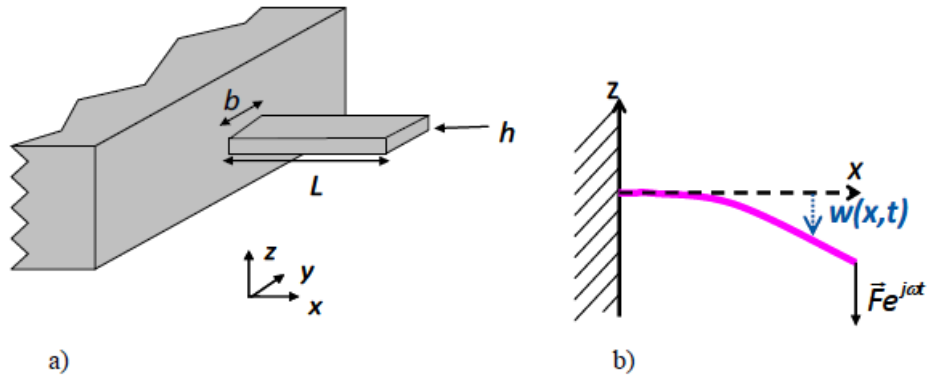
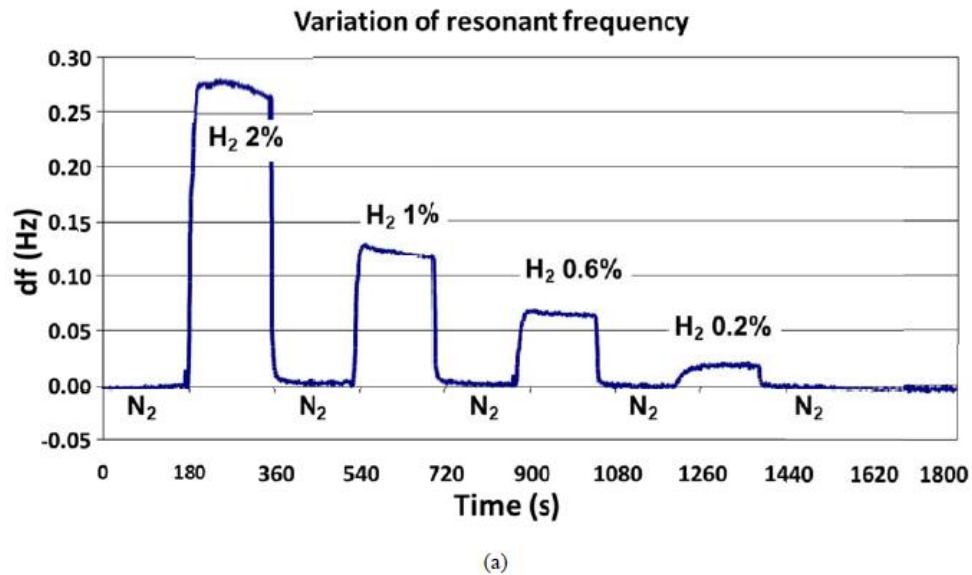


Fig. 2. a) cantilever geometry, b) Example of actuation force and deflection for an out-of-plane flexural mode.



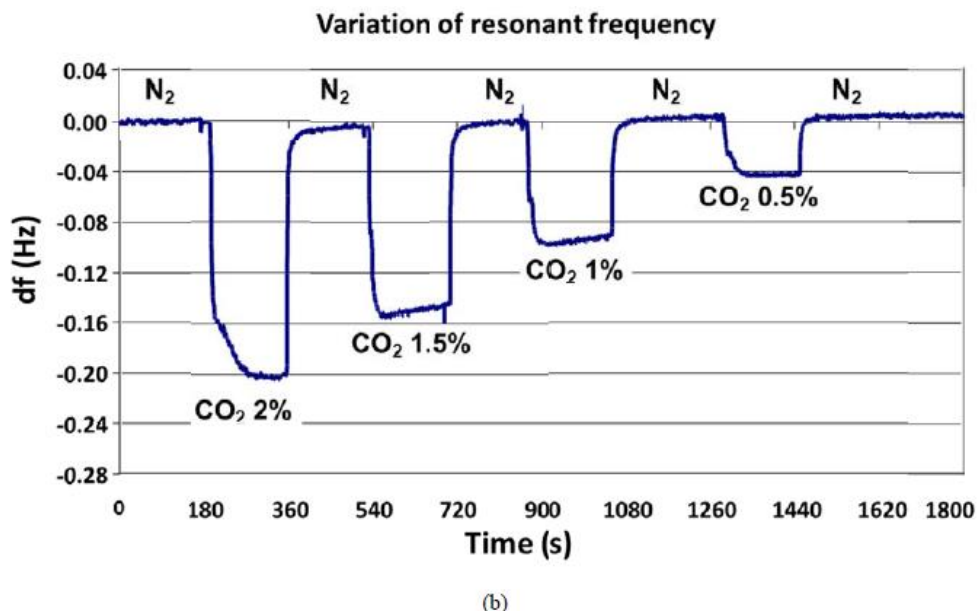


Fig. 3. Example of (a) detection of hydrogen in nitrogen and (b) carbon dioxide in nitrogen with an uncoated 2000 x 400 x 15 μm^3 silicon cantilever

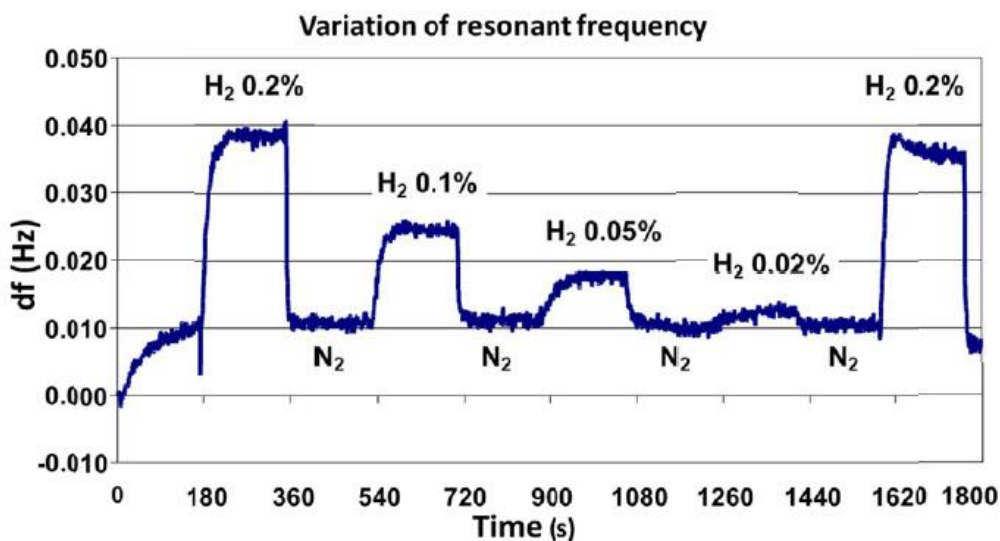


Fig. 4. Example of detection of small concentration of hydrogen in nitrogen with an uncoated 2000 x 400 x 15 μm^3 silicon cantilever

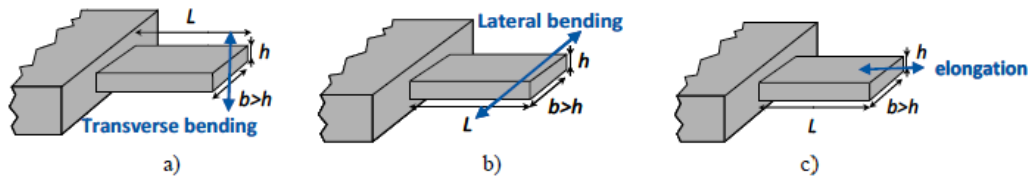


Fig. 5. Out-of-plane and in-plane modes. a) transverse mode (out-of-plane) b) lateral bending mode (in-plane), c) longitudinal (elongation) mode (in-plane)

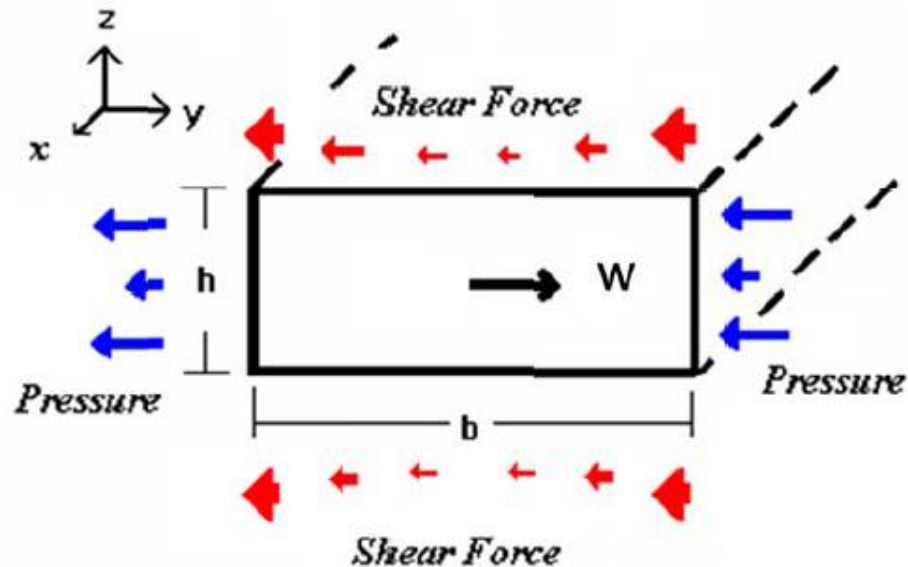
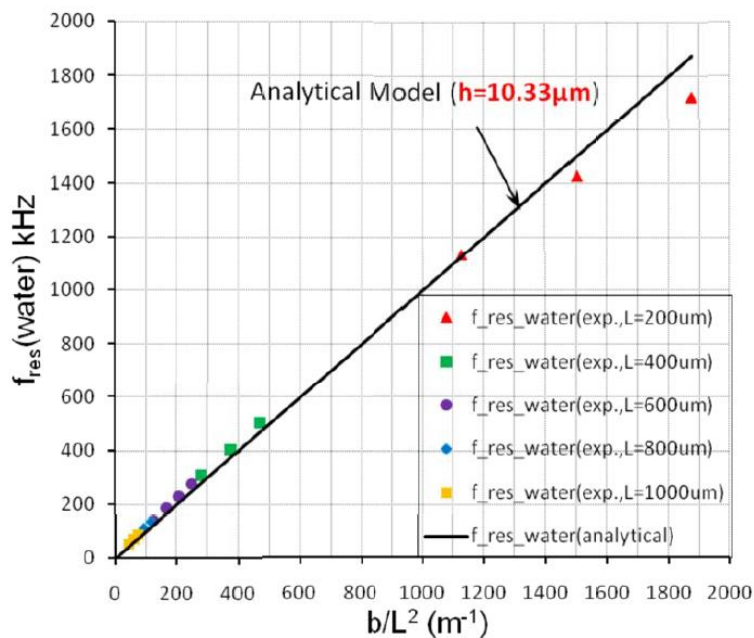
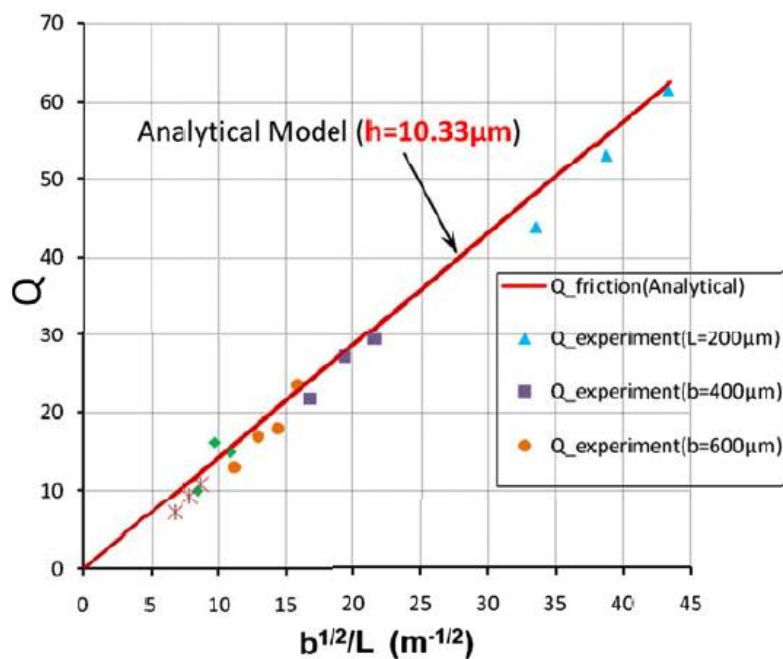
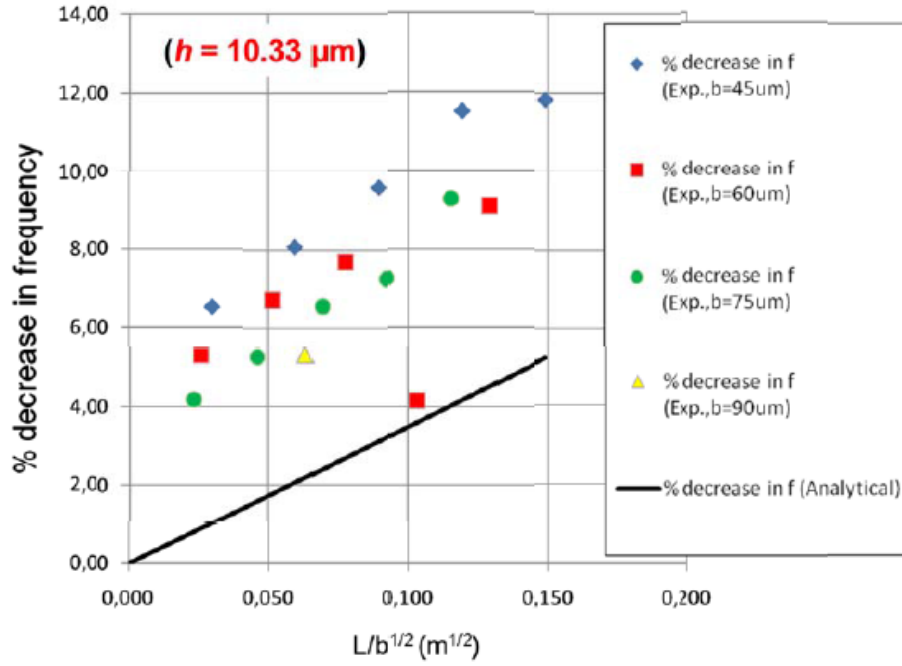


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(c)

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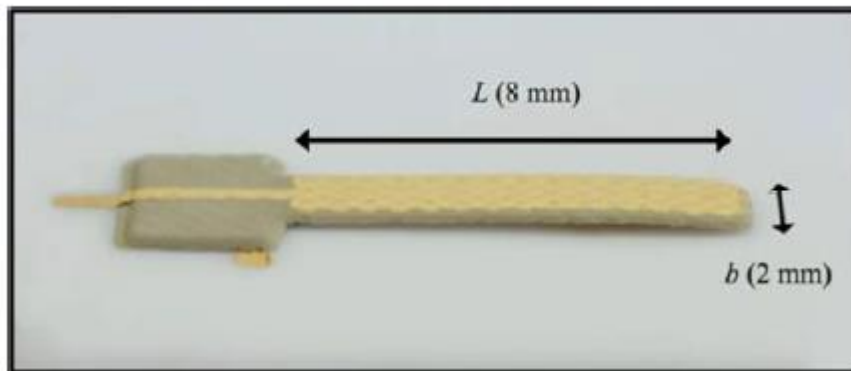


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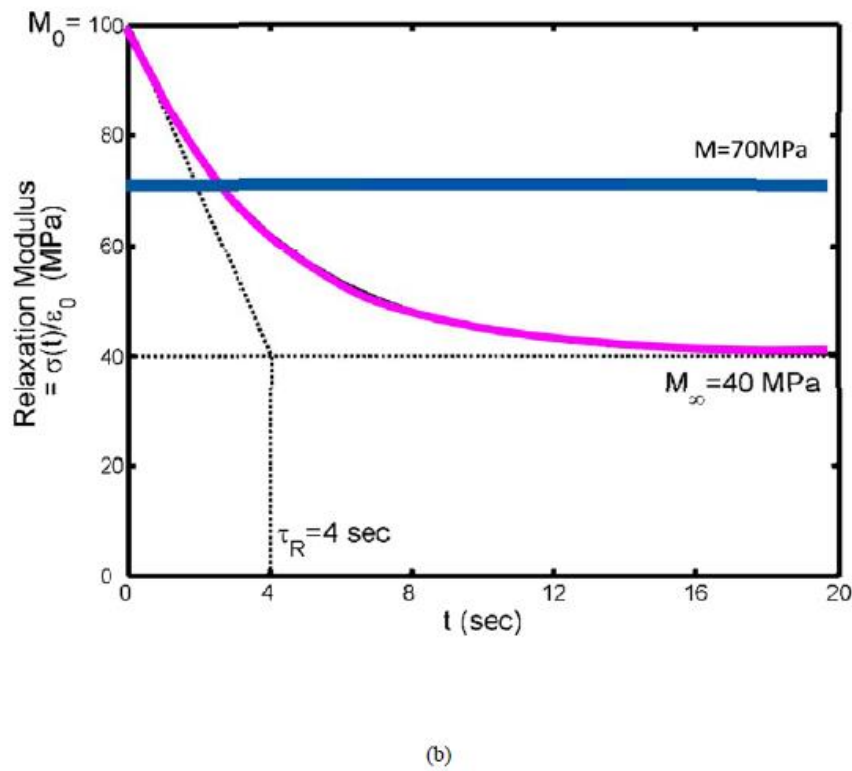
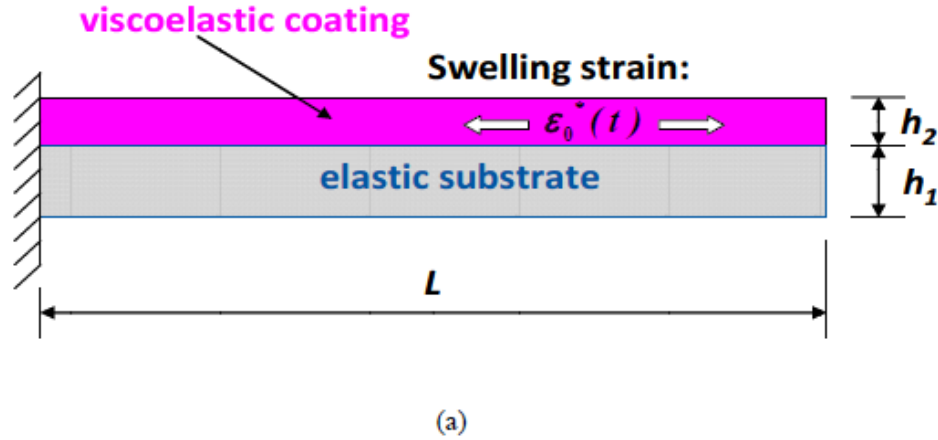


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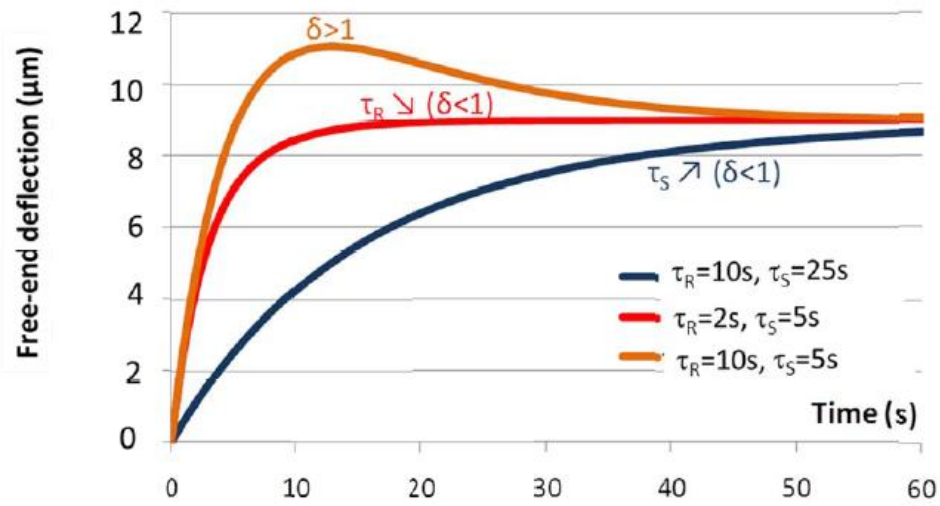


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