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Effect of Coating Viscoelasticity on Quality Factor and Limit of Detection of Microcantilever Chemical Sensors

Isabelle Dufour, Frédéric Lochon, Stephen M. Heinrich, Fabien Josse, *Senior Member, IEEE*, and Dominique Rebière

Abstract—Microcantilevers with polymer coatings hold great promise as resonant chemical sensors. It is known that the sensitivity of the coated cantilever increases with coating thickness; however, increasing this thickness also results in an increase of the frequency noise due to a decrease of the quality factor. By taking into account only the losses associated with the silicon beam and the surrounding medium, the decrease of the quality factor cannot be explained. In this paper, an analytical expression is obtained for the quality factor, which accounts for viscoelastic losses in the coating. This expression explains the observed decrease of the quality factor with increasing polymer thickness. This result is then used to demonstrate that an optimum coating thickness exists that will maximize the signal-to-noise ratio and, thus, minimize the sensor limit of detection.

Index Terms—Beam vibrations, chemical sensors, fluid losses, limit of detection (LOD), microcantilevers, polymer coatings, quality factor, resonant frequency, sensitivity, viscoelastic losses.

I. INTRODUCTION

RESONANT microcantilever-based sensors have emerged as a new sensitive detection technique. In chemical sensing applications, the device consists of a microcantilever and a chemically sensitive coating, which absorbs the molecule of interest (Fig. 1). The absorbed molecules can then be detected by monitoring the shift in the mechanical resonant frequency [1]–[9].

The choice of the coating thickness for a maximum signal-to-noise ratio (SNR) and, thus, a minimum limit of detection (LOD), is known to be a difficult task because of the various effects that the coating thickness has on the sensor characteristics. In particular, the sensor sensitivity increases with coating thickness, but the frequency noise also increases.

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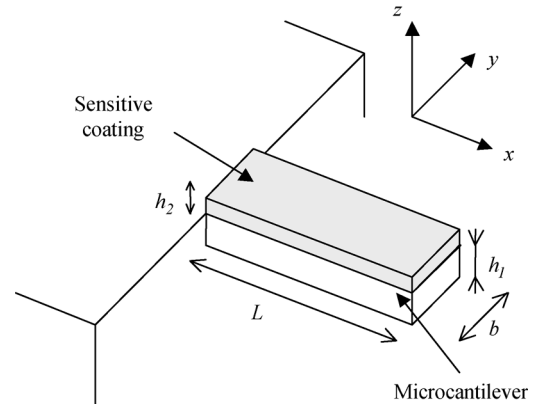


Fig. 1. Geometry of the cantilever with the sensitive coating.

The aim of this paper is to model the coating as a viscoelastic layer to determine if the associated losses are of sufficient magnitude to account for the observed decrease in the quality factor and to demonstrate that there exists an optimal coating thickness leading to the minimal LOD. In the first part of this paper, a summary of some recently developed analytical results is presented for a two-layer, hybrid (elastic/viscoelastic) beam, the results of which show the effect of the viscoelasticity of the coating on the sensor quality factor Q . In the second part, theoretical results are then compared with preliminary measurements. Then, using the analytical expression of the sensor quality factor, the dependence of the LOD on the coating thickness is derived for a resonant frequency-based sensor for the cases of: 1) intrinsic noise and 2) operation within an oscillator configuration.

II. EFFECT OF SENSITIVE-LAYER VISCOELASTICITY ON SENSOR QUALITY FACTOR

A. Quality Factor

The total quality factor of a damped system is given by [10]

$$Q_{\text{total}} = \frac{2\pi W_{\text{total}}}{\Delta W_{\text{total}}} \quad (1)$$

where W_{total} is the stored vibrational energy and ΔW_{total} the total energy lost per cycle of vibration.

The total energy lost ΔW_{total} can be written as

$$\Delta W_{\text{total}} = \sum_i \Delta W_i \quad (2)$$

where ΔW_i represents the energy lost due to the different mechanical loss mechanisms: thermoelastic losses in the microcantilever [11], viscous [12] and acoustic losses [13] in the surrounding medium, and losses due to radiation of elastic waves at the support [14].

Each of these loss mechanisms has an associated quality factor $Q_i = 2\pi W_{\text{total}}/\Delta W_i$, and the overall quality factor Q_{total} is obtained by

$$\frac{1}{Q_{\text{total}}} = \sum_i \frac{1}{Q_i}. \quad (3)$$

The quality factor is a measure of the spread of the resonance peak. The smaller the quality factor, the greater the energy lost in the resonant sensor system and the wider the resonance peak. Typically, the peak width and, thus, the quality factor of the sensor is measured at the 3-dB point of the amplitude spectrum, which is the point at which the amplitude of the response is $1/\sqrt{2}$ times the maximum (resonant) amplitude.

If only viscous losses, acoustic losses, support losses, and thermoelastic losses are considered, the quality factor increases with added mass. Thus, the observed decrease in the quality factor as coating thickness increases must be explained by taking into account another loss phenomenon. It is hypothesized that this decrease is primarily due to internal losses in the viscoelastic sensitive coating.

B. Viscoelastic Losses

The hybrid (elastic/viscoelastic) beam can be assumed to be replaced by an equivalent homogeneous viscoelastic beam whose complex flexural rigidity, $(EI)^*$, is given as [15]

$$(EI)^* = E_1 I_1 + E_2'(\omega) I_2 + j E_2''(\omega) I_2 \quad (4)$$

where E_1 is the Young's modulus of the elastic material and $E_2' + j E_2''$ the complex Young's modulus of the viscoelastic sensitive layer. I_1 and I_2 are the moments of inertia of the elastic and viscoelastic beam layers given, respectively, by

$$I_1 = \frac{b h_1^3}{12} + b h_1 \left(h_N - h_2 - \frac{h_1}{2} \right)^2 \quad (5)$$

$$I_2 = \frac{b h_2^3}{12} + b h_2 \left(h_N - \frac{h_2}{2} \right)^2 \quad (6)$$

where b is the beam's width. The geometric properties (5) and (6) are with respect to an equivalent fixed neutral axis, which is associated with the time-varying neutral axis of the hybrid beam. The position of this axis is given by the coordinate h_N , which is measured from the top of the cross section

$$h_N = \frac{h_2}{2} + \frac{h_1 E_1 (h_1 + h_2) (h_1 E_1 + h_2 E_2')}{2 (h_1 E_1 + h_2 E_2')^2 + h_2^2 E_2''^2}. \quad (7)$$

Using the complex flexural rigidity given by (4), the equation of motion of a harmonically excited hybrid beam, considering

the only loss mechanism to be associated with the loss modulus of the sensitive layer, takes the well-known form (e.g., [16])

$$(EI)^* \frac{\partial^4 w(x, t)}{\partial x^4} + m_L \frac{\partial^2 w(x, t)}{\partial t^2} = F(x) e^{j\omega t} \quad (8)$$

where $w(x, t)$ is the (complex and harmonically varying) transverse displacement, $F(x)$ is the arbitrary distribution of the force amplitude, m_L is the mass per unit length of the beam (including coating), and ω is the angular forcing frequency. Following standard procedures for solving (8) (e.g., [16]), an expression for the resonant frequency f_{res} can be obtained

$$f_{\text{res}} = \frac{1.875^2}{2\pi L^2} \sqrt{\frac{E_1 I_1 + E_2' I_2}{m_L}}. \quad (9)$$

Mathematically, the expression for the quality factor is given by

$$Q = \frac{f_{\text{res}}}{\Delta f_{-3 \text{ dB}}} \quad (10)$$

where $\Delta f_{-3 \text{ dB}}$ is the frequency bandwidth taken with 3-dB attenuation from maximum gain.

The magnitude of the deflection curve near the resonance peak is given by the solution of (8) and allows the use of (10) to obtain an expression for the quality factor associated with the viscoelastic losses in the sensitive layer, Q_{viscoel} [15]

$$Q_{\text{viscoel}} = \frac{1}{2 \left(1 - \sqrt{1 - \frac{E_2'' I_2}{E_1 I_1 + E_2' I_2}} \right)}. \quad (11)$$

For a typical silicon cantilever and a polymeric-sensitive coating, $E_2' \ll E_1$ and $E_2'' \ll E_1$. This provides the motivation to consider a first-order approximation to (9) and (11) as

$$f_{\text{res}} \approx \frac{1.875^2}{2\pi L^2} \sqrt{\frac{E_1 h_1^3}{12(\rho_1 h_1 + \rho_2 h_2)}} \times \left(1 + \frac{h_2 (h_2^2 + 3(h_1 + h_2)^2) E_2'}{2h_1^3 E_1} \right) \quad (12)$$

$$Q_{\text{viscoel}} \approx \frac{h_1^3}{h_2 (h_2^2 + 3(h_1 + h_2)^2) E_2''}. \quad (13)$$

Equation (13) clearly shows that the quality factor due to coating losses decreases with increasing coating thickness. In the case of a small coating thickness $h_2 \ll h_1$, the same expression as that of surface losses developed in [10] and [17] is found. An advantage of (11) is that it is valid for more general values of thicknesses and moduli for the hybrid beam.

C. Total Sensor Quality Factor

As explained previously, when a microcantilever resonates in a gas or liquid medium, there are different mechanical loss mechanisms: viscoelastic and thermoelastic losses in the microcantilever, viscous and acoustic losses in the surrounding

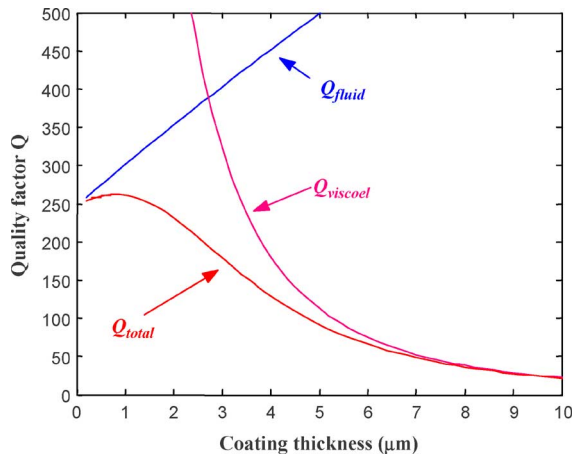


Fig. 2. Quality factors versus coating thickness (PIB coating, silicon cantilever $4 \mu\text{m} \times 50 \mu\text{m} \times 300 \mu\text{m}$ in air).

medium, and losses due to radiation of elastic waves at the support. Each of these loss mechanisms has an associated quality factor Q_i and the overall quality factor Q_{total} is obtained by (3).

In the case of chemical sensors, the surrounding medium is usually either a gas at atmospheric pressure or a liquid. Consequently, without the sensitive coating, the dominant losses are due to viscous damping, Q_{fluid} [18]. The principal aim of this paper is to understand the modification of the total quality factor when a sensitive coating is added. Therefore, the effects of the additional mass, stiffness, and viscoelastic losses of the coating on the quality factor have been incorporated using the proposed model; in addition, the viscous losses due to the surrounding fluid have also been considered by using Sader's approach [12].

In Fig. 2, the two quality factors, as well as the total quality factor using (3), Q_{total} , are plotted as a function of the viscoelastic layer thickness. These plots and other simulations in this paper are based on a polyisobutylene (PIB) coating with storage and loss moduli of $E'_2 = 43 \text{ MPa}$ and $E''_2 = 65 \text{ MPa}$ at the resonant frequency of 58 kHz [19], [20] and a silicon substrate with $E_1 = 150 \text{ GPa}$. In addition, all simulations are made for an air environment (air parameters: mass density 1.29 kg/m^3 and viscosity $1.8 \times 10^{-5} \text{ kg/m-s}$) and, unless stated otherwise, for a $4 \mu\text{m} \times 50 \mu\text{m} \times 300 \mu\text{m}$ beam geometry (not including coating). Fig. 2 clearly shows that, if only viscous damping of the surrounding fluid (here air) is considered, the quality factor increases with the added mass of the sensitive layer. However, when the viscoelasticity of the coating is taken into account, the simulations show that the inclusion of the viscoelastic layer losses does indeed result in a decrease in the total quality factor as coating thickness increases. Thus, the new hybrid beam model is capable of explaining the observed decrease of the quality factor in experiments. In Section III, a quantitative comparison will be made between the results of the hybrid beam model and experimental results on quality factor.

III. PRELIMINARY EXPERIMENTAL RESULTS

In order to validate (11) for the quality factor associated with viscoelastic coating losses in the layer, measurements were made on a silicon microcantilever of relatively large dimension ($L = 6000 \mu\text{m}$, $b = 200 \mu\text{m}$, $h_1 = 221 \mu\text{m}$) for which values

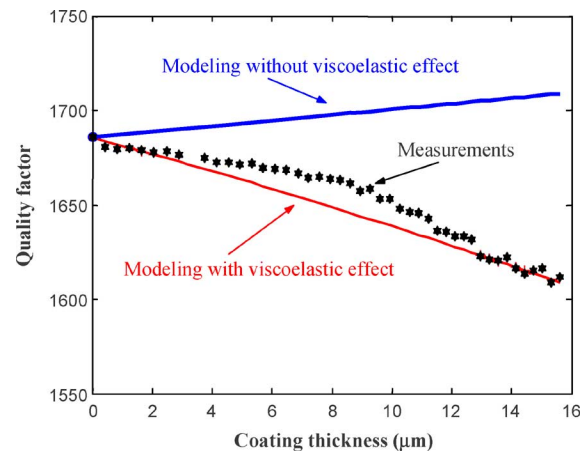


Fig. 3. Total quality factor versus coating thickness (PIB coating and silicon cantilever) [21]. Measurements and modeling (with and without viscoelastic losses) are shown for an air environment.

of $f_{\text{res}} = 7.3 \text{ kHz}$ and $Q_{\text{total}} = 1686$ have been measured in air. (Fabrication limitations in our facilities required that the device dimensions be somewhat larger than are typically found in microcantilever applications.) PIB coatings of various thicknesses were sprayed onto the cantilever, and the quality factors were measured with a gain/phase analyzer (HP 4194A). The measurements are presented in Fig. 3. The modeling results with and without the viscoelastic losses (using $E'_2 = 11.4 \text{ MPa}$ and $E''_2 = 19.6 \text{ MPa}$, which correspond to 7.3 kHz [19], [20]) are also shown.

The observed agreement shown in Fig. 3 strongly suggests that the viscoelastic loss mechanism is the primary factor responsible for the observed decrease in the (total) quality factor. This is true even if the quality factor associated to the viscoelastic losses is very large (more than 27 500 in the present example) compared with the total quality factor (less than 1686). The simulation for smaller cantilevers presented in Fig. 2 shows a more important decrease of the total quality factor. While measurements with such microcantilevers have not been performed in the present work, Lange *et al.* [2] have observed, from such measurements in air, that the quality factor was approximately 950 without coating and decreased to 400 with $10 \mu\text{m}$ of polymer (PEUT).

In conclusion, (3) and (11) should be used in obtaining the total quality factor accounting for layer losses. All other loss mechanisms may be accounted for through appropriate expressions [18].

An important area of practical application for the results in this study is in specifying the appropriate coating thickness for the best performance of the coated cantilever as a chemical sensor. Because the sensor signal (frequency shift) increases with coating thickness within practical ranges, one might wrongly assume that a continued increase in coating thickness will continue to improve the sensor performance. However, the analysis of a hybrid (elastic/viscoelastic) beam presented in this work indicates that the quality factor decreases with the viscoelastic coating thickness, which results in an increase in the frequency noise of an oscillator system with the coated beam as the frequency-determining element. In other words, the

decrease in quality factor caused by the coating losses results in a less precise measurement of the frequency shift. Thus, an optimum coating thickness that will maximize the SNR, hence minimizing the LOD of those devices in sensor applications, could exist. In fact, the existence of such an optimum coating thickness has been shown experimentally in coated microcantilevers [2], [22]. In Section IV, the analytical expression of the quality factor developed in this paper is used to predict such an optimum.

IV. DEPENDENCE OF LOD ON COATING THICKNESS

The choice of a coating thickness that minimizes the LOD is known to be a difficult task because of the various effects the thickness has on sensor characteristics. The aim of this section is to theoretically demonstrate using (11) that there exists such an optimal coating thickness that minimizes the LOD.

It is noted that the LOD is defined as the smallest amount of a particular substance that is detectable by the device. The LOD is thus inversely proportional to the SNR, which, in turn, depends on the quality factor and on the sensitivity. First the analytical expression of the sensor sensitivity is presented and then the LOD is studied in two cases: 1) the case of intrinsic noise which is observed in direct spectrum analysis and 2) the case of operation within an oscillator configuration.

A. Sensor Sensitivity

When a resonant microcantilever with sensitive coating is placed in a gas or liquid environment with target molecules, some of the target molecules are adsorbed by the sensitive layer. As a result, the microcantilever's mass and stiffness may be modified. The primary cause of the decrease of the resonant frequency is the mass variation [2]–[7]. Then, in a first-order approach, the stiffness variation can be neglected and only the mass variation is taken into account. Assuming that the partition coefficient is constant for the analyte concentration range that is used, using (9), the resonant frequency in presence of analyte can be expressed by

$$f_{\text{res}}(C_A) = \frac{1.875^2}{2\pi L^2} \sqrt{\frac{E_1 I_1 + E_2' I_2}{\rho_1 h_1 + (\rho_2 + KC_A) h_2}} \quad (14)$$

where C_A is the analyte concentration in the fluid [mass per unit volume], K is the (dimensionless) partition coefficient of the coating/analyte pair in the fluid environment, h_1 and h_2 are the microcantilever and sensitive coating thicknesses, and ρ_1 and ρ_2 are the respective mass densities.

Then, using (14) and the definition of the sensitivity and assuming that the mass increase is small compared with the coating mass ($KC_A \ll \rho_2$), the sensitivity S of microcantilever-based chemical sensors is given by

$$S \equiv \frac{\Delta f}{C_A} = \frac{Kh_2 f_{\text{res}}(0)}{2(\rho_1 h_1 + \rho_2 h_2)} \quad (15)$$

where $f_{\text{res}}(0)$ is the resonant frequency without analyte and Δf is the frequency shift due to analyte sorption, and is plotted in Fig. 4. According to (15), the sensitivity may be improved by

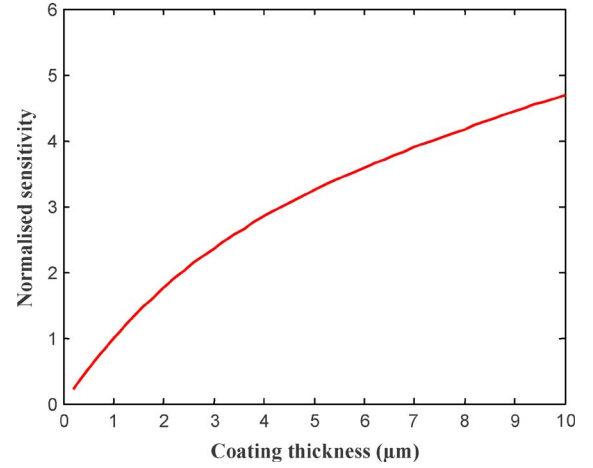


Fig. 4. Normalized sensitivity versus coating thickness (PIB coating, silicon cantilever $4 \mu\text{m} \times 50 \mu\text{m} \times 300 \mu\text{m}$). Normalization: $S = 1$ for $h_2 = 1 \mu\text{m}$.

increasing the thickness h_2 of the polymeric sensitive coating, provided that $\rho_2 h_2 < 2\rho_1 h_1$ [1]. This analytical expression of the sensitivity has been verified experimentally in [7] and [22], which validate the fact that the mass effect is predominant and that the mass increase of the coating is small compared with the coating mass.

B. Intrinsic Noise

Here, the focus is on the intrinsic noise mechanisms since they determine the ultimate limits of the sensor's performance. When a microcantilever is in an ambient thermal environment, there is a continuous exchange of the mechanical energy accumulated in the microcantilever and the thermal energy of the environment. This exchange results in spontaneous microcantilever vibration. Due to this energy exchange, the resonant frequency f_{res} is subjected to frequency fluctuations $\Delta f_{\text{noise_intrinsic}}$ given by [3], [23], [24]

$$\Delta f_{\text{noise_intrinsic}} = \frac{1}{z_{\text{max}}} \sqrt{\frac{f_{\text{res}} k_B T B}{2\pi k Q_{\text{total}}}} \quad (16)$$

where k_B is the Boltzmann constant, T is the absolute temperature, B is the measurement bandwidth, k is the microcantilever stiffness (in the hybrid beam case $k = 3(E_1 I_1 + E_2' I_2)/L^3$), and z_{max} is the noise-related microcantilever vibration amplitude (mean-square amplitude of the self-oscillating cantilever).

The LOD is usually defined as the analyte concentration corresponding to a frequency shift equal to three times the frequency noise of the system measurement. Thus, (15) and (16) may be combined to yield the sensor's LOD when intrinsic noise is considered

$$\text{LOD}_{\text{noise_intrinsic}} \equiv \frac{3\Delta f_{\text{noise_intrinsic}}}{S} \propto \frac{\rho_1 h_1 + \rho_2 h_2}{h_2 \sqrt{k} f_{\text{res}} Q_{\text{total}}} \quad (17)$$

In (17), only the terms depending on the coating thickness are kept in order to study the LOD dependence on the coating thickness. Implicit in the derivation of (17) is the assumption that the dependence of z_{max} on the coating thickness is negligible. This

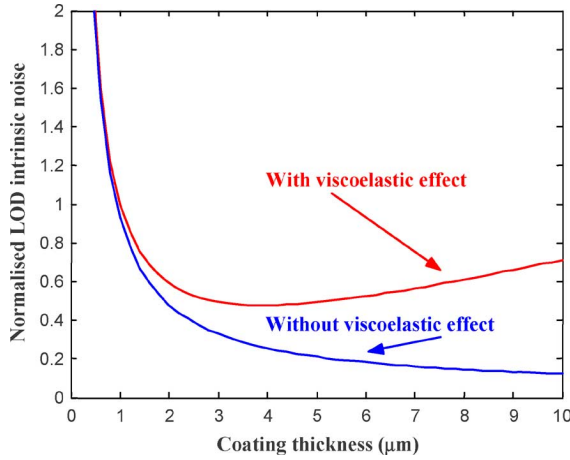


Fig. 5. Normalized limit of detection versus coating thickness in the case of intrinsic noise (PIB coating, silicon cantilever $4 \mu\text{m} \times 50 \mu\text{m} \times 300 \mu\text{m}$). Normalization: $\text{LOD} = 1$ for $h_2 = 1 \mu\text{m}$.

expression allows the determination of the optimum sensitive coating thickness for minimum LOD.

The case of a PIB coating on a silicon microcantilever $4 \mu\text{m} \times 50 \mu\text{m} \times 300 \mu\text{m}$ is presented in Fig. 5. Due to the viscoelastic effect, an optimum coating thickness exists, but, if the coating thickness exceeds the optimal value, the resulting increase in LOD is not very significant.

C. Oscillator Configuration

Usually, in order to accurately measure the resonant frequency change, the microcantilever is inserted into the feedback loop of an oscillator (as the frequency determining element). The output signal is then the oscillation frequency. For a high degree of accuracy, the oscillator must be as stable as possible. In fact, for a given amplifier, the frequency noise is essentially due to the variation of the amplifier phase $\delta\theta$. According to the Barkhausen condition, which is satisfied in all oscillators, the frequency noise, $\Delta f_{\text{noise_oscil}}$, can be expressed with a first-order limited development (small phase variations)

$$\Delta f_{\text{noise_oscil}} \approx \frac{-\delta\theta}{\left(\frac{\partial\Phi}{\partial f}\right)_{f_0}}. \quad (18)$$

The expression of the phase of the micromechanical resonator near the resonant frequency allows one to obtain the expression of the oscillator stability, $\Delta f_{\text{noise_oscil}}$, as a function of the resonant frequency and quality factor [25]

$$\Delta f_{\text{noise_oscil}} \approx \frac{f_{\text{res}}\delta\theta}{2Q_{\text{total}}}. \quad (19)$$

As in the case of intrinsic noise, the frequency fluctuation $\Delta f_{\text{noise_oscil}}$ can be used to relate the LOD to h_2 :

$$\text{LOD}_{\text{noise_oscil}} \equiv \frac{3\Delta f_{\text{noise_oscil}}}{S} \propto \frac{\rho_1 h_1 + \rho_2 h_2}{h_2 Q_{\text{total}}}. \quad (20)$$

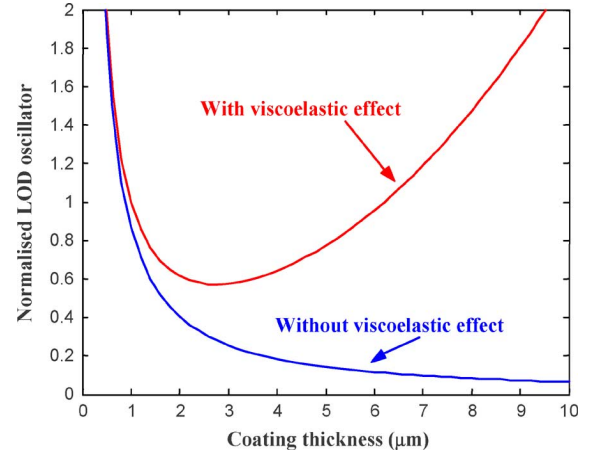


Fig. 6. Normalized limit of detection versus coating thickness in the case of an oscillator configuration (PIB coating, silicon cantilever $4 \mu\text{m} \times 50 \mu\text{m} \times 300 \mu\text{m}$). Normalization: $\text{LOD} = 1$ for $h_2 = 1 \mu\text{m}$.

Expression (20) may be used to determine the optimum sensitive coating thickness for minimum LOD. The case of a PIB coating on a silicon microcantilever ($4 \mu\text{m} \times 50 \mu\text{m} \times 300 \mu\text{m}$) is presented in Fig. 6. Clearly, when the viscoelastic losses are included, an optimum coating thickness exists. However, the oscillator configuration case differs significantly from the intrinsic noise case, in that an increase in coating thickness beyond the optimal value may seriously compromise the LOD. This phenomenon has been observed experimentally in [2] and [22].

V. CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORK

A new analytical model for the characteristics of a coated microcantilever sensor has been presented for the purpose of quantifying the effect of coating viscoelasticity on the resonant frequency and quality factor. Previous models that have only included energy losses in the surrounding fluid have been unable to reproduce the trend of decreasing quality factor with increasing coating thickness, although such a relationship has been observed experimentally in gas environments. The present model overcomes this defect by modelling the coating as viscoelastic, thereby including the coating's inherent losses in the formulation.

The major conclusions provided by the study include the following.

- The losses in the coating appear to be the primary factor responsible for the qualitative trend observed in gaseous environments that the quality factor decreases as the coating thickness increases.
- Preliminary results show that the new model is capable of giving excellent quantitative agreement with experimental data for the quality factor. Additional experimental work is necessary to confirm the model over broader ranges of coating thickness.
- The new model predicts the existence of an optimal coating thickness in the sense of maximizing the SNR and, thus, minimizing the limit of detection of the sensor; this provides a theoretical basis for previous experimental results

that have suggested that the LOD has a relative minimum with respect to coating thickness.

Based on the results of the present study, additional research is recommended on the following topics.

- The derivation of a general analytical expression for determining the value of the optimal coating thickness is warranted.
- The present model clearly shows that the coating losses cannot be ignored relative to those in the surrounding air, i.e., that the viscoelastic quality factor is sufficiently small compared with the quality factor based on fluid losses alone that it should be included. However, as losses in the surroundings are increased—for example, as the environment changes from gas to liquid—the losses in the coating are expected to be less important. The development of useful guidelines in this regard would therefore be a welcome addition to the sensors literature.

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