



Transmittance correlation of porous silicon multilayers used as a chemical sensor platform

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ABSTRACT

This work presents a system of two optical microcavities made of mesoporous silicon that have been analyzed as a platform for either chemical sensing or biosensing. When a porous microcavity is exposed to an analyte, the effective refractive index of its layers change, and its optical transmittance shifts towards lower wavenumbers. We constructed a device that employs two identical porous silicon microcavities, one of them is allowed to be in contact with the analyte, whereas the other remains unexposed. The transmitted intensity of the system results in the integrated product of the transmittances of both multilayers, which can be approximated to the autocorrelation function of the transmittance of the microcavity. Its value depends on the analyte concentration, so it can be used for sensing purposes. This results in a sensor that requires neither a wavelength-sensitive detector nor a monochromatic source of illumination, and is robust to changes in temperature, because it only depends on the relative changes in the microcavities. The sensor's response can be optimized by modifying the angular position of the second microcavity. A sensor based on this principle is demonstrated for isopropyl alcohol detection. The minimum concentration change that can be measured is about 30 ppm, which is equivalent to a minimum measurable change of refractive index of 5×10^{-5} .

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1. Introduction

Since the observation of photoluminescence from the porous silicon, there has been much work on its structural and optical properties [1–3]. Porous silicon (PS) thin films are usually fabricated via the electrochemical anodization of single crystalline silicon (c-Si) wafers in a fluorine-containing electrolyte [2]. The porosity of PS (i.e. the ratio between the void space volume and the total volume of the material), depends on the preparation conditions such as the electrolyte composition and current density. Since the PS structure is much smaller than the infrared light wavelengths, the PS behaves as an effective medium for such wavelengths. The dielectric constant is an average between the constants of the silicon, air and

other components eventually present in the inner pore surfaces. Therefore, its optical properties can be estimated using effective medium theories. The complex dielectric function of the PS (ε_{ps}) can be obtained by an appropriate effective medium model given the porosity and the dielectric functions of the components are known [1]. An important feature of the PS preparation process is that the tailored in-depth profiles of the porosities can be obtained using a suitable current density time profile during anodization [3–5]. This process is self-limiting, which means that the current density changes during the fabrication process, which leads to changes in the morphology, but does not alter the already-fabricated PS structure. This characteristic, along with its large internal surface area and high chemical reactivity make PS a very attractive material for gas sensing applications. By controlling the current density values and etching times, it is possible to produce multilayered structures with pre-designed optical thicknesses. These multilayers can be designed to produce specific optical responses. It is also possible, using high current pulses, to remove the porous multilayers that preserve their entire structure from the c-Si to transfer it to another

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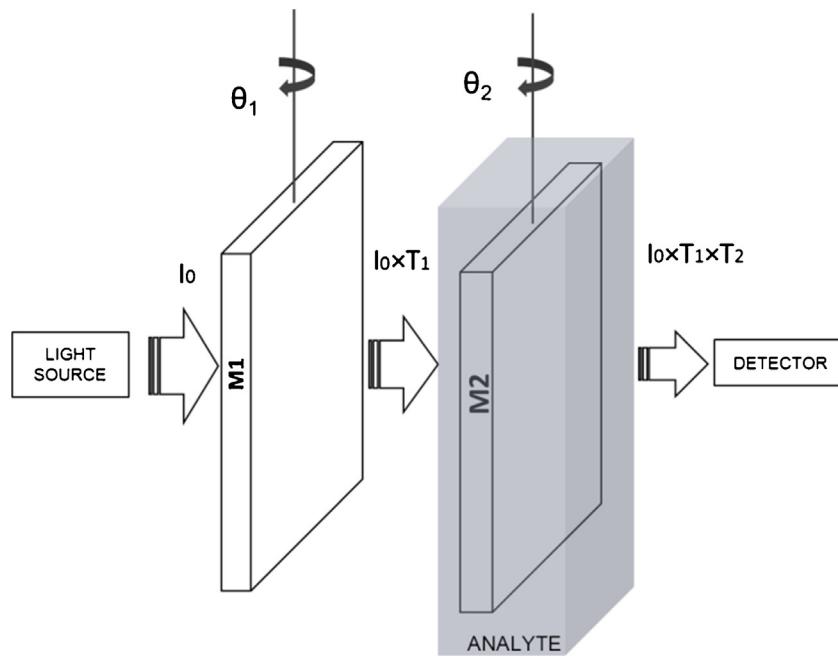


Fig. 1. Scheme of the sensor; two microcavities M_1 and M_2 are placed sequentially to form a composed filter to an incident light beam. The transmitted intensity will be the integral of the product between the intensity of the incident radiation I_0 and the transmittance of the system $T_1 \times T_2$. The second microcavity can be exposed to a contaminated atmosphere with a specific analyte. Both microcavities can be tilted to control the output intensity and the sensor's range of operation.

substrate [4,5]. Optical microcavities present sharp resonances in their optical transmittance and reflectance spectra because of the photon confinement inside their cavities [6]. These features confer the required sensitivity to PS devices for their use as label-free external analyte (EA) sensors, from changes in the average refractive index of the porous matrix. The interest in the research of PS optical gas sensors was initially stimulated by the 1997 work of Zangoie et al., during the detection of vapors using spectroscopic ellipsometry [7]. Recently, many works have been presented that use multilayer PS structures as optical-based sensors. Kim et al. [8], Ivanov et al. [9] and Kovacs et al. [10] developed a Bragg reflector and the measured reflectance was used to detect shifts in the reflection bandwidth due to interactions with EA. Park et al. [11] developed rugate filters and measured the evolutions of the peak wavelength in the reflectivity spectra for isopropanol vapor detection. However, for all these works, the systems require a dispersive element such as a spectrometer that senses the refractive index change [12]. In this article, we propose a method for sensing analytes, based on the effect of the decorrelation of the transmittances of the two optical microcavities made of porous silicon. We use a device that employs two identical porous silicon optical microcavities, one of which remains unexposed while the other is allowed to make contact with the analyte. An LED diode with an emission spectrum centered on the microcavity resonance was used as a light source, and the transmitted intensity was measured using a phototransistor. The system's total transmittance depends on the analyte concentration, and so it can be used for sensing. Here, we report a study of the performance of the sensor based on this principle when exposed to a mixture of isopropyl alcohol vapor and nitrogen. The proposed system is inherently stable against temperature changes, since the sensor detects the relative displacement of the transmittance spectra. Additionally, the proposed method does not need to resolve the shift of the transmittance spectrally; i.e., it is not necessary to have a dispersive element or a wavelength-sensitive detector. However, it is possible to determine sub-nanometer shifts of the exposed microcavity spectrum from the resulting signal.

2. Material and methods

2.1. Theoretical considerations

One of the characteristics of the electromagnetism of dielectric media is that, since there is no fundamental length scale other than the assumption that the system is macroscopic; simple scaling rules can be stated [13]. When a freestanding optical microcavity made of porous silicon is transferred to a transparent substrate and exposed to an EA, the effective dielectric function of all the layers in the multilayer change. Such a change results in the modification of the multilayer's transmittance spectrum. According to the scaling rules, if the change of the refractive index is small enough, the modification of the transmittance spectrum can be approximated to a rigid translation to lower wavenumbers by the following expression:

$$T'(k) \cong T(k + \delta) \quad (1)$$

where $T(k)$ is transmittance of an optical microcavity as a function of the wavenumber. Consider two identical optical microcavities of silicon porous placed between a light source and a detector (Fig. 1). Suppose that the wavenumbers of the incident light are within the range of k_1 to k_2 .

The intensity of the light beam incident to the detector is:

$$I \cong I_0 \cdot \int_{k_1}^{k_2} T_1(k) \times T_2(k) dk \quad (2)$$

where I_0 is the intensity of the light source, and $T_1(k)$ and $T_2(k)$ are the transmittance of the microcavities. Here, the intensity of the light source is considered independent of the wavenumbers in the considered range. If one of the multilayers is exposed to an environment containing a chemical or biological analyte, the effective dielectric functions of all the layers in the multilayer will change, which results in the modification of the transmittance spectrum of

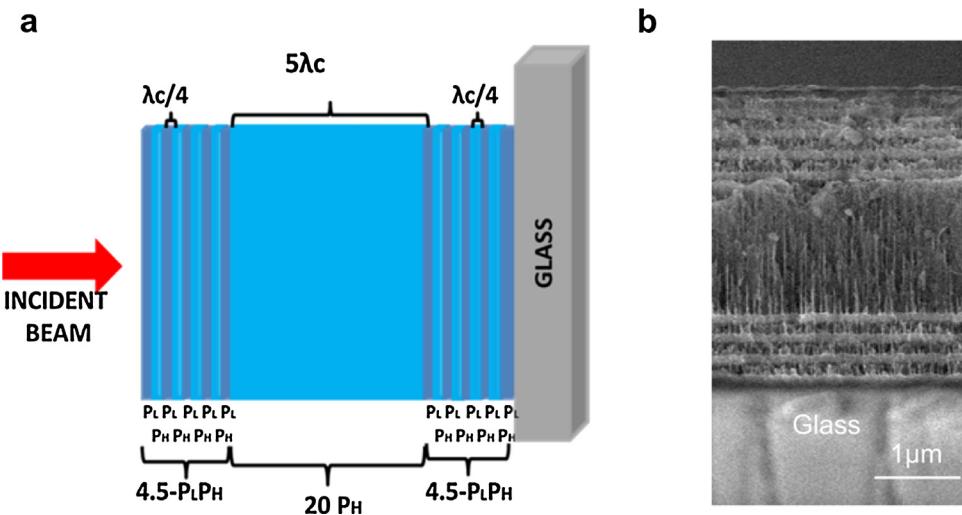


Fig. 2. Scheme of the PS microcavity and porosity sequence. Darker slabs represent low-porosity layers (P_L). The used sequence is (P_L –4(P_H P_L)–20 P_H –4(P_L P_H)– P_L), and the total physical thickness of the microcavities is about 4.5 μm (resulting in λ_c = 840 nm) (a). SEM cross-sectional view of a porous silicon optical microcavity onto glass (b).

the multilayer. Therefore, the transmittance of the whole system can be approximated by:

$$T = \frac{I}{I_0} \cong \int_{k_1}^{k_2} T_1(k) \times T_2(k + \delta) dk = M(\delta) \quad (3)$$

where $M(\delta)$ is the self-correlation function of the transmittance, and δ is the shift in wavenumber due to the presence of the analyte M decreasing steeply as δ increases. Since δ is univocally related to the analyte concentration, the system can be employed for sensing purposes. The dependence of M with δ is related to the spectral features of the transmittance; rapid variations in the transmittance produce a large variation of M (small δ produces high decorrelation). To fulfill such a condition, a light source must be chosen that has a lower bandwidth than the width of the stopping band of the multilayer (for example, an LED). Since I_0 depends on the photon energy, the integrated intensity measured by the detector is:

$$I \cong \int_{k_1}^{k_2} I_0(k) \cdot T_1(k) \times T_2(k + \delta) dk \quad (4)$$

Taking the spectral response $SR(k)$ of the detector into account, the output signal becomes:

$$S \cong \int_{k_1}^{k_2} SR(k) \cdot I_0(k) \cdot T_1(k) \times T_2(k + \delta) dk \quad (5)$$

For a given device, the relative sensitivity is defined as:

$$\beta = \left| \frac{1}{S} \frac{\partial S}{\partial n} \right| \quad (6)$$

where S is the output signal and n is the refractive index of the gas phase in the microcavity. Since the correlation function is maximized when the analyte concentration is zero, the sensitivity will then be small for low concentrations of the contaminant. To optimize the sensitivity to a specific range of analyte concentration, the microcavity M_1 can be tilted at an angle θ_1 , so that the T_1 spectrum shifts to larger wavenumbers: $T_1(k, \theta_1) - T_1(k - \Gamma, 0)$ and Eq. (5) becomes:

$$(7) S_1 \cong \int_{k_1}^{k_2} I_0(k) \cdot T_1(k - \Gamma, 0) \times T_2(k + \delta) dk$$

In this way, it is possible to adjust the angle θ_1 to obtain maximum sensitivity.

Alternatively, the microcavity M_2 can be tilted at angle θ_2 , so that the T_2 spectrum shifts to the larger wavenumbers: $T_2(k, \theta_3) - T_2(k - \Gamma, 0)$ and Eq. (5) becomes:

$$S_2 \cong \int_{k_1}^{k_2} I_0(k) \cdot T_1(k - \Gamma, 0) \times T_2(k + \delta) dk \quad (8)$$

The sensitivity can be optimized for its range of use by choosing θ_1 or θ_2 adequately. We use a computer simulation code that calculates the transmittance of the multilayers as a function of the wavelength using the matrix formalism [14]. We study the dependence of the signal (S_1 and S_2) and the relative sensitivity (Eq. (6)) as a function of the change in the refractive index (Δn) of the gas phase due to the presence of the analyte in the microcavity M_2 (Fig. 1). In this work, we use the Looyenga–Landau–Lifshitz effective medium to calculate the refractive index of each layer in the microcavity [2], which is a function of the porosity (p) and the dielectric functions of its components [15].

To correlate Δn with the analyte concentration in the gas phase, we perform a calibration using pure gases of known refractive index (He and N₂). The results of simulation are presented in section 3.

2.2. Experimental aspects

Porous silicon (PS) layers were prepared via electrochemical anodization of heavily p-type boron doped c-Si wafers with resistivity of $2\text{--}4 \times 10^{-3}$ Ω cm, (100) orientation, in a 1:2 (v/v) HF (50%):C₂H₅OH electrolyte solution. The electrochemical cell is a Teflon beaker where the Si wafer acts as the anode and the cathode is formed by a platinum wire [6–8]. A computer-controlled current source was used to fabricate the PS microcavities using an appropriate current density sequence. Then, a 4 s electropolishing pulse of about 230 mA/cm², in a 1:7 (v/v) solution of HF (50%) and C₂H₅OH, was applied to separate the PS layers from the c-Si substrates. The resultant freestanding multilayer was then transferred onto a glass substrate using a gentle jet of ethyl alcohol. The capillary forces exerted during the drying with pure nitrogen produces a tight union of the sample on the glass substrate without requiring any bound material (Fig. 2). The microcavities were designed to present central resonance at λ_c = 840 nm by alternating the layers of high (P_H) and low (P_L) porosities with the sequence shown in Fig. 2. The current densities of J_H = 63 mA/cm² and J_L = 2.1 mA/cm²

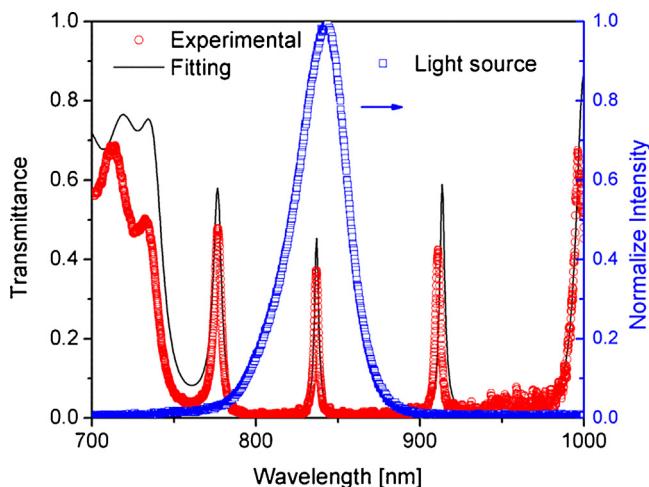


Fig. 3. Measured (circles) and fitted (line) transmittance spectra of the PS microcavity onto a glass substrate at normal incidence. The parameters used to fit the experimental data are shown in Table 1. It also shows the normalized intensity of the light source used in the experiments (squares).

were used for $t_H = 5$ s and $t_L = 56.7$ s to get the layers of the lateral Bragg mirrors. The optical thicknesses of each layer of the lateral Bragg mirrors are $1/4\lambda_c$. By performing a sensitivity analysis similar to [4], we decided to use microcavities with a central defect of $5\lambda_c$ and 4.5-period Bragg mirrors. The extra P_L layer in front of the Bragg mirror results in improved definition of the central resonance peak transmission.

The obtained microcavities were characterized by fitting their transmittance spectrum, using the layers porosities (P_H and P_L) and thicknesses (D_H and D_L) as free parameters. Spectra were taken with an Ocean Optics HR4000 spectrometer at normal incidence to the substrate surface in the range 700–1000 nm. Fig. 3 shows the measured and fitted transmittance spectra of the microcavity as a function of the wavelength at normal incidence. The same figure shows the normalized emission spectrum of the light emission diode that is used as the light source.

To test the sensor characteristics, the optical microcavity M_2 sample was placed in a contamination chamber to expose it to solvent vapors, and the flow cell was connected to a gas-mixer/flowmeters controller. The nitrogen carrier gas was saturated with isopropyl alcohol (IPA) by passing it through a bubbler containing the solvent. The saturated vapor was then mixed with the nitrogen mainstream. It is possible to obtain different concentrations of solvent vapor by adjusting the flow of the carrying nitrogen and the saturated vapor. The setup used to produce a controlled concentration of analyte on the microcavity is shown in Fig. 4. A constant flow of N_2 is maintained for 60 min prior to each measurement to dry the sample. The area of the sample was approximately 1 cm^2 . The sensing area is defined by the size of the light spot formed by the LED (through the lens) on the microcavity, and is about 4 mm^2 . The transmitted light of the system was measured using a simple infrared phototransistor and a 61/2-digit multimeter (M3500A Picotest).

Table 1
Physical parameters of the microcavity that best fit the measured transmittance spectra (Fig. 3).

	Porosity (%)	Layer width (nm)	Refractive index
High porosity layer	76.5	122	1.52
Low porosity layer	49.5	112	2.21

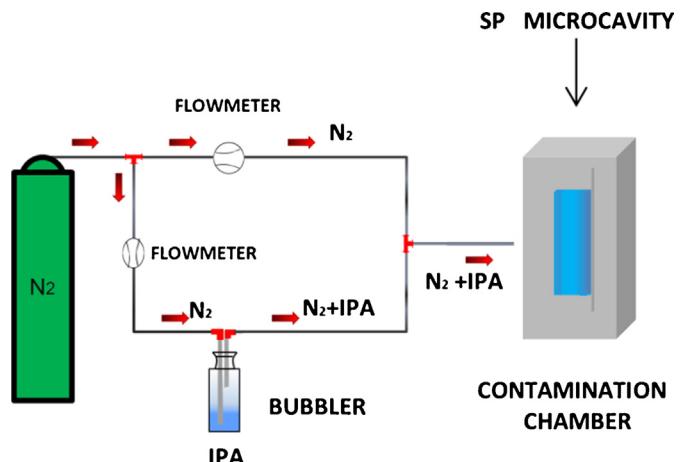


Fig. 4. The experimental setup used to control the analyte concentration on the microcavity M_2 . Pure nitrogen (99.998%) is divided in two flows, one of which is saturated in IPA at ambient temperature and then mixed with the main flow. By choosing the ratio of these two currents, we can control the concentration of IPA in the outflow.

3. Results and discussions

The sensor performance can be evaluated numerically using the microcavity parameters obtained from the fit of the measured transmittance spectra (Fig. 3). We considered the measured emission spectra of the used LED (Fig. 3) in the p-polarized direction. The computed values of S_1 (Eq. (7)) and S_2 (Eq. (8)) are shown in Fig. 5a and c respectively, as a function of the rotated angle (θ_1 and θ_2), and the refractive index change Δn in the void volume of the microcavity. The relative sensitivity (Eq. (6)) for S_1 and S_2 are shown in Fig. 5b and Fig. 5d, respectively. In Fig. 5a, three different regions of high correlation of the transmittance can be observed. Besides the region of θ_1 near zero in which the transmittance spectra are identical, the high transmittance condition is obtained when a resonance at a higher wavelength is blue-shifted until it coincides with the central resonance at 840 nm. Then, the high values of S_1 at θ_1 around 40° are obtained when the resonance at 910 nm (Fig. 3) of M_1 is shifted until it overlaps with the central resonance at 840 nm of M_2 . An increase of the refractive index in M_2 produces a red shift in the transmittance spectrum, which can be compensated by a diminution of θ_1 . If M_2 is rotated (Fig. 5c), an increase of Δn is compensated with an increase of θ_2 , and a change in the slope of the high transmittance region is produced.

Fig. 5b and Fig. 5d show that a maximum in the signal produces a minimum in sensitivity. The regions of maximum sensitivity depend on the angle between the microcavities, and they can be used to obtain the maximum sensitivity in the concentration range of interest.

Measuring the performance of the sensor allows us to adopt a configuration in which M_1 is fixed while θ_2 changes to optimize the sensitivity. Fig. 6 shows the measured total transmittance as a function of θ_2 in pure nitrogen stream. The inset shows the response of the reduced regions, when M_2 is contaminated with mixed N_2 -IPA at different concentrations. Although the three regions of high transmittance can be clearly identified, the measured angular position of maximum correlation does not correspond exactly to the numerical predictions (Fig. 5c). This can be attributed to a slight anisotropy (birefringence) in the porous silicon material [16], which is not included in the simulations. When M_2 is contaminated (inset in Fig. 6), the peak position is shifted to larger values of θ_2 , as predicted by calculation (Fig. 5c). Furthermore, a diminution in the peak intensity is observed in the experiment, which is not predicted by calculation. By considering this, we

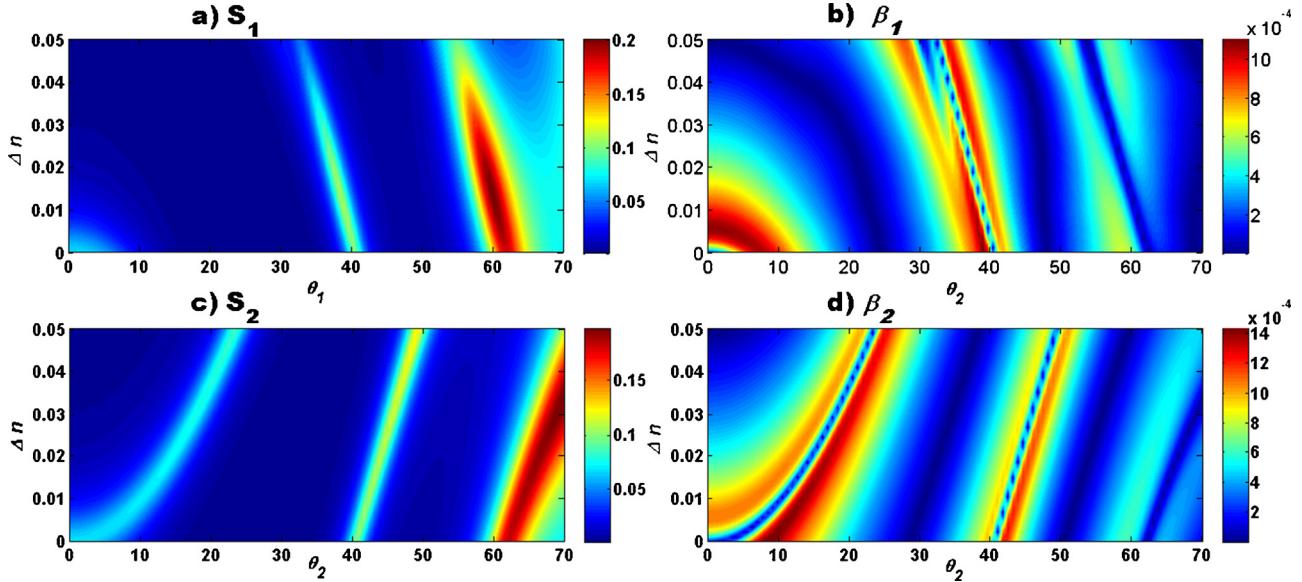


Fig. 5. The computed total transmittance when (a) M_1 is rotated or (c) M_2 is rotated as a function of the change in the refractive index change in M_2 . The respective sensitivities (b) and (d) are calculated using Eq. (6). This figure was calculated using the microcavity parameters obtained from the fit in Fig. 3, and the LED spectrum in the p-type polarization.

use the measured signal at different concentrations to estimate the relative sensitivity (Eq. (6)). We found that the maximum sensitivity for low concentrations is achieved at approximately $\theta_2=27^\circ$.

Fig. 7 shows the measured signal of the system when the IPA concentration is varied in the range 0–5000 ppm at $\theta_2=27^\circ$. A mainstream of N₂ at 3 l/min was used at 23 °C. Each step is produced by increasing the concentration of IPA. The rise and fall times were 25 s, and almost constant for all concentrations in accordance with the volume of the detector chamber (400 ml). The organic vapor sensing process is completely reversible how observed in Fig. 7 initial and end signal values are the same after 1500 s of exposure.

Fig. 8 shows the detector signal change as a function of the IPA concentration obtained from the equilibrium values of Fig. 7. The data can fit reasonably well to a potential relationship with the IPA concentration, in agreement with the previous works on oxidized porous silicon rugate filters [17].

To obtain the relationship between the detector response and the change of refractive index of the gas mixture, we used two

different gases of known refractive index, namely helium and nitrogen.

We use N₂ and He as reference gases, because of their large refractive index contrast (1.000296 and 1.000034, respectively, at 840 nm [18]), and the measured value of the signal difference was about 2 mV at $\theta_2=27^\circ$. Considering that the standard deviation noise over 100 s was $\sigma=0.5$ mV, the minimum measurable change of refractive index is estimated at 5×10^{-5} .

The smallest concentration that we experimentally explore was 180 ppm (Fig. 8). The signal measured in this case was 6.3 mV. Considering that the minimum signal that can be measured unambiguously must to be at last twice the noise (i.e. 1 mV) the minimum concentration that we could measure is about 30 ppm. This value represents the detection limit of the system. These values are comparable to the minimum concentrations measurable by detection systems using other techniques such as spectrophotometry [12,19,20], electrical capacitance [21] or conductivity [22]. Moreover, the time response of the system proposed here is comparable to the fastest reported sensors [4,12].

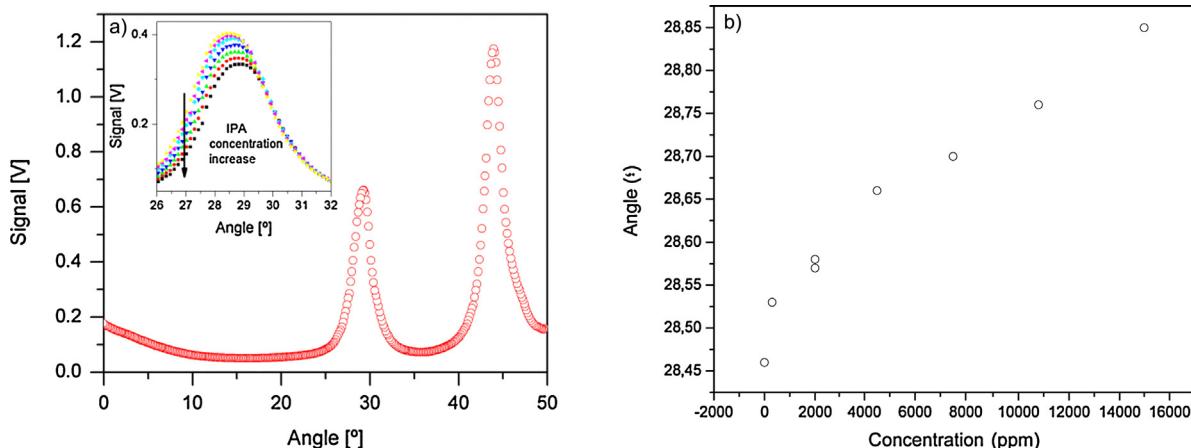


Fig. 6. Total transmittance measured as a function of the angular position of the second microcavity for pure nitrogen (a). Inset shows a detail of one peak for different IPA concentrations. Peak position as a function of IPA concentration (b).

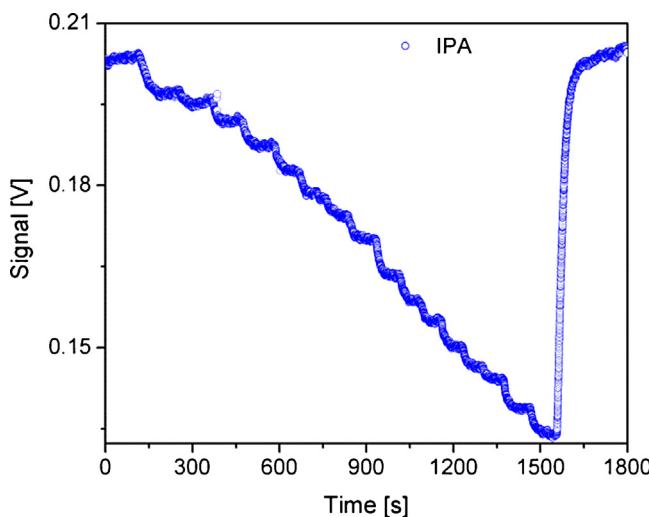


Fig. 7. Detector signal for a sequence of sudden changes of the analyte concentration from 0 ppm to 5000 ppm at $\theta_2 = 27^\circ$.

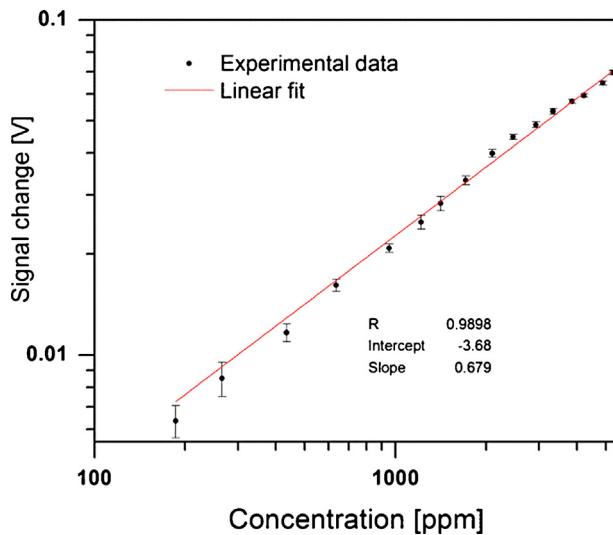


Fig. 8. Detector response when the M_2 microcavity is at 27° and exposed to a controlled concentration of isopropyl alcohol vapor.

4. Conclusions

This work presents a new strategy for sensing purposes based on the use of the autocorrelation of the transmittance spectrum of porous silicon microcavities. This scheme avoids the use of wavelength-sensitive detectors or monochromatic light sources, using two identical microcavities, one of which works as a filter, and the other as a sensing element. The concept idea is proven by sensing the mixture of N_2 and IPA vapors in the range 0–5000 ppm. We have optimized the sensitivity of the system by controlling the angular position of the second microcavity, achieving a minimum measurable change of the refractive index as low as 5×10^{-5} .

Since the concept is directly extendable to another wavelength range of application, this configuration may be useful for biosensors that require a large pore size to allow for the penetration of large molecules. Then, this platform could be useful in a variety of environmental and biological scenarios in which the possible wavelength range is restricted to a specific region.

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