VERTICAL RESONANT MICROCATIVITES BASED ON PILLARS ANALYZED BY BEAM PROFILE ELLIPSOMETRY AND REFLECTOMETRY R. Casquel¹, M. Holgado^{*,1}, A. Lavín¹, C. A. Barrios², C. Molpeceres¹, M. Morales¹, J.L. Ocaña¹.

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Abstract: A biosensor design is presented by a combination of ellipsometry, reflectometry and spectrometry based techniques is presented. It consists of a lattice of columns forming resonant microcavities. Calculations for reflectivity profiles are shown, and estimations for detection limit in refractive index units are obtained.

Keywords: Biosensing, Ellipsometry, Reflectometry.

1. Introduction.

Integrated photonic sensors for biomolecular detection is a field with continuous research recently [1-3]. Fabrication processes based on mature CMOS technologies are employed, and high sensitivity is reached. One of the main disadvantages of these sensors is that they require complex systems for coupling light, such as grating couplers and inverted tapers [4].

With the use of vertical interrogation optical techniques the complexity of coupling light is avoided. Recent investigations have demonstrated that by combination of the simultaneous characterization of reflectivity as a function of wavelength, reflectivity as a function of angle of incidence, and phase shift of reflected light in advanced photonic structures a high sensitivity refractive index sensor vertically interrogated is achieved [5].

In this work it is proposed a integrated device compatible with standard micro-fabrication technology, consisting of a lattice of micro-pillars, forming resonant microcavities, vertically characterized, as seen schematically in Fig.1. The feasibility of measuring at submicron spot size level by means of spectrometry, reflectometry and ellipsometry based techniques has been recently demonstrated [5,6]. Change in response for reflectivity and phase shift is calculated for different refractive indices surrounding the pillars.



Figure 1. Proposed device.

The goal of the design has been obtaining a high sensitivity device for measuring in continuous flow regime, which makes it suitable for biochemical sensing. The sensor is placed inside a microchannel, with a port for the fluid to enter and another port to exit from the structure, as schematically shown in Figure 2. Further considerations in more complex fluidics will be taking into account in future developments.



Figure 2. Schematic of the proposed device.

The design of the photonic structure has been carried out into two main aspects, first of all the vertical design of the columns (number of layers and different materials), and secondly planar design, which means defining lattice parameter and width of the pillars. There are limitations both from the fabrication, in the size of the columns, and in the fluidic point of view: there has to be enough space for the fluid to pass through the gap among pillars.

To estimate sensitivity and ensure that the device can work as a biosensor, two different situations have been calculated. Firstly, the pillars with an attached layer of standard protein immobilized in their walls (the layer of protein with 15 nm in thickness, and a refractive index of 1.4, which is the standard biolayer measured in biosensing), and secondly the structure with no protein in it, being this the reference data. Limit of detection of the sensor is obtained with the shift in the dips for both situations

Results for two different structures are presented: A configuration with pillars of SiO_2 with two aluminium thin layers as reflectors, and another with two Si_3N_4 layers. Different lattice parameters and diameter of pillars has been also calculated. Detection limit obtained is in the order of 10^{-5} R.I.U.

2. Design. Theoretical Calculations

Reflectivity and phase shift changes have to be obtained for estimate sensitivity. For this purpose, it has been used a multilayer model consisting of four different layers, each one with its effective refractive index. Figure 3 shows the actual situation and the equivalent theoretical model. The structure of pillars with several layers, and a biomolecular layer attached turns into a multilayer structure with reflectivity profiles using Fresnel equations and thin layer interference models. The effective refractive index of each layer has been calculated using Beam Propagation Method [9]. It has been simulated a Gaussian beam focusing normally on the pillars, with the protein layer, and measured the phase change of the light, considering a length equal to the thickness of each layer. With the total phase change obtained, n_{eff} is estimated. The suitability of this model has been checked out with similar layer structure (holes instead of columns) previously [5]. For the top layer, refractive index is the index of the protein, and also its thickness.



Figure 3. Theoretical multilayer model

Several aspects has been taking into account whe carriyng out the design. The detection is performed measuring the shift of peaks and dips in the interference reflectivity pattern, both in wavelength and in incident angle of light. These reflectivity profiles are obtained using multilayer model equations.

The final goal is to achieve a high throughput sensing system implemented with high sensitivity photonic structures by means of the proper design of high Q-factor resonant micro-cavities. Optical response of this structure will be particularly sensitive to ultra-small refractive index changes induced for the bio-layers attached on the micropillars surface. Two main limitations have to be considered in the design process. In one hand limitations from the fluidic point of view, and in the other from the fabrication point of view.

With respect to fluidics, pillars lattice has been chosen because it allows measuring in continous flow regime, which is an standard for conventional bioassays. Figure 1 shows the schematic for measuring. There is a port for the fluid to come into the channel, and after it passes through the columns. Functionalization and biosensing in continous flow inside channels of 150 nm has been recently reported [8], and also kinetiks in channels as narrow as 27 nm [9]. We have considered that with a minimun separation of 100 nm is feasible, although it will be discussed later how the results will be affected in case this separation is too small.

With respect to microfabrication, limitations mainly related to the etching process should be considered. The higher the columns are, the steeper the walls, obtaining not cylindrical pillars but cone shaped ones, which should be considered in the calculations. Technology allows to obtain a column with a width in the order of 800 nm/ 1 μ m in height, which may be considered as a limit.

We propose using three layer columns in order to create a vertical microcavity, similar to a Fabry-Perot interferometer, with the top and the bottom layer acting as reflectors, and the middle layer as the cavity itself, of SiO₂. Two different materials are considered for the reflectors: alluminium and Si₃N₄. For the aluminium reflectors, 25 nm is the thickness proposed, and for Si₃N₄ 125 nm each one. And the central layer is 800 nm thick for both cases [Fig. 4].



Figure 4. Proposed structures

Lattice parameters (a) and diameter (d) [Fig. 5] should be also defined. One of the most important characteristics of this sensor is that the lattice confines the light in the space among holes. The closer they are to each other, the more intense is the evanescent field in there. It is interesting to maximize this field because this is the area where the biomolecules will be attached, meaning this that a change in the refractive index in this gap will result in a higher change in effective refractive index, and therefore higher sensitivity.



Figure 5. Parameters of the lattice.

Figure 6 shows the different field distribution for a configuration of a lattice of pillars and a single one. The field in the gap is much more intense in the first case. This effect becomes stronger when the lattice parameter is reduced, mantaining the diameter of the column, and so the pillars are closer to each other. The limitation in this case comes from the fluidic aspects. A diameter of 400 nm and lattice parameter o 500 nm is chosen.



Figure 6. Field distribution for a lattice of columns (a) and a single pillar (b).

Finally, we have used parameters of abovementioned techniques intented to be used in characterization, which are employing a white light source with a spot size of 2.5 μ m for spectrometry, and a laser in 675 nm with and spot of 0.9 μ m for reflectivity as a function of angle of incidence and beam profile ellipsometry [6].

3. Results and discussion.

Figure 7 shows spectrometry measurements for both structures: a) Aluminium reflectors, b) Si_3N_4 reflectors. Solid curve represents the reference structure profile, and dashed one the profile with a protein layer attached.



Figure 7. Spectrometry calculations.

Figure 8 shows results for reflectometry, for s polarization (p polarization is similar). As above, a) is for metallic reflectors and b) for silicon nitride.



Figure 8. Reflectivity profile

Figure 9 shows phase shift change in reflected light calculated for beam profile ellipsometry [6] as a function of thickness of the attached layer over the walls of the pillars, from 0 to 15 nm.



Figure 9. Equivalent phase shift [5] for a) aluminium reflectors b) Si₃N4 layers

The detection limit of the device is the minimum change in refractive index of the bio-molecule layer that the system is able to resolve. To calculate this is the relation between the shift of the different dips and refractive index change, and the resolution of the measurement tool used. Table 1 summarizes the results for the different techniques.

Detection Limit	Reflectometry (R.I.U)	Spectrometry (R.I.U.)	Ellipsometry (R.I.U.)
Aluminium reflectors	5x10 ⁻³	5x10 ⁻⁴	7x10 ⁻⁶
Si ₃ N ₄	10 ⁻³	3x10 ⁻⁴	10 ⁻⁵

Table 1. Limit of detection for the different techniques.

For spectrometry, considering a resolution of 10 pm, commercially available:

$$\Delta n_{\min} = \lambda_{\text{resolution}} \times \Delta n / \Delta \lambda_{\text{dips}}$$
(1)

For reflectometry, considering a resolution of 0.01°:

$$\Delta n_{\min} = \theta_{\text{resolution}} \times \Delta n / \Delta \theta_{\text{dips}}$$
(2)

For ellipsometry, considering a resolution of 0.01°:

$$\Delta n_{\min} = \theta_{\text{resolution}} \times \Delta n / \Delta (\text{phase shift})$$
(3)

Results obtained remain competitive compared with nowadays state of the art [1]. Fabrication with standard CMOS technology is proposed: obtaining multilayer structure by CVD deposition, Deep UV lithography for drawing the pillars, after this creating a lift-off mask with chromium, and etching with an ICP equipment.

There are several points to be remarked. As mentioned before, the final shape of the fabricated pillars is not cylindrical but conical; however the influence in the detection limit results is not important for higher aspect ratio pillars. For lower aspect ratios it should be considered in calculations. And from the fluidic point of view, if gap between colums is increased in order to ease flow into channels and biofunctionalization process, for example for a diameter of 250 nm and a lattice parameter of 500, detection limits get reduced by a factor of 0.5.

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