Study of silicon nitride O-ring resonator for gas-sensing applications

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Abstract. In this work, we experimentally studied the influence of different gaseous surroundings on silicon nitride O-ring resonator transmission. We compared the obtained results with numerical calculations and theoretical analysis and found a good agreement between them. Our results have a great potential for gas sensing applications, where a compact footprint and high efficiency are desired simultaneously.

1. Introduction

A photonic integrated circuit (PIC) is a very powerful platform for miniaturized and precision sensors. It can be used for defining properties of different gases [1], chemical and biological materials [2], etc. Typically, such devices use the evanescent field for detecting small perturbations in refractive indices of the cladding: the distribution of the electric field in the device changes via the evanescent field. The use of light, coupled with fluorescence and Raman scattering made optical sensors capable of detecting concentrations of different substances with high sensitivity and spectral resolution with multiplexed systems [3, 4]. The advancement in fabrication technologies has led to the miniaturization of optical sensing systems. The physical structure of optical waveguides or resonators can also be used for biological sample flow, creating compact liquid waveguides [4]. Optical structures fabricated on the silicon on insulator (SOI) platform are promising for a wide range of integrated optical applications [5]. These structures can be realized in a very small area on a chip due to the high refractive index contrast between the waveguides and the surrounding claddings. The research on SOI optical structures is recently extending beyond the telecom to other applications such as optical bio-molecule and gas sensing [6]. The most sensitive devices use resonance circuits, for example spherical, disc, and ring cavities, and more complex ones such as toroidal, tubular, and microbubble [7-9]. Ring resonators are simple for fabrication and they are sensitive and precise. The response time for ring resonator is low enough to use them for gas-sensing [7].

In this paper, we use the silicon nitride platform, which along with SOI is one of the promising platforms for creating fully integrated sensors on a chip. A distinctive feature of this platform is a wide bandgap (~5 eV), which allows working both in the visible and infrared wavelengths, as well as lower optical contrast, which is not so sensitive to fabrication errors and wall roughness as SOI. Here we studied silicon nitride O-ring resonator (ORR) as a gas sensor for helium and propane small concentration detection.
Figure 1 (a,b). (a) Quasi TE-mode distribution in the cross section of the silicon nitride O-ring resonator waveguide with air cladding; (b) Normalized electric field along the silicon nitride waveguide (dashed line), demonstrated evanescent mode profile at different gases claddings: propane, air, helium. The inset shows the change in the intensity of the evanescent mode.

2. Simulation

The principle of the ORR operation is close to the Fabry-Perot resonator cavity. On the transmission spectrum, one can determine interference peaks, which location directly depends on the refractive index of the ring’s waveguide material and its environment. If we change the environment of the ring, we thereby change the group refractive index, which, in turn, leads to changes in positions of the resonant peaks inside the transmission spectrum.

To describe the transmission spectrum and to calculate the optimal ORRs dimensions, we used both analytical calculation and numerical simulation. The main ORR parameters for gas sensing applications: free spectrum range (FSR) and peaks’ positions ($\lambda_{res}$) can be found by well-known equations [7]. For the FSR calculation, group refractive index ($n_g$) should be found, on a base of effective refractive index for a given waveguide geometry ($n_{eff}$):

$$n_g = n_{eff} + \frac{\lambda d n_{eff}}{\lambda_0}, \quad (1)$$

where and $\lambda_0$ is wavelength in vacuum.

Knowing the group refractive index FSR can be defined as:

$$FSR = \frac{\lambda_0^2}{n_g 2\pi R}, \quad (2)$$

where $R$ is the radius of the O-ring resonator.

The calculations in MathCAD gave us some information about how a perfect resonator would work with different gaseous claddings in general, but lose the detail information about waveguide effective refractive index and shape of the resonance peak. Therefore, we decided to use numerical Finite Element Method (FEM) to simulate the resonance curves using COMSOL Multiphysics. We used a standard 2D O-ring resonator model, adapted for silicon nitride device geometry. In the first step, we defined the effective refractive index by modeling the cross-section of the substrate dielectric layers with a half-etched silicon nitride waveguide (Figure 1a) and air as a cladding $n_{air} = 1.000292 \ @ \ \lambda=1550 \ nm$ [10].
In the second step, we calculated the transmission spectrum of the ORR with two gas claddings: helium ($n_{He} = 1.000035 @ \lambda = 1550 \text{ nm}$) and propane ($n_{C3H8} = 1.2898 @ \lambda = 1550 \text{ nm}$) [10, 11].

![Microphotograph of the fabricated silicon nitride O-ring resonator.](image)

Figure 2 (a-d). (a) Microphotograph of the fabricated silicon nitride O-ring resonator. The input and the output of the light are shown by the white arrows; (b) Schematic view of the used experimental setup with optical (blue lines) and electrical (orange line) connections; (c) Experimentally measured raw ORR transmission spectra before normalization. The envelope of the spectrum (shown with blue arrow) corresponds to focusing grating couplers and thin resonance peaks correspond to ORR; (d) Zoomed image of ORR transmission spectra for three gaseous: helium, air, and propane. The position of the minimum shifts with the change of the gas refractive index.

We obtained how the evanescent modes of the three gases differ. In Figure 1b is shown that both waveguide and the evanescent modes are changed at different gas claddings. For the part of the field that propagated through SiO$_2$, there is quite a small TE-mode modification, but for the evanescent mode, propagated through the gas there is a shift under variation of refractive index. This behavior preliminary showed us that this waveguide configuration can operate as a gas sensor and can be fabricated and tested.

3. Device design and fabrication

For nanophotonic device fabrication, we used commercially available silicon substrates with thermal silicon oxide SiO$_2=2.6 \mu \text{m}$ and silicon nitride Si$_3$N$_4=450$ nm layers atop. We used one step of e-beam lithography, based on Crestec CABL-9050C and positive resist ZEP 520 A, following by reactive ion etching (RIE) in CHF$_3$ atmosphere for device finalizing. In Figure 2a the fabricated O-ring resonator is shown. This device consists of two focusing grating couplers (FGCs) for input/output light with coupling efficiency about 15% and 1 \mu m waveguide bus, separated by a gap of 1.4 \mu m with the O-ring waveguide.
The radius of the O-ring was chosen equal to 63.728 μm to ensure a free spectral range (FSR) of about 3 nm, and its width was chosen 1.6 μm to increase Q-factor.

4. Experimental setup and results

Experimental setup for measurements of nanophotonic device transmission spectra consists of a tunable laser (NewFocus TLB-6600 with tuning range 1510-1620 nm), a polarization controller, x, y, z – rotation sample holder with piezo motors, a fast photodetector, as well as a fast analog-to-digital converter (Figure 2b). To match the light in an optical fiber with the ORR, we used a fiber array and FGС on a chip [12].

For measurements in various gases, we made a thin pipe for blowing gas around the sample, while the sample itself was fixed on the table. The measured O-ring transmission spectra at 1mW input power (1510-1620 nm) for different gas claddings are shown in Figure 2c. After obtaining the transmission spectra, they were normalized to transmission spectra of FGСs (the envelope of spectrum in Figure 2c) and converted into [dB] relatively to maximum transmission. The position of the resonance peak substantially depends on the environment. For the cladding with a lower refractive index the peak is shifted to the lower wavelengths and vice versa. This fact is in qualitative agreement with the previous computations.

5. Discussion

It should be noted, during the experiment there cannot be obtained pure gas as there is used the flow of the gas. Therefore, some approximations should be used in order to compare simulated and measured transmission spectra. Using the fact that the refractive index of each separated gas doesn’t sufficiently change in the telecommunication wavelength range, we used the constant values of the gases refractive indices to calculate the total refractive index for a mixture of air with helium and air with propane, correspondingly.

For the mixture of gases, the total refractive index is a molar (or volumetric) average value of the gases refractive indices [13-15]. From these assumptions, 2D models of ring resonators were built for various proportions of two gases in a mixture (Figure 3a). For the models, we used not only the design parameters of the ORR but also some nearby parameters in order to compare them with the experimental results. The refractive index of a mixture of gases can be found as:

$$n_{1+2} = n_1 \frac{V_1}{V_{1+2}} + n_2 \frac{V_2}{V_{1+2}},$$

where $n_1$ is the refractive index of the first gas, $n_2$ is the refractive index of the second gas, $n_{1+2}$ is the refractive index of the mixture and $V_1, V_2, V_{1+2}$ are the corresponding volumes of the gases (or numbers of moles) in the mixture.

Qualitatively, the observed dependence of FSR on the wavelength (Figure 3b) is consistent with the calculation data: FSR increases directly proportional to the swept wavelength. But in contrast to the simulation in MathCAD, we did not see any difference between studied gases. This may be due to both a low concentration of gases and the technical performance of the tunable laser source we are using. Laser wavelength sweep rate may not be a constant in time or too fast, thereby distorting information about the measured FSR.

After simulation of the resonance curves for different concentrations of the two gases, we analyzed a minimum detected concentration, which is possible with the fabricated O-ring resonator. In Figure 3c the calculated dependence of concentration in parts per million units (ppm) versus pick shift is shown. For used here O-ring resonator with FWHM of 0.01 nm, determined from the experimental data (Figure 2d), the estimated minimum concentration for propane is 2.23 x10^5 ppm and for helium is 3.625x10^5 ppm.

Future work will be devoted to a more precise computation in COMSOL Multiphysics (decreasing the parametric sweep wavelength step from 0.01 nm to 0.0001 nm), as well as a new FSR computation using the Finite Difference Time Domain method (FDTD), realized in Meep software.

In addition, we will pay attention to better control of gas concentration, as well as its pressure.
Figure 3 (a-b). (a) Experimentally obtained transmission spectra for helium and propane on the same plot with numerically simulated transmission spectra of these gases at different concentrations. (b) Comparison of FSR for the three gases obtained experimentally and by MathCAD computations. The refractive indices for gases are wavelength depended. (c) Dependence of detected gas concentration in parts per million (ppm) on the peak shift for helium and propane. The dashed line shows the estimation of minimum detected gas concentration for fabricated silicon nitride ORR with FWHM of 0.01 nm.

In general, the experimental results coincide well with theoretical ones, making the introduced silicon nitride ORR a good example of a gas sensing circuit, which can be integrated with more complex lab-on-chip devices.

6. Conclusions
We studied the dependence of the transmission spectrum of the ring resonator depending on the gas surroundings through evanescent mode interaction. Obtained experimental results with a resonance pick position are in a good agreement with theoretical data and numerical calculation. The minimum concentration for propane is $2.23 \times 10^5$ ppm and for helium is $3.625 \times 10^5$ ppm for the fabricated O-ring resonator were estimated. This work is important for the miniaturization of nanophotonics, biomedical devices and chemical devices detection.

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