Employing a Dual Polarisation Microring to Determine Refractive Index and Thickness of a Thin Polymer Layer

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ABSTRACT
Dual polarisation biosensing is a novel optical technique that focuses on retrieving structural information from a bound or adsorbed layer of molecules, by using a silicon-on-insulator (SOI) microring. Both density and thickness of the layer can be monitored simultaneously. Due to a self-calibrating protocol that is quickly performed at the start of every experiment, a high accuracy can be obtained. In this paper, we determine the thickness and refractive index of a thin layer of polyethyleneimine (PEI), which adsorbs to the silicon surface of the microring. This results in a layer with a thickness of 1.158 and a refractive index of 1.453 RIU.
Keywords: optical resonators, biological sensing, polymers.

1. INTRODUCTION

In a 2011 Nature Chemistry Insight, Connelly [1] points out that a major part in the current development crisis of the pharmaceutical industry lies in our poor understanding of how our drug candidates function at a molecular level. Easy targets no longer exist and as is touched upon by Scannel [2], a lot of current drugs are not magic bullets, but magic shotguns, targeting multiple targets at once. In order to find these magic bullets we have to figure out what we are aiming at and how these bullets work. As such, there is a need for a high-throughput tool which can monitor different aspects of binding dynamics such as conformation, kinetic response and affinity of binding.

Traditional refractive index sensing optical methods usually have a response that is related to the overlap of an optical mode with the sensing medium. This optical mode responds due to changes in the optical density and can't distinguish between a thin dense layer and a thick sparse layer, which is a requirement that has to be fulfilled in order to monitor the binding conformation. However, by employing two optical modes that interact with the molecules of interest, the thickness of the layer and the density can be disentangled and monitored simultaneously. This article presents this solution under the form of a silicon-on-insulator (SOI) microring which is excited with quasi-TE and quasi-TM polarisations at the same time [3].

2. SENSOR METHODS

2.1 Sensing principle

The sensor is a racetrack microresonator with the waveguide profile of a silicon box on a silicon dioxide substrate, fabricated according to [4]. This wire profile has a height of 220 nm and a width of 550 nm. Such a profile supports a fundamental TE mode and a fundamental TM mode. Due to a difference in confinement and the discrete jump of the TM mode at the top surface, the evanescent field in the aqueous cladding where the biolayer is formed is very different for both modes. This is depicted in on Fig. 1.

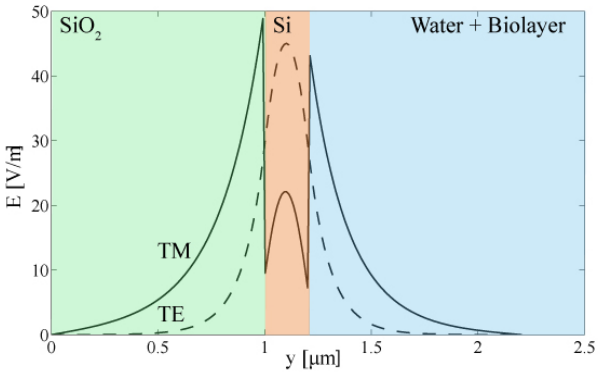

Figure 1. Electric field profile of quasi-TE and quasi-TM mode in the horizontal centre of the 2D waveguide cross section, as a function of the vertical coordinate. For both polarisations, only the dominant electric field component in the 2D cross section is shown.

It is this evanescent field that probes the layer of biomolecules and causes the resonant wavelength of both modes to shift whenever the composition or the mass of this layer changes. The penetration depth is defined as the distance over which the energy density of the modes decreases by a factor 1/e and this amounts to 57 nm for the TE mode and 106 nm for the TM mode. Due to these different fields the wavelengths at which the modes resonate as well as the shifts of these wavelengths are different for both modes. The shifts are equal to the following equation:

$$\Delta\lambda(n,t) = \frac{\Delta n_{eff}(n,t)\cdot\lambda}{n_g}$$

2.2 Design of a dual polarisation microring

The coupling section of the microring is an asymmetrical directional coupler. This allows for the light to be routed around the photonics circuit in TE polarization, while being coupled to a TM mode of the ring waveguide due to careful phase matching in this coupler [5]. This phase matching is done by choosing a specific combination of widths for the access waveguide and the ring waveguide in the coupling section. This results in a large phase mismatch between the TE mode in the access waveguide and the TE mode in the ring waveguide, rendering coupling almost impossible at normal gaps. When the gap of the coupling section is small enough, this phase mismatch can be overcome by substantial overlap between the TE profile of the access waveguide and the TE profile of the ring waveguide. These devices were designed using the IPKISS parametric design framework.

Figure 2 shows the measured (loaded) Q-factor and the extinction ratio (E.R.) of a microring resonator with an access waveguide width of 290 nm and a ring waveguide width of 550 nm, as a function of the gap in the directional coupler. For biosensing, a Q-factor > 5000 is advisable, while the E.R. should not be less than 5 dB. We see from the graph, that for gaps smaller than 120 nm, the E.R. is adequate but the Q factor is too low, indicating that the loss of the TE mode in the ring is too high due to too much coupling in the coupling section. For small gaps it is also not guaranteed that the gap is completely etched, increasing coupling even further. For a gap of 140 nm (and higher), we see that there are no TE resonances at all. For these gaps, the overlap can no longer compensate the phase mismatching due to the difference in widths of the two coupling waveguides. Only for gaps of 120 nm and specific coupling lengths the TE modes are suitable for sensing. These gap widths are dimensions as put on the lithographic mask. For similarly small gaps, the printed gaps can be quite different from the ones on the mask. We can conclude that the design window in order to excite both a TE and a TM mode simultaneously with one coupling section, is relatively small.

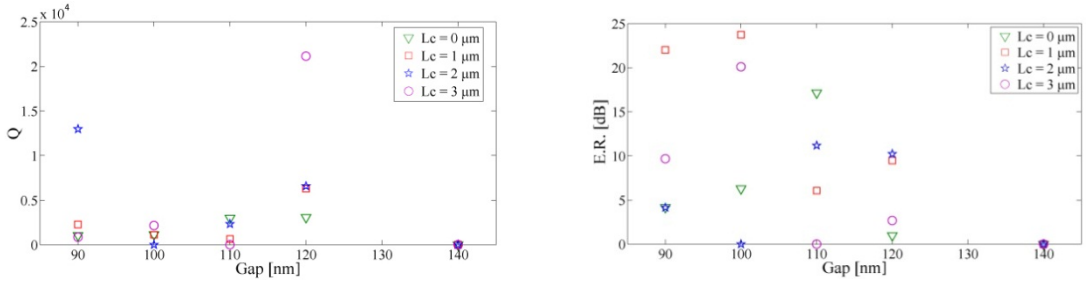

Figure 2. Measured Q (a) and ER(b) of a TE mode for a ring with water cladding and a width of 550 nm in function of the gap of the coupling section, for various coupling lengths.

2.3 Determining the thickness and the refractive index of a thin biolayer

By tracking the change of the resonance wavelength of both modes, which are dependent on the thickness (t) and the refractive index (n) of the layer of biomolecules, we can obtain t and n. In order to do this we need a set of equations that link the wavelength shifts to these independent variables. This set consists of the following equation for both modes:

$$\Delta\lambda(n,t) = B\frac{(n-n_b)\,n\,f_p(t,n)}{1+n^2 f_p(t,n)+n_b^2 f_b(t,n)}$$

Here, n_b denotes the index of the bulk fluid while the functions f_b,f_b and the constant B can be determined with the help of simulation package *Fimmwave.* The bulk fluid is the fluid which is present above the biolayer. These functions are highly dependent on the waveguide width (W) and height (H). Since the deviation due to fabrication on both W and H is too high to obtain accurate results, a calibration has to be performed with each sensor prior to the experiment. To obtain the correct width and height, water is streamed over the waveguide and the FSR of both modes is measured. Due to a high linearity of both modes in either W or H, these can be determined accurately with a 3σ error of 96 pm on W and 26 pm on H. The mathematical model, the solving procedure and the calibration protocol are described in more detail in [3]. In the experiment that follows the bulk fluid does not have to be calibrated for, since we use water.

3. ADSORPTION OF PEI LAYER

In order to show the possibilities of this technique, we apply it to measure the thickness and refractive index of a thin polymer layer. Previously, this technique has been applied to the field of proteins [3]. A fluidic cell made out of PDMS is bonded to the silicon chip by means of a plasma activation step, followed by a heating step combined with mechanical pressure. This causes the PDMS to form covalent bonds with the silicon surface, and thus creating fluidic channels. To read out the sensors, a laser is swept from 1525 nm to 1545 nm at 2 nm/sec with a resolution of 5 pm. This light is coupled vertically in the plane of the chip by using grating couplers. It goes through the sensor and eventually couples back out of the chip into an infrared camera. As such every spectral feature is tracked once per ten seconds, which is not the limit of the system, but sufficient for this measurement.

A 2 mg/ml solution of polyethyleneimine (PEI) in water is prepared. This is a hydrophilic polymer which can be used for its adhesive properties, and has recently been used as a surface modification polymer for silicon biosensors [6]. First, water is injected over the chip for 12 minutes at a flow rate of 50 µl/min, such that a stable resonant wavelength for both modes is attained. Next, the PEI solution streams over the chip for 4 minutes, followed by a 3 minute incubation time by stopping the flow. Finally, the stream is switched back to water in order to obtain a net shift due to adsorption of the PEI. The shifts in resonance wavelength of both the fundamental TE and the fundamental TM mode have been recorded and are displayed in Fig. 3.

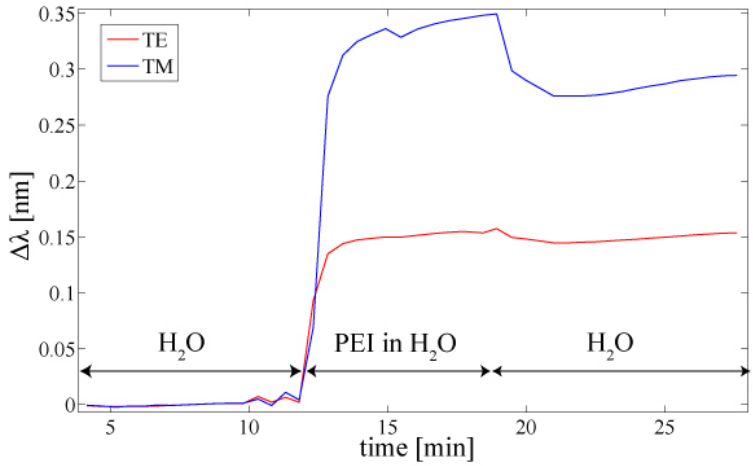

Figure 3. Shift of the resonant wavelength for a TE mode at λ=1542 nm and a TM mode at λ=1536 nm, during the adsorption of a 2mg/ml solution of PEI in water on the silicon surface of the microring.

When the fluids are switched back from PEI in water to water, the negative shift is relatively small, indicating that only a small amount of PEI is being washed off by the subsequent water step, owing to the adhesive properties of PEI. After the second streaming of water the signal stabilises to a net shift of 294 pm for TM, and 153 pm for TE. Note that at this water stage the signal is not flat. We can see a small positive shift, especially visible in the TM curve, implying that there is a slight surface modification in this step as well. This small positive shift amounts to 18 pm for TM and 8 pm for TE. Since we measured the temperature drift for TM as 34 pm/K for TM and 63 pm/K for TE, we can conclude that this is not a temperature effect. Using the calibration scheme and the solving model outlined above, we can use these curves to solve for the thickness and refractive index profile, shown in Fig. 4.

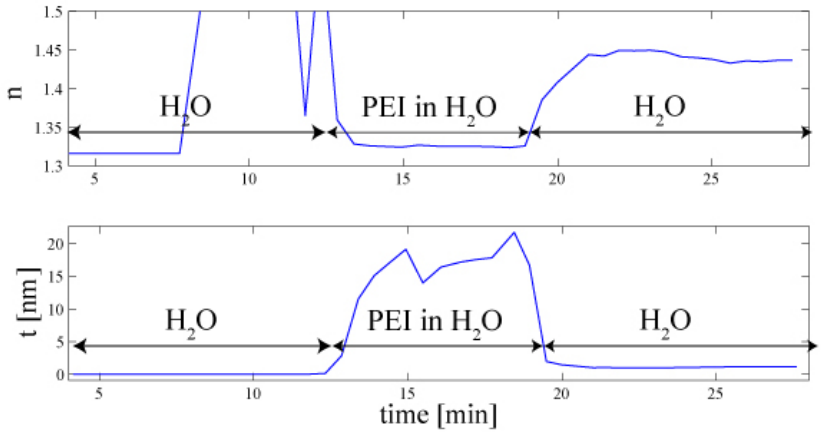

Figure 4. Refractive index and thickness profile of the adsorption of PEI on the silicon surface of the microring.

When solving for (*t, n*) we defined the bulk index as water. This way, we can use the sensor response during the second streaming of water to solve for a layer with unknown thickness and refractive index. As a consequence, the (*t*, *n*) profile during the middle stage, where the bulk index is not water, is not solved adequately. This is of no concern, since we merely want to show what the layer looks like with water as a top cladding. The spikes in the density profile in the first water section are due to the mathematical model: when there is no layer present, the model can solve for any arbitrary thickness. In the last section the thickness stabilises to 1.158 nm with a standard deviation of 9 pm over the 5 last points. The refractive index stabilises to 1.453 with a standard deviation of 0.002 RIU. In [6], a similar thickness is determined with XPS on crystalline silicon. We can see that the small and slow redshift of the TM mode in the second water stage is reflected by a slight decrease in refractive index, and a slight increase in thickness. This shows that the PEI molecules adopt a slightly expanded conformation, adapting to the new regime.

4. CONCLUSIONS

We conclude that we have designed a dual polarisation SOI microring biosensor capable of resolving thin layers of polymers. We have shown that in order to excite both polarisations at the same time, an asymmetrical coupling section can be used, which should have a carefully designed gap of 120 nm. Furthermore, a thin layer of PEI molecules has been adsorbed to the microring surface which amounted to a layer with refractive index of 1.453 and a standard deviation of 0.002 RIU and thickness of 1.158 nm and a standard deviation of 9 pm.

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