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Silicon nitride ring resonator biosensors operated at 1310 nm wavelengths via TM-mode grating couplers

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ABSTRACT

In this work, we demonstrate a full photonic integrated device for performing highly-sensitive biosensing using silicon nitride ring resonators propagating transverse-magnetic modes at wavelengths around 1310 nm. The device includes fully-etched grating couplers as interfaces with external optical fibres. Sensing experiments are performed in a microfluidic environment using bovine serum albumin and anti-bovine serum albumin as biological agents. In comparison with other photonic platforms based on ring resonators propagating transverse-electric modes, our device shows a better detection performance though the efficiency of the grating couplers can be further improved.

Introduction

In recent years, silicon photonics has become the mainstream technology for photonic integrated circuits (PICs) ^{1,2}. The main reason is that silicon PICs can be fabricated monolithically in large volumes at low cost by use of mainstream CMOS processes and technology. Amongst the different fields where silicon PICs show practical utility, the realisation of photonic biosensors for lab-on-chip applications is particularly appealing ³. Indeed, photonic biosensors implemented on silicon PICs perform label-free, show high detection sensitivities, and are ultimately disposable, which is remarkably interesting for point-of-care diagnostics without requiring specialized personnel⁴. Essentially, a biosensing PIC includes a structure whose response to a certain excitation changes when its surroundings are modified by the presence of the analyte to be detected. This happens because the analyte changes locally the refractive index thus modifying the optical response. Notably, silicon nitride (Si_3N_4) is usually preferred as guiding material over silicon due to the lower scattering losses associated with sidewall roughness ⁵ while remaining low-cost and allowing a reasonable level of integration ^{6,7}. In the context of biosensing, Si_3N_4 exhibits additional advantages since it is resistant to diffusion of moisture and sodium ions, hence maintaining a stable refractive index even when exposed to biochemical fluids ^{8,9}.

One of the most widely used photonic structures for biosensing are ring resonators (RRs), constituting a compact circular sensing element which provides a fast readout and presents a high degree of integration with other optical and fluidic components ^{10,11}. Typically, RR-based biosensors operate using the transverse-electric (TE) or even-parity mode at wavelengths around 1550 nm (third optical communications window) ¹². However, it was suggested that the performance could be improved by operating the RR in the transverse-magnetic (TM) or odd-parity mode in the wavelength window around 1310 nm (second optical communications window) ¹³. We recently confirmed experimentally the predicted improvement using Si_3N_4 RRs¹⁴. In such experiments, light was coupled into a waveguide by aligning a lensed optical fibre with the waveguide end at the chip edge, enabling the propagation of both TE and TM modes in the same chip.

However, focusing the light beam onto the waveguide end using a lensed fibre is not a practical approach for a commercial device since it imposes sub-micrometre alignment tolerances. Instead, interfacing between the silicon nitride nanophotonic platform and optical fibres can be performed by grating couplers (GCs) for out-of-plane coupling^{15,1}, which enables testing with relaxed fibre alignment tolerances and a more robust light coupling process for a certain polarisation.

In this work, we go a step further and demonstrate a full PIC for biosensing purposes using Si₃N₄ RRs operated with the TM mode and performing at wavelengths in the 1310 nm window. To this end, we design, fabricate and measure fully-etched TM GCs operating at that wavelength regime. The RR is coupled to the waveguide connecting the input and output GCs via a 50:50 multimode interferometer (MMI) coupler. Our results show that the designed GCs can be used as interfaces to propagate through a PIC the TM mode at 1310 nm wavelengths, which we have previously demonstrated that leads to an improved biosensing performance compared to working with the TE mode at 1550 nm¹⁴.

TM grating coupler: Design and simulations

Although Si₃N₄ waveguides can usually support both TE and TM modes at telecom wavelengths¹⁶, GCs based on Si₃N₄ have been reported predominantly for the TE mode¹⁷. Therefore, TM operating Si₃N₄ GCs must be developed to exploit additional optical functionalities that require TM-polarised light. A fully-etched TM GC based on Si₃N₄, optimised to work at wavelengths around 1550 nm, was demonstrated recently¹⁸, but no significant advances in this area have been reported ever since. Here, we design a fully-etched TM-mode GC based on Si₃N₄ optimised around 1310 nm wavelengths. Notice that fully-etching simplifies the whole fabrication processes since a single lithography plus etching step is required. The design parameters and measuring configuration of the GC are represented in Fig. 1. The thickness of the Si₃N₄ layer (300 nm) and the oxide lower cladding (3.26 µm) were determined by the available wafers. After etching, the GC was covered by silicon dioxide to protect the GC as well as to maximise the coupling efficiency.

The response of the designed GC was calculated using the finite difference time domain (FDTD) method (FullWave tool by Synopsys). We simplified the whole system to two dimensions and disregarded the transverse direction (y-axis), as usually done when designing GCs for integrated optics. In accordance with the previously reported TM GC¹⁸, the numerical simulations showed that the resulting coupling efficiency was low when targeting conventional coupling angles (around 10°). This is mainly due to the large leakage of optical power into the silicon substrate originating from the constructive interference of diffracted light beams at the Si-SiO₂ interface. This means that the coupling loss can be reduced if destructive interference occurs between reflections at the Si-SiO₂ interface, which leads to a decreased power leakage into the silicon substrate. By increasing the coupling angle, the optical power leakage towards the silicon substrate is reduced, hence coupling higher power levels into the Si₃N₄ waveguide. Therefore, the angle was included as a variable parameter to be optimised in simulations, along with the other parameters (pitch, trench, etch depth and oxide uppercladding thickness). The design parameters used in Ref. 18 served as a starting point for the optimisation process. The buried oxide thickness and the waveguide height were defined by the wafer used in the fabrication process, and the etch depth was fixed to its maximum to obtain a fully-etched GC.

Successive parametric optimisations were carried out by varying simultaneously the parameters corresponding to angle, pitch, trench, and oxide uppercladding thickness. In each iteration, the optimum values were selected to maximise the fibre power. To perform the next sweep for each parameter around the optimum value resulting from the previous sweep, the start and end values, as well as the step width, were specified. Large ranges of values along with large steps were introduced for initial optimisation sweeps, reaching a trade-off between resolution, span, and time. In each iteration, the value ranges and the amplitude of steps were reduced, until the optimum values were obtained with the required precision. In

this case, the precision was marked by the fabrication resolution of 20 nm. The final design parameter values and output performance metric resulting from the optimisation procedure are summarised in Table 1. A fibre power value of 0.221 was obtained for the TM mode at 1310 nm, which is equivalent to a coupling loss of 6.6 dB. In Ref. 18 (for the TM mode at 1550 nm) a coupling loss of 6.5 dB was obtained by simulations. Therefore, simulation coupling efficiencies at 1310 nm wavelengths are similar to the ones previously reported for 1550 nm.

Fig. 1. Scheme of the TM GC. **a** Schematic sideview of a cross-section of the whole GC structure, displaying the optical fibre, the silicon nitride core, and the silicon dioxide cladding, along with the design parameters and fixed dimensions. **b** SEM image of a focusing GC with 19 periods before being covered by a silicon dioxide uppercladding.

Table 1. Input and output parameters for the TM GC simulation-based optimisation process.

Parameter		Designed GC
Input	Wavelength	1310 nm
	Polarisation	TM
	Angle	34°
	Buried oxide thickness	3.26 μm
	Waveguide height	0.3 μm
	Pitch	1.415 μm
	Trench	0.605 μm
	Etch depth	0.3 μm
	Oxide uppercladding thickness	0.595 μm
Output	Fibre power	0.221

To later compare with experimental results, the power output obtained from the simulation of a single GC was squared (to consider both the input and the output GC) and then converted to normalised transmission values. These transformations, which consider waveguide losses negligible compared to coupling losses, are summarised by:

$$T_{2GC} = 10 \cdot \log_{10} \left(\frac{P_{mW}^{out}}{P_{mW}^{in}} \right)^2 \quad (1)$$

where T_{2GC} is the simulated normalised transmission considering two GCs, and $\frac{P_{mW}^{out}}{P_{mW}^{in}}$ is the simulated power coupling efficiency of a single GC. Fig. 2 a shows the values obtained by simulations according to equation (1) and Fig. 2 b represents a contour colour map resulting from the two-dimensional (2D) simulation of the TM mode at a wavelength of 1310 nm, illustrating the electric field distribution around the GC and the optical fibre.

Fig. 2. **a** Simulated transmission spectrum for a waveguide integrating the designed TM mode GC for 1310 nm wavelength light input and output. **b** Snapshot of the transversal magnetic field (H_y) around the designed GC for TM mode at 1310 nm (FDTD simulation).

Following the parametric optimisation process, calculations were performed to adapt the dimensions of the GC to the beam spot size, which is larger for 34° compared to conventional 10° angles. Therefore, the TM GC was redesigned in terms of length and width to fit the larger beam spot originated by the increased angle. The fact that the beam spot is larger implies that alignment tolerances are greatly relaxed since the area of overlap is scaled up²¹, which is an advantage only if the GC is redesigned accordingly. The model of geometrical optics, which is valid for light propagation in an optical fibre, was applied to perform the calculations, considering a generic position of the optical fibre with respect to the chip surface and applying trigonometry. To adjust the effective coupling length of the GC to the size of the mode field from the fibre, hence improving the coupling efficiency by increasing the overlap of the waveguide mode and the fibre mode, 31 periods were used.

Fabrication and Experimental measurements

Fabrication

The photonic biosensors were fabricated by standard silicon micro-fabrication methods in a class 10-1000 clean room. The silicon nitride waveguides, resting on a silicon dioxide lower cladding ($3.26 \mu\text{m}$

depth), have a rectangular cross-section with thickness $t = 300$ nm and width $w = 1100$ nm. The fabrication process was based on a direct writing electron beam (e-beam) process on a poly-methyl methacrylate (PMMA) positive resist layer with a thickness of 100 nm. After developing the resist, a metal mask was created by chromium evaporation prior to a lift-off process. This metal mask was used to perform the inductively coupled plasma-reactive ion etching (ICP-RIE) of the silicon nitride layer using fluoride gases (CF_3 and C_4F_8). With the mentioned process, we achieved a good performance in terms of roughness and side wall verticality in the final fabricated samples (propagation losses ~ 2.5 dB/cm). Finally, a 595 nm SiO_2 layer (the optimum uppercladding thickness obtained through simulations) was deposited by plasma enhanced chemical vapor deposition (PECVD). To allow for sensing, windows were opened over the rings. The window opening process required two new e-beam processes also using PMMA. To protect the silicon nitride rings during the silica etching, a first e-beam exposure was performed prior to an evaporation and lift-off of 35 nm of chromium. After that, a second e-beam process was carried out prior to the silica upper cladding etching by using again ICP-RIE etching. Finally, the protection chromium layer was removed by using a chromium etchant bath.

The GCs were fabricated in a focusing fashion (see Fig. 1 b). This way the GC not only transform the wave vector of the incident signal to an in-plane one but also adiabatically adapts the transverse width of the field from the ≈ 10 μm transversal extension of the optical fibre mode to the ≈ 1 μm transverse confinement of the fabricated waveguide. Two sets of devices were fabricated, having the GCs 19 and 31 periods, respectively. The experimental measurements reported below were obtained for devices with 31 periods GCs.

Experimental set-up and methodology

The set-up used to perform the measurements relies on GCs to couple light from the input optical fibre to the chip and from the chip to the output optical fibre. A schematic of the set-up is shown in Fig. 3. A tunable continuous wave (CW) laser, computer controlled and stepped in wavelength, was used to perform a wavelength sweep, and the transmission spectrum was obtained by measuring the received power with an optical power meter. The peculiarity of these measurements is the fact that the input and output optical fibres form an angle of 34° with the vertical, instead of the standard 10° used for TE-modes GCs. An ad-hoc set-up, shown in Fig. 4, was built using 3D-printed wedges to reach the required 34° angle. This change in the inclination of the fibres also imposed different coupling distances, adjusted by the 3-axis positioners.

Fig. 3. Schematic of the vertical coupling set-up for testing the photonic RR biosensors.

The polarisation controller was used to regulate the polarisation of light, by bending the optical fibre with three paddles. The paddles were rotated such that the power received by the power meter was maximised. This polarisation adjustment was made before performing any measurement since the GCs are designed for a specific polarisation (in this case, TM). The sample was placed on a holder and retained by air suction. The movement of the input and output cleaved fibres was controlled in the X, Y and Z directions by adjusting 3-axis positioners. The optical power meter measured the output power in dB units (for 0 dBm input power), which was represented on the computer along with the wavelength of the laser, using a software based on LabView. A camera and a light source were used to visualise the sample and optical fibres during the alignment process.

Fig. 4. Components of the set-up used for the experimental characterisation of the GCs at a wavelength of 1310 nm and a coupling angle of 34° (excluding the microfluidic system used for biosensing).

The measurement process involved a series of steps. First, the input and output fibres were aligned by adjusting their positions until the maximum output power reading was obtained at the power meter. Then, the polarisation of light was adjusted with the polarisation controller, to continue maximising the output power. This process was repeated until the power reading no longer improved. Finally, a wavelength sweep was performed, and the transmission spectrum was recorded.

To characterise the performance of the GCs as part of a biosensing platform based on RRs, the sensors were subjected to bovine serum albumin (BSA) and anti-bovine serum albumin (antiBSA) via a microfluidic channel. A custom flow cell, composed of a double-sided adhesive with a central channel (7x1.5 mm) alongside a piece of methacrylate with two transversal holes (an inlet and an outlet) matching the distance between the ends of the channel, was used to create a fluidic channel on the chip. The required tubing to connect the microfluidic channel with the pump and the analyte container to handle the fluidics properly was inserted through the inlet and the outlet of the methacrylate piece. It was silicone elastomer tubing (with an inner and outer diameter of 0.51 mm and 0.94 mm, respectively) matching to the inner diameter of the inlet and the outlet. The whole fluidic system was assembled by attaching one side of the

double-sided adhesive tape to the chip, leaving the RRs centred at the channel and, subsequently, the other side to the methacrylate piece with the tubing. A syringe pump was operated in withdrawal mode to pull solutions across the sensors at a controlled flow rate (10 $\mu\text{L}/\text{min}$).

Grating coupler characterisation

To experimentally validate the design and optimisation work based on simulations, a fabricated chip containing a series of waveguides with input and output GCs at the ends was measured in the optical set-up. RRs were also included to characterise the whole system used for biosensing. Fig. 5a shows the experimental measurements for the fabricated GCs connected by a waveguide, enabling a comparison to be made with simulation results (considering propagation losses negligible). The representation of experimental transmission spectra includes measurements of several GCs to appreciate the dispersion and calculate the mean to reduce random noise in the readings. A coupling loss of 15 dB per GC is obtained experimentally propagating the TM mode at 1310 nm wavelengths. Despite the coupling efficiency being lower than predicted by simulations, Fig. 5 b shows that the whole system including the RR works, since the characteristic periodic resonances appear in the transmission spectrum.

Although the simulations can be used to optimise the performance of the GC around the design wavelength, it should be noted that the absolute power value is not accurate. Considering that waveguide losses are usually considered negligible compared to GC losses, some of the possible sources of disagreement could be fabrication imperfections which cause undesired losses, as well as the implicit assumptions and the oversimplified 2D model used to simulate the GCs (not even accounting for the GC curvature and the adiabatic reduction of the waveguide width). Due to these approximations, there is often a large mismatch between simulations and measurements, which in our case is even more evident because of the larger angle of incidence. Nevertheless, it can be appreciated that the maximum coupling efficiency is obtained around the design wavelength of 1310 nm, which was the main goal of the design process. Still, more complex strategies to build the GC, for instance via dielectric metasurfaces^{20,21}, can be explored in order to increase the coupling efficiency up to values comparable with TE GCs.

Fig. 5. Experimental transmission spectra for input and output GCs connected by **a** a waveguide (showing the mean of the measurements in red) and **b** a waveguide coupled to a RR by an MMI. Insets: Optical microscope images of the structures being measured.

Biosensing assay using ring resonator sensors

The designed GC was used to couple light to and from a PIC containing a RR exposed to the surrounding media which acts as a biosensor. The biosensing experiment required a series of steps to finally obtain the sensorgram represented in Fig. 6 a. The results obtained from the biosensing assay demonstrate the successful propagation of the TM mode at 1310 nm wavelengths through the Si_3N_4 based PIC via the designed GCs, since the sensitivity values obtained from the experiment are close to those predicted by simulations and to the butt-coupling measurements guiding TM mode¹⁴. Moreover, it is proved that, although the GC design could be improved further, the relatively low coupling efficiency is not an impediment to carry out biosensing assays. It can be appreciated from Fig. 6 that a wavelength shift of 0.46 nm occurs after the first PBS (phosphate-buffered saline) buffer rinse (due to the BSA binding), whereas a shift of 0.95 nm remains after the final PBS rinse (due to the antiBSA binding to the BSA attached in the previous step).

Fig. 6. a Sensorgram resulting from a biosensing experiment with BSA and antiBSA in PBS buffer, for the TM mode at 1310 nm wavelengths. The dashed vertical lines indicate a change in the flowed solution, which takes around 8 minutes to reach the RR. **b** Measured transmission spectra during the experiment at the times pointed by arrows on the sensorgram (which correspond top to bottom, to the initial spectrum in PBS, the displacement caused by the BSA and the displacement caused by the antiBSA binding to the previously attached BSA). The position of the peak shown in green is tracked throughout the experiment to obtain the sensorgram representation.

Conclusions

In this work, we have demonstrated a complete platform towards practical high-performance photonic biosensing in a silicon-based PIC using the TM mode at wavelengths in the second optical communications window. To this end, we have designed and experimentally validated for the first time a fully-etched Si_3N_4 GC operating for TM modes at wavelengths around 1310 nm which can be fabricated in the same lithography step as the rest of the PIC components. This TM-performing GC extends the range of currently available silicon nitride based components while enabling low-cost and mass manufacturing of the corresponding PICs. In the context of biosensing applications, TM modes offer higher sensitivities over their TE mode counterparts while operating at 1310 nm increases the quality factor compared to 1550 nm wavelengths. A PIC including the GCs designed in this work has already been fabricated and proved to

work satisfactorily for biosensing assays. Other fields where a relatively high loss is not critical but guiding TM mode at 1310 nm is a requisite can benefit from the use of this GC design. Moreover, the proposed TM GC design can serve as a starting point for further optimisation work to extend its use to other applications, such as optical processing and communications.

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