Optical Materials 72 (2017) 596–601

Contents lists available at ScienceDirect

Optical Materials

journal homepage: www.elsevier.com/locate/optmat

Porous silicon micro-resonator implemented by standard photolithography process for sensing application

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ARTICLE INFO

Article history:
Received 7 March 2017
Received in revised form
21 June 2017
Accepted 2 July 2017

Keywords:
Sensors
Integrated optics
Optical design and fabrication
Resonators
Porous silicon

ABSTRACT

A micro-resonator based on porous silicon ridge waveguides is implemented by a large scale standard photolithography process to obtain a low cost and sensitive sensor based on volume detection principle instead of the evanescent one usually used. The porous nature of the ridge waveguides allows the target molecules to be infiltrated in the core and to be detected by direct interaction with the propagated light. Racetrack resonator with radius of 100 µm and a coupling length of 70 µm is optically characterized for the volume detection of different concentrations of glucose. A high sensitivity of 560 nm/RIU is reached with only one micro-resonator and a limit of detection of 8.10^{-5} RIU, equivalent to a glucose concentration of 0.7 g/L, is obtained.

1. Introduction

The ability to rapidly detect, identify and monitor chemical or biological species is critical in many economic and societal problems such as environmental monitoring, health monitoring and security applications. The detection of bio-chemicals traces requires analytical tools which can detect these traces within an acceptable time, sensitive enough to detect really low concentrations and selective not to be affected by other factors in the environment. It is now well-established that the development of compact portable sensors and analyzers can be of great help to overcome the inherent limitations of laboratory techniques in terms of both spatial and temporal resolutions.

Micro Resonators (MRs) are now widely investigated for sensing applications. High quality factors up to 10^8 can be obtained with microspheres [1,2] which allow to achieve low optical detection limits as they are able to react to a monolayer of molecules adsorption [3–5]. However, microsphere resonators lack of integration capability [6], which limits their use in practical applications in integrated optics. To solve these problems, integrated micro-ring, racetrack or micro-disk resonators are used, albeit with reduced Q factors [7] in the range of 10^4–10^5. They can be integrated on Photonic Integrated Circuits (PIC) which provide a route toward small, low-cost and very rugged optical systems and could therefore be a game-changer for sensor systems. Most integrated sensors developed up to now are geared toward classical surface detection by using evanescent wave sensing: the most studied materials used for sensing applications are bulk semiconductors or polymers and the detection principle is based on interaction of the probed molecules with the evanescent part of a wave [7]. This interaction leads to a wavelength shift of the MR response depending on the refractive index variation induced by the probed molecules. The sensitivity is thus defined as the ratio between this wavelength shift and the refractive index variation in nm/RIU (Refractive Index Unit). Sensitivities of 200 nm/RIU and of 230 nm/RIU have been obtained respectively using bulk material with one polymer micro-ring for glucose detection [8] or with one Si_3N_4 micro-disk for LiCl detection [9].

However, the sensitivity of integrated MRs can be improved by optimizing the interaction between the molecules and the optical wave. This optimization can be provided by the use of a porous material such as porous silicon (PS) for the waveguide core. In this case, the principle of the sensor is based on a volumic detection which allows a direct interaction between the propagated light and

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http://dx.doi.org/10.1016/j.optmat.2017.07.005
0925-3467/

the molecules to be detected [10–12].

Indeed PS is a widely studied material regarding its optical properties in many applications in optoelectronics [13] and also over the past decade for chemical [14,15] and biological sensing [16,17]. The large internal surface of porous silicon and its biocompatibility constitute significant advantages for biosensing [18]. This material is used as a host to various molecules that can be in solution in the pores, or grafted to the internal surface of silicon after its functionalization [17]. Optical PS sensors using refractive index variation have already been the subject of various studies [11,12], the number of which continues to grow due to the interest of this porous material to increase the sensitivity of the sensors. Recently the first all porous single side coupled micro-ring resonators obtained with e-beam lithography, allowed to reach a detection sensitivity of 380 nm/RIU when salt water solutions are infiltrated into the device [19].

In this paper, the work aims to study an all porous single side coupled micro-racetrack resonator fabricated with a large scale standard photolithography process and to enhance the sensitivity of the sensor. In a first part, the design, the fabrication and the setup of optical characterization around 1550 nm are described. On the second part, the results of optical characterization of the MR are reported and homogeneous sensing experiments using glucose aqueous solution with high sensitivity are presented.

2. Experimental

2.1. Materials and design

Three consecutives PS layers have been prepared by electrochemical anodization of a heavily doped P (100) silicon substrate with a 5 mΩ cm resistivity and using applied current densities of respectively 1, 50 and 80 mA/cm^2 for specific times. The electrolyte was formed by combining hydrofluoric acid (50%) with ethanol and deionized water in the ratio of 2-2-1 respectively. Following this, the PS layers were partially oxidized at 500 °C for 5 min to passivate the surface and to obtain a hydrophilic surface.

The first PS layer (1 mA/cm^2) is a thin barrier layer with a very low porosity on the top of the two other layers constituting the core and the cladding layers of the PS waveguide. The current densities of these two layers have been chosen both to get high porosities after oxidation treatment to reach high MR sensor sensitivity and to get a single mode propagation with micronic dimension waveguides. This structure with three layers will be submitted to a photolithographic process inducing a photosensitive resin deposit, thus the first layer constitutes a barrier layer that will prevent the infiltration of this resin in the two lower layers.

The thickness of each porous layer has been fixed and then verified by cross section SEM (Scanning Electron Microscope) measurements. From these thicknesses, porosities and then refractive indices are determined by the adjustment of the calculated reflectance spectra of each porous layer with the experimental ones using Bruggemann model [20,21]. The partial oxidation step of porous layers induces a porosity decreasing due to silica expansion [22]. The values of porosities and refractive indices of the PS layers (core and cladding), are given in Table 1. Values of refractive indices for the porous layers are given initially with air superstrate ($n_{\text{air superstrate}}$) and have also been calculated with a deionized water superstrate ($n_{\text{water superstrate}}$) as the MR will be used for sensing experiment with glucose solubilized in deionized water. The water infiltrates pores, that is why the refractive index of each layer increases. The characteristics of the first layer are not indicated as it is a barrier and a sacrificial layer that will be removed thereafter during the process.

Table 1
Thickness, porosity and refractive index (@1550 nm) for air and deionized water superstrate for the partially oxidized PS layers.

Porous layers	Thickness (μm)	Porosity before oxidation (%)	Porosity after partial oxidation (%)	$n_{\text{air superstrate (after oxidation)}}$	$n_{\text{water superstrate (after oxidation)}}$
Core	2.0 ± 0.1	66 ± 2	56 ± 2	1.55 ± 0.06	1.77 ± 0.06
Cladding	5.0 ± 0.1	73 ± 2	63 ± 2	1.38 ± 0.06	1.62 ± 0.06

2.2. Fabrication: straight ridge waveguide and micro-resonator

PS ridge waveguides have been implemented using a large scale standard photolithography process on the PS layers. The process is described for the PS MR in Fig. 1a, b and c. A positive SPR photosensitive resin layer is deposited by spin coating on the top of the structure, constituted by the thin barrier. Patterns are then produced under UV exposure through a well-defined chrome mask designed using commercial Olympios software. To obtain the aimed PS ridge waveguides, a first trifluoromethane (CHF_3) RIE (Reactive Ion Etching) - ICP (Inductively Coupled Plasma) plasma is performed to remove both the barrier and core layers unprotected by the resin (Fig. 1b). Once the cladding layer is reached, a O_2 RIE - ICP plasma is applied to etch the residual resin. Finally, the barrier layer is removed from the obtained waveguides by a CHF_3 RIE - ICP plasma.

The aimed dimensions of the PS ridge waveguides are a height of 2 μm and a width of 2 μm in order to obtain a single mode propagation. A previous study on porous silica MR has led to the design of MR with several radius, coupling length and gap values [23]. Among the different studied MR geometries, a racetrack PS MR with a radius R of 100 μm and a coupling length Lc of 70 μm has been chosen (Fig. 1d). The straight and the racetrack MR ridge waveguides are separated by a gap of 0.5 μm. These values have been chosen because they allow to obtain the coupling ratio near the critical one which enables optimal contrast of the transfer function with a deionized water superstrate.

Following the fabrication steps, SEM measurements have been performed in order to verify the implementation of the PS MR. The cross section of the etched waveguide is reported in Fig. 1e showing the PS core and cladding layers. A square core waveguide is obtained with good etched edges with low roughness. The top view SEM image of the separation between the straight and the racetrack MR ridge waveguides, which appears in dark grey light (Fig. 1f), shows that the gap has been successfully made along the coupling length of the racetrack.

2.3. Optical characterization setup

As shown in Fig. 2, the output from a tunable wavelength laser (Yenista Tunics T100S-HP), around 1550 nm, is coupled in the waveguide using a lensed fiber with a mode radius of 2 μm. A second lensed single mode fiber is also used to couple the output of the straight waveguide to a power-meter. Piezo-controlled stages are used to position the input and output fibers with the help of an infra-red camera. To make easier the analyses of the spectral responses of the MR, it is suitable to have one selected polarization to optimize the characteristics values, such as the quality factor Q and the contrast C, of the PS MR. Therefore, a polarization controller is