An integrated optical sensor in Silicon-on-Insulator for detection of hydrogen gas

Nebiyu Adello Yebo

Promoters: prof. dr. ir. Roel Baets, prof. dr. ir. Dries Van Thourhout
Supervisors: dr.ir.Dirk Taillaert, ir. Joris Roels

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Abstract

Numerical and Experimental study of ring resonator based integrated optical hydrogen sensors have been carried out in this thesis. Two schemes of sensor implementation are adopted through two separate sensitive coatings on SOI micro-ring resonators. The first scheme potentially exploits optical and mechanical effects from nanometer scale palladium coating. Where as, thermo-optic effect from combustion of hydrogen in air by the aid of Tungsten Oxide (WO₃) catalytic coating is used in the second sensor implementation.

While sensitivity to hydrogen is achieved from both implementations, significant performance has been achieved from the thermo-optically functioning WO₃ coated ring resonator based sensor. Around 1.2nm resonance shift is measured for hydrogen concentration below the Lower Explosive Limit (LEL) with a response time of less than a minute. Further enhancements in sensor sensitivity are promised with proper choice of operating conditions, such as temperature and gas flow rate.
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Abstract—Simulation and experimental study of two integrated hydrogen sensing schemes on SOI ring resonators has been carried out in this thesis. Resonance shift of 1.2nm for 3% hydrogen in air is achieved from one of the sensing schemes. This scheme uses the heat generated from catalytic combustion of hydrogen to modify the resonator performance through thermo-optic effect.

I. INTRODUCTION

Hydrogen has been of a considerable interest as a clean source of energy. However, the highly volatile and flammable nature of this gas has been an impediment to its potential applications.

Several sensor implementations have been proposed and utilized as part of early warning systems in hydrogen environments. Majority of optical hydrogen sensors reported to date have been based on optical fibres [18-22]. Though some forms of these sensors offer good features such as, multiplexing of sensor arrays and high sensitivity, they are not suitable for integrated sensor realization. On the other hand, the capability to integrate sensors provides advantages such as low cost production, easy implementation of sensor arrays and high compactness.

Moreover, integrated optical hydrogen sensors reported to date have been built on photonic wires or waveguides [10, 23]. Such sensor implementation, however, provides less multiplexing capability and requires adequate waveguide length for effective interaction between guided SPR waves and the matter to be sensed.

High index contrast Silicon-on –Insulator (SOI) ring resonators have recently been used for sensing applications, such as bio-chemical sensing[4]. They can be very compact compared to guided mode /SPR waveguide sensors, and, are inherently suited for wavelength multiplexing of sensor arrays.

II. THE HYDROGEN SENSORS

Two forms of SOI ring resonator based integrated hydrogen sensors are studied and fabricated in this thesis. In the first sensing strategy palladium (Pd) is coated on BCB clad SOI ring. Hydrogen sensing in this scheme can potentially come from both optical and mechanical effects. In an inert gas environment such as nitrogen, hydrogen is well adsorbed in palladium to form Palladium – hydride system. This leads to change in volume and dielectric permittivity of Pd. This change can be coupled to the underlying ring structure through mechanical strain and evanescent field interaction. The resulting shift in resonance is used to sense hydrogen. The mechanical strain can manifest itself through elasto-optic effect and physical stretching of the ring.

In the second scheme, Tungsten Oxide (WO₃) catalytic coating is made on SiO₂ clad SOI ring. The WO₃ catalytic coating facilitates hydrogen combustion in air at lower concentrations without creating fire. The resulting heat reaching the waveguide region modifies the effective index through thermo-optic effect, hence leading to resonance shift and sensing.

Basic Ring Resonator Based Sensing

In an optical ring resonator, light propagates in the form of whispering gallery modes (WGM), which result from the total internal reflection of light along the curved surface.

The change in resonance wavelength with sensing is expressed by

$$\frac{\Delta \lambda}{\lambda} = \frac{\Delta L}{L} \cdot \frac{\Delta n_{eff}}{n_{eff}}$$

Where $\lambda$, $n_{eff}$, and $L$ are resonance wavelength, effective mode index and roundtrip length of the ring

Accordingly, change in effective index or physical length of the ring can be exploited for sensing applications.

III. SIMULATION RESULTS

Pd Coated Sensor

Optical simulations in FIMMWAVE have shown that the optimum cladding (either SiO₂ or BCB) thickness required for relatively better sensor operation is around 400nm. The sensor sensitivity due to evanescent field interaction promised with such thicknesses and 10 nm Pd coating is about 20pm for 1% hydrogen in Pd. Though lower thicknesses have been predicted to offer better sensitivity, the accompanying wider resonance bandwidths (higher than 0.6nm) make them not
feasible to work with the expected small resonance shifts. The mechanical simulations in COMSOL have further predicted that the sensing contributions from mechanical strain are insignificant for thinner Pd coatings. Additional resonance shifts of 5pm and 40pm are estimated from stress optic effect and physical change in ring diameter respectively for 500nm Pd coating. However, deposition of such higher coatings has happened to be impractical due to poor adhesion of Pd on BCB and due to stresses developed at interfaces. Consequently, all the sensing in this scheme is expected to come from evanescent field interaction.

**WO\textsubscript{3} Coated Sensor**

With the help of Heat transfer simulation in COMSOL and the accompanying Optical simulation in FIMMWAVE, it has been estimated that around 78pm resonance shift is expected for 1 °C temperature change on the SOI sample surface. The SiO\textsubscript{2} cladding used for these simulations is 720nm thick. Similar simulations have further shown that around 10 and 15pm improvements in resonance shift can be achieved by lowering the cladding thickness to 600nm and 540nm respectively.

**IV. SENSOR FABRICATION AND EXPERIMENTAL RESULTS**

**Fabrication**

The basic SOI ring resonator structures are designed in the Photonics Research group and fabricated at IMEC – Leuven using 248nm deep UV lithography. The fabricated structures have 220nm thick Silicon core. The top BCB cladding and the Pd layer are coated at the University clean room. The WO\textsubscript{3} coating on SiO\textsubscript{2} clad structures is done in collaboration with the sensors research group “Materia Nova, Cellule Capteurs” in Mons.

**Experimental Setup**

Input light from Tunics Plus tuneable laser is vertically coupled through glass window to the sample contained in sealed gas chamber. Coupling from optical fibre to the waveguide structures is achieved with grating couplers. The output light from the rings (through grating couplers) is collected by XenICs (Xeva-511) IR camera.

**Measurement Results from Pd Coated Sensor**

Two ring resonators in series are used in this sensor implementation. One of them is Pd coated and the other serves as reference for temperature effects. Thermal effects have been observed on this sensor through blue shifts on both the reference and Pd coated rings. 40pm net red shift on the Pd coated ring is measured from the difference in blue shifts on the two rings for 2% hydrogen in nitrogen. The blue shifts are to be attributed to temperature gradient between the sample surface and the hydrogen gas. Low sensitivity and slow response for subsequent hydrogen exposures along with the aforementioned thermal influence are the limitations observed with this sensor.

**Measurement Results from WO\textsubscript{3} Coated Sensor**

Far better results have been achieved from this sensor implementation. 340pm and 1.2nm resonance shifts are measured for 1.4% and 3% hydrogen in air at 40 °C sample temperature and 1.3L/min constant air flow. This approximately corresponds to 10 °C sensor temperature rise for 3% hydrogen concentration.

![Image of resonance shift at different hydrogen concentrations](image)

It has also been noticed that the sensitivity can be tuned to a required level through appropriate choice of operating temperature and gas flow rate.

**V. CONCLUSION**

Novel integrated hydrogen sensor implementation has been achieved on SOI ring resonator structures. While sensitivity to hydrogen is observed from both the Pd coated and WO\textsubscript{3} coated sensors, impressive results have been achieved from the latter. Beyond the larger resonance shifts (more than a nanometer) achieved for hydrogen concentrations below Lower Explosive Limit, the WO\textsubscript{3} coated sensor is accompanied with further interesting features. Fast response and tuneable sensitivity with operating temperature and flow rate are among such features. Possibility for optical heating of the sensor further ensures its safe implementation in potentially flammable hydrogen environment. Ultra-high compactness, inherent multiplexing capability, compatibility with fibre networks, and potential for inexpensive production are other interesting features. With such qualities, expected from ideal hydrogen sensor, the thermo-optically functioning SOI ring resonator based sensor can largely contribute to solve the current safety concern in hydrogen environment.
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<tr>
<td>BCB</td>
<td>bisbenzocyclobutene</td>
</tr>
<tr>
<td>CMOS</td>
<td>Complementary Metal –oxide Semiconductor</td>
</tr>
<tr>
<td>FBG</td>
<td>Fibre Bragg Grating</td>
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<tr>
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<td>Infra Red</td>
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<tr>
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<tr>
<td>SOI</td>
<td>Silion –on - Insulator</td>
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<tr>
<td>SPR</td>
<td>Surface Plasmon Resonance</td>
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<tr>
<td>TE</td>
<td>Transverse Electric</td>
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<tr>
<td>TM</td>
<td>Transverse Magnetic</td>
</tr>
<tr>
<td>UV</td>
<td>Ultra Violet</td>
</tr>
<tr>
<td>WGM</td>
<td>Whispering Gallery Mode</td>
</tr>
<tr>
<td>WO₃</td>
<td>Tungsten Oxide</td>
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Chapter 1

Introduction

1.1 Motivation

Hydrogen has been of a considerable interest as a clean, sustainable and abundant energy source. However, a public concern exists about using hydrogen as fuel. It is highly volatile, easily flammable and explosive gas; hydrogen concentration as low as 4% in air can cause it to burn. This concentration threshold is often termed as Lower Explosive Limit (LEL). Owing to this fact, reliable safety systems have to established before the potential benefits of the hydrogen fuel can be practically exploited.

As part of the solution to safety issues, there has been a large demand for highly sensitive, fast, inexpensive, lightweight and reliable hydrogen gas sensors for leak detection and early warning.

Various semiconductor based sensors have been proposed and commercialized [15]. Most of them relied on electrical means of detection. [15]. Palladium (Pd) – gated FET is one such sensor [12]. Optical sensors appear to be attractive for hazardous environments as there is no concern for sparkling possibilities. Immunity to electromagnetic interference, multiplexing capabilities, remote sensing, and possibility of high sensitivity are other factors which make these sensors interesting.

Several optical hydrogen sensors, such as based on bulk components [16, 17], fibre optics [18-22], and integrated optics [10] have been reported. Optical hydrogen sensing has been largely based on optical fibres. The field of optical fibre hydrogen sensors includes Fibre Brag Gratings (FBG)[14,22], micromirrors[19], evanescent fields, surface plasmon resonance (SPR) effects, and Interferometric techniques[18]. Majority of these sensors were used to detect hydrogen concentrations in excess of 1% to the LEL (4%).
1.1 Motivation

Owing to the sensitive nature of palladium to hydrogen, many of the sensors demonstrated so far, including the electrical ones, incorporate it as sensitive component. The change in elastic and optical properties of palladium under exposure to hydrogen is exploited to achieve sensing in these devices.

In micromirror fibre optical sensor configuration [19], a thin palladium film is evaporated at cleaved end of a multimode fibre, and the change in reflection is monitored. Despite, the simplicity and inexpensive implementation of this sensor, its multiplexing capability is very limited.

In another sensor configuration, in which interferometric techniques are used to detect hydrogen, Pd is coated on one arm and the optical path difference induced due to elastic deformation in the presence of hydrogen is used for detection. However, this sensor involves complex read-out mechanism and is unsuitable for multiplexing [22].

Hydrogen sensors based on fibre brag gratings, among other fibre optic sensors, seem to offer better features in that they easily offer multiplexing capabilities for sensor arrays, thereby reducing the number of detection (read-out) units involved. Gratings of different periods can be written on the same fibre for implementation of various sensors. In one of FBG based sensors reported [22], Pd layer of a few hundred microns is coated on FBGs. The refractive index change induced in the gratings due to elasto-optic effect on exposure to hydrogen shifts the Brag wavelength.

Recently, FBG based sensor which uses Tungsten Oxide sensitive layer instead of the widely used Pd, is reported [14]. This sensor takes the advantage of heat produced from burning of hydrogen in air to modify the grating period and the corresponding shift in brag wavelength is measured as function of hydrogen concentration. The response time of such sensors is better than Pd based sensors. Hysteresis and change in diffusion rate of hydrogen with Pd thickness and hydrogen concentration are noted limitations which affect the response of Pd based sensors.
In general, the Pd based sensors are often suited to hydrogen in nitrogen atmosphere since inert gas carrier is needed for better hydrogen adsorption in Pd. Tungsten Oxide based sensors, on the other hand, are suited for hydrogen sensing in air due to the oxygen requirement for combustion.

### 1.2 Optical Sensor Integration and the Silicon-on-Insulator (SOI) Ring Resonators

The fibre optic hydrogen sensors mentioned so far are not suitable for integration. On the other hand, the capability to integrate sensors provides advantages such as low cost production, easy implementation of sensor arrays, and suitability to application which require very small and light sensors.

Integrated sensors using guided mode planar waveguide with pd sensing layer [23], and sensor based on surface plasmon resonance (SPR) [10] on palladium have been reported to date. However, the size of these sensors is likely to be limited by the minimum length required for adequate interaction between the gas and the evanescent waves or the surface plasmon waves. Furthermore, such sensor implementation gives less flexibility for multiplexing of sensor arrays.

High index contrast (HIC) waveguide structures in Silicon-on-Insulator platform are becoming more attractive for wide range of integrated photonic applications. The high index contrast provides increased control over the propagation direction of light in small structures. Such waveguides can be bent to small radii to form very small ring resonators, which can support guided modes which propagate very close to the ring surface.

HIC ring resonators have recently been used for sensing applications, such as bio-chemical sensing [3]. They can be very compact compared to guided mode waveguide sensors, and SPR waveguide sensors. This is due to the fact that light circulates along the ring repeatedly interacting with matter which we want sense.
Moreover, sensor arrays can easily be implemented using resonators as the basic sensing scheme lends itself directly to multiplexing advantages. Sensing using these structures manifests itself with shift in resonance wavelength of the rings. Hence several such sensors working at separate resonances and designed for sensing various matters can easily be multiplexed. Compatibility of these sensors with optical networks along with the multiplexing feature provides wide opportunity for ideal sensor implementation.

The silicon on insulator platform, which these ring resonator sensors are built on, makes them largely suited to inexpensive mass production. In situation where, ring resonators are made from Silicon as a waveguide material, compatibility with the already well established CMOS fabrication tools is perfectly met and cheap production is possible.

In this thesis, sensors built on high index contrast SOI ring resonators are studied for hydrogen gas detection. The resonators are constructed from 220nm thick silicon core material on top of 2µm thick buried silicon oxide layer which lies in between the core and the Silicon substrate. The top cladding material can be air, SiO₂ or a Polymer.

1.3 Overview of the Thesis Work

Two separate schemes using sensitive layers of different materials are studied for the sensor implementation. The sensitive materials are coated on top cladding of the ring structures as depicted on fig 1.1. One of the sensors uses Pd as sensing layer where as Tungsten oxide (WO₃) catalytic layer is used in the second sensor scheme. Pd based sensing potentially combines both optical and mechanical changes induced in the metal due to hydrogen. The optical change makes itself felt to the ring through evanescent field from the ring , where as the mechanical change can deform the ring mechanically or modify the optical property of the ring through elasto optic effect. On the other hand , sensing with WO₃ exploits the heat generated from catalytic combustion of hydrogen in oxygen environment. This heat alters the optical properties of the ring through thermo optic effect and causes the resonance to shift.
In this work, simulation, fabrication and characterization of the hydrogen sensors is carried out. The following chapters provide detailed descriptions of these tasks. Interesting results have been achieved from this work.
Chapter 2

Theory of Guided Mode Ring Resonator Based Sensing and Simulation Strategies

Introduction

Interests in integrated optical sensors have recently been increasing because of their attractive features such as, their immunity to electromagnetic interference, high sensitivity, good compactness, robustness and high compatibility with fibre networks. Various forms of guided-wave optical sensors have been proposed, such as those based on directional couplers[1], grating assisted couplers[2], and optical micro cavites [3]. Optical microring resonators, in particular, are becoming attractive for bio/chemical and gas sensing applications.[4,5].

The basic sensing scheme of guided mode sensors is based either on the effective index change of the mode or/and on the change in physical sensor dimension. While guided wave sensing can be achieved using waveguides or photonic wires, ring resonators provide an added feature for more compact sensor implementation. Since light passes only once in a waveguide, longer waveguide may be required in order for adequate interaction between the evanescent light and the matter to be sensed. This often sets threshold sensing limit with such sensors.

In contrast, in an optical ring resonator, light propagates in the form of whispering gallery modes (WGM), which result from the total internal reflection of light along the curved surface. WGM is a surface mode which circulates along the resonator surface and interacts repeatedly with the analytes (matter to be sensed) on its surface through WGM evanescent field. Accordingly, the effective light-analyte interaction is not limited by the sensor physical size; it is determined rather by the number of revolutions of the modes supported by the ring.
2.1 Guided Wave Ring Resonator Based Sensing

2.1.1 Ring Resonator Overview

Ring resonator is a travelling wave resonator in which the wave interferes with itself. In resonator arrangement in which the ring is coupled to two waveguides as shown in figure 2.1, the input light is transferred (completely, if lossless cavity) to the output port at resonant wavelengths. As demonstrated on the figure 1, input light coupled through port A of one of the waveguides (WG1), is transmitted straight to port B under off-resonance condition. At resonance, this input is transferred to the second waveguide (WG2) and leaves through port C. The gap between the ring and the waveguides determines the coupling.

Figure 1, ring resonator in channel- drop configuration

The main performance characteristics of these resonators are the free-spectral range (FSR), the quality-factor, the transmission at resonance, and the extinction ratio. The major physical characteristics underlying these performance criteria are the size of the ring, the propagation loss, and the input and output coupling ratios (equivalent to the reflectivities of a Fabry–Perot resonator). There are various components of losses, including sidewall scattering loss, bending radiation loss, and substrate leakage loss.
2.1 Guided Wave Ring Resonator Based Sensing

**The free spectral range**

The Free Spectral Range is the spectral spacing between resonant wavelengths, given by:

\[ FSR = \frac{\lambda^2}{n_{eff} L} \]  \hspace{1cm} (2.1)

**Quality factor**

The quality factor is the measure of lifetime or wavelength selectivity of the resonator; given as the ratio between the resonant wavelength and the 3dB bandwidth.

\[ Q = \frac{\lambda}{\Delta \lambda_{3db}} = \frac{\tau A \pi n_{eff}}{(1 - \tau^2 A^2) \lambda} \]  \hspace{1cm} (2.2)

Where
- \( \tau \) is coupling loss
- \( A = e^{-\frac{\pi}{2} \tau L} \) is the amplitude over half the round trip of the ring
- \( L \) is the round trip length of the ring
- \( n_{eff} \) is the effective mode index

Particularly for sensing applications, reasonable FSR and high Q are useful. Owing to the small dimension of the resonator (radius of 4 \( \mu m \) - 5\( \mu m \)) large spectral range is achievable. The Q factor is determined largely by the losses. Sensing layers added on the ring surface may contribute to further to these losses and reduce the quality factor.

**2.1.2 Fundamental Sensing Principle**

As mentioned in the introductory section, guided mode ring resonator based sensing fundamentally relies on effective index change or change in physical length of the resonator. The sensing contribution from physical modifications comes in cases where the structure is under mechanical strain.

The WGM spectral position or resonant wavelength is \( \lambda \), is related to effective index according to the resonant condition by:

\[ \lambda = \frac{2 \pi n_{eff}}{m} = \frac{L n_{eff}}{m} \]  \hspace{1cm} (2.3)
2.1 Guided Wave Ring Resonator Based Sensing

Where,
- $r$ is the ring radius
- $n_{\text{eff}}$ is effective mode index
- $m$ is order number
- $L$ is the ring round trip length

Following equation 2.3, the shift in resonant wavelength with effective index and physical dimension change can be given by:

$$\frac{\Delta \lambda}{\lambda} = \frac{\Delta L}{L} + \frac{\Delta n_{\text{eff}}}{n_{\text{eff}}}$$ \hspace{1cm} (2.4)

$n_{\text{eff}}$ changes when the refractive index on the surface of the ring is modified by the interaction of analytes with sensing layer. Mechanical influence on the ring structure could also modify the physical length of the ring.

The effective light-matter interaction length is determined by the quality factor of the ring, given by the relation,
Where $Q$ is the quality factor, $\lambda$ is the resonance wavelength and $n_{\text{eff}}$ is the effective index of the guided mode. Hence, the effective interaction or sensitivity is enhanced with quality factor.

### 2.2 Physical Effects Leading to Resonance Shift in Micro-ring Structures

In this section, possible effects which induce optical or mechanical modifications, and contribute to sensing effect in ring resonators are discussed.

In the presence of analytes on the surface of the ring structure, the reaction between these analytes and the sensing layer may lead to optical, mechanical or thermal changes in the ring.

#### 2.2.1 Optical Modifications

In common sensing applications, such as bio/chemical sensing, the interaction between molecules and sensing layer leads to change in refractive index in the layer. Consequently, the light mode supported by the ring experiences this change through its evanescent field, leading to change in effective index. This, in turn, results in shift in resonance wavelength of the mode as described by equation (2.4).

Particularly, in the hydrogen sensor dealt in this thesis, the reaction between hydrogen and the palladium (Pd) sensing layer results in change in optical properties of Pd. As the result, both the refractive index and optical absorption of Pd are modified depending on the hydrogen concentration. More on this reaction will be covered in the next chapter.

In addition, the same Palladium – Hydrogen interaction mentioned above, modifies the mechanical properties of Pd, leading to physical expansion of the layer. This strain is likely to be felt by the underlying core and cladding layers. The resulting stress in these layers can lead to optical modification in the layers through elasto-optic effect.
2.2 Physical Effects Leading to Resonance Shift in Micro-ring Structures

**Elasto-optic Effect**

Elasto-optic effect, also called photo-elasticity, is change in refractive index of some materials under the influence of elastic deformation. This effect couples the mechanical strain/stress to the optical index of refraction of the material.

The elasto-optic effect is described by

\[ \Delta \eta_{ij} = \Delta \left( \frac{1}{n^2} \right)_{ij} = p_{ijkl} s_{kl} \]  \hspace{1cm} (2.6)

Where
- \( \Delta \eta \) is the change in optical permeability (dielectric) tensor
- \( S \) is the strain tensor.
- \( p \) is the elasto-optic coefficient tensor described by 6x6 matrix.
- \( i, j, k, l \) are integers 1, 2, 3..., 6 which indicate specific tensor component

The index ellipsoid of a crystal in the presence of an applied stress field is thus given by

\[ (\eta_{ij} + p_{ijkl} s_{kl}) x_i x_j = 1 \]  \hspace{1cm} (2.7)

Taking into account the symmetry of the permeability and stress tensors, it is often common to write in contracted form as:

\[ \Delta \eta_{ij} = \Delta \left( \frac{1}{n^2} \right)_{ij} = p_{ij} s_j \]  \hspace{1cm} (2.8)

The equation for index ellipsoid in the presence of a strain field can then be written as:
2.2 Physical Effects Leading to Resonance Shift in Micro-ring Structures

\[ x^2\left(\frac{1}{n_x} + p_{11}s_1 + p_{12}s_2 + p_{13}s_3 + p_{14}s_4 + p_{15}s_5 + p_{16}s_6\right) \]
\[ + y^2\left(\frac{1}{n_y} + p_{21}s_1 + p_{22}s_2 + p_{23}s_3 + p_{24}s_4 + p_{25}s_5 + p_{26}s_6\right) \]
\[ + y^2\left(\frac{1}{n_z} + p_{31}s_1 + p_{32}s_2 + p_{33}s_3 + p_{34}s_4 + p_{35}s_5 + p_{36}s_6\right) \]
\[ + 2yz(p_{41}s_1 + p_{42}s_2 + p_{43}s_3 + p_{44}s_4 + p_{45}s_5 + p_{46}s_6) \]
\[ + 2zx(p_{51}s_1 + p_{52}s_2 + p_{53}s_3 + p_{54}s_4 + p_{55}s_5 + p_{56}s_6) \]
\[ + 2xy(p_{61}s_1 + p_{62}s_2 + p_{63}s_3 + p_{64}s_4 + p_{65}s_5 + p_{66}s_6) \]
\[ = 1 \quad (2.9) \]

Where

- \( n_x, n_y, n_z \) are the principal indices of refraction
- \( x, y, z \) are the crystal principal axes
- \( s_1, s_2, s_3 \) are stresses in principal directions, and \( s_4, s_5, s_6 \) are the shear strains in \( yz, xz, xy \) directions.

Once the independent components of the photo-elastic tensor have been identified, the change in refractive index due to applied strain field can be calculated from equation (2.9). The independent tensor components are often deduced from symmetry of the system. For instance, for silicon crystal, there are only three photo-elastic coefficients, \( p_{11}, p_{12}, p_{44} \) [6].

One can also calculate the change in refractive index in terms of stress instead of strain. The stress induced change in refractive index of a material for TE and TM polarized lights is given by the following relations [6]:

\[ n_{TE} = n_0 - C_1\sigma_x - C_2(\sigma_y + \sigma_z) \]
\[ n_{TM} = n_0 - C_1\sigma_y - C_2(\sigma_x + \sigma_z) \]

\[ (2.10) \]
2.2 Physical Effects Leading to Resonance Shift in Micro-ring Structures

Where $C_1, C_2$, are the stress optic constants of the material related to the photo-elastic coefficients through the following equations:[6]

\[
C_1 = \frac{n_o^3}{2E}(p_{11} - 2\nu p_{12}) \\
C_2 = \frac{n_o^3}{2E}[-\nu p_{11} + (1 - \nu)p_{12}]
\]

(2.11)

Where $E$ is the Young’s Modulus, and $\nu$ is the Poison’s ratio of the material.

These stress induced local index modifications can contribute to effective index change of the guided mode, and hence to sensing.

**Thermo-optic effect**

Ring resonators can be used for direct temperature sensing or for the sensing of other quantities such as chemical or gas concentrations through thermal effects which follow reactions with the sensing layer. In such cases the temperature rise in the vicinity of the ring resonator may lead to change in the optical properties, i.e. refractive indices of the core and cladding. This, consequently, will modify the effective index of the guided mode leading to shift in ring resonance.

The optical properties of matters are usually determined by coupling various types of oscillators to the electromagnetic radiation field. The amplitude of these oscillations depends on the frequency of the incident electromagnetic field, on the oscillator eigenfrequencies, on the different coupling strengths, $f$, between the electromagnetic field and the oscillators, and on their damping functions. Thermal energy being one such electromagnetic radiation which can be coupled to electrons and hole pairs (oscillators) in a material, its effect can be modeled with oscillator models. A simplified model can be introduced to analyze the refractive index of a material in its transparency region. In this case, only the real part of the dielectric function may be considered, obtaining

\[
\varepsilon = n^2 = 1 + E_p^2 \sum_k \frac{f_{cv}(k)}{E_{cv}(k) - E^2}
\]

(2.12)
2.2 Physical Effects Leading to Resonance Shift in Micro-ring Structures

Where

\[ E_p = \sqrt{4\pi N \hbar^2 e^2 / m} \]

is the electronic plasma energy, with \( N \) the number of oscillators per unit volume. \( E \) is the photon energy, \( E_{cv}(k) \) and \( f_{cv}(k) \) are, the transition energy and the inter-band oscillator strength between the valence and conduction band. \( K \) is the wave vector.

With single oscillator model approximation, the dielectric function can be written as:

\[ n^2 = 1 + \frac{E_p^2}{(E_g^2 - E^2)} \]  \hspace{1cm} (2.13)

Where \( E_g \) is the optical band gap average energy.

Using the appropriate relation for plasma energy with its temperature dependence, and considering the band gap temperature dependence, the thermo-optic coefficient of a material can be written as

\[ \frac{dn}{dT} = n^2 \left[ -3k_{ex} - \frac{2}{E_g} \frac{dE_g}{dT} \frac{1}{1 - (E/E_g)^2} \right] \]  \hspace{1cm} (2.14)

Where \( k_{ex} \) is the material thermal expansion coefficient.

While this being one theoretical model to explain thermo-optic effect, other models have also been reported to account for the same phenomena [8]. Making use of these explanations and noting the agreement with experimental findings, the thermo –optic coefficients of silicon and silicon oxide are approximately \( 1.8 \times 10^{-4} \), and \( 10^{-3} \) respectively.

2.2.2 Mechanical Modifications

In some sensing schemes, shift in ring resonance is caused by physical dimensional changes due to sensor – analyte interaction. For instance, in case of the hydrogen- Pd interaction, Pd is physically stretched. This effect can be felt by the region where the optical mode is guided through. Looking back to equation for shift in resonance,
2.3 Simulation Strategies and Tools Used

\[ \frac{\Delta \lambda}{\lambda} = \frac{\Delta L}{L} + \frac{\Delta n_{\text{eff}}}{n_{\text{eff}}} \]

the physical deformation may contribute positively to the resonance shift achieved from optical effect if they both exist together.

**2.3 Simulation Strategies and Tools Used**

The two simulation tools used to model the hydrogen sensor are Fimmwave and COMSOL Multiphysics. The optical modelling is done through FIMMWAVE while COMSOL is used to simulate the structural and thermal aspects of the sensor.

**FIMMWAVE- optical simulation tool**

FIMMWAVE is designed to model a wide variety of 2-dimensional and 3-dimensional waveguide structures using a rigorous fully vectorial formalism. The program is based on a fully vectorial waveguide solver based on the film mode matching method. The vector mode solver will locate almost any horizontal or vertical mode order of arbitrary or mixed polarization. [26]

In this thesis this tool is used to model ridge waveguide (RWG) structure on which the resonator is based and to estimate the mode effective index and associated propagation loss.

**COMSOL Multiphysics**

CMOSOL Multiphysics contains several modules suited to different application areas. In this thesis the structural Mechanics module and Heat transfer module are used. The underlying mathematical structure in COMSOL Multiphysics is a system of partial differential equations (PDEs). Finite element method (FEM) is used in solving the PDEs. COMSOL allows both 3D and 2D analysis of structures. [27]

**2.3.1 Overview of mechanical simulation through COMSOL**

To study the extent of strains and stresses felt by the overall structure due to mechanical changes in the sensing layer, the structural mechanics module is used. Basic concepts behind the mechanical aspects are outlined in this section.
2.3 Simulation Strategies and Tools Used

**Stress – strain relationship**

The symmetrical stress and strain in a material consist both principal and shear components, given below in tensor form.

\[
\sigma = \begin{bmatrix}
\sigma_x & \tau_{xy} & \tau_{xz} \\
\tau_{yx} & \sigma_y & \tau_{yz} \\
\tau_{zx} & \tau_{zy} & \sigma_z
\end{bmatrix}
\]

\[
\xi = \begin{bmatrix}
\varepsilon_x & \varepsilon_{xy} & \varepsilon_{xz} \\
\varepsilon_{yx} & \varepsilon_y & \varepsilon_{yz} \\
\varepsilon_{zx} & \varepsilon_{zy} & \varepsilon_z
\end{bmatrix}
\]

For instance, the stress tensor consists of three normal stresses \((\sigma_x, \sigma_y, \sigma_z)\) and six (or, if symmetry is used, three) shear stresses \((\tau_{xy}, \tau_{yz}, \tau_{xz})\).

The stress–strain relation for linear elastic materials is given by

\[
\sigma = D(\varepsilon - \varepsilon_e) + \sigma_0
\]  
(2.15)

Where

\(D\) is 6X6 elasticity matrix, and the stress and strain are both column vectors:

\[
\sigma = \begin{bmatrix}
\sigma_x \\
\sigma_y \\
\sigma_z \\
\sigma_{xy} \\
\sigma_{yz} \\
\sigma_{xz}
\end{bmatrix}, \quad \xi = \begin{bmatrix}
\varepsilon_x \\
\varepsilon_y \\
\varepsilon_z \\
\varepsilon_{xy} \\
\varepsilon_{yz} \\
\varepsilon_{xz}
\end{bmatrix}
\]

The Elasticity matrix \(D\) takes different forms depending on the material properties, isotropic, orthotropic and anisotropic materials. It depends on the young’s modulus \((E)\) or shear modulus \((G)\), and Poisson’s ratio \(v\).

Equation (15) takes into account thermal strain and initial conditions.
2.3 Simulation Strategies and Tools Used

**Thermal strain**
Thermal strain is related to the present temperature, \(T\), the stress-free reference temperature, \(T_{ref}\), and the thermal expansion vector, \(\alpha_{\text{vec}}\), as:

\[
\varepsilon_{\text{th}} = \begin{bmatrix} \varepsilon_x \\ \varepsilon_y \\ \varepsilon_z \\ \gamma_{xy} \\ \gamma_{yz} \\ \gamma_{xz} \end{bmatrix}_{th} = \alpha_{\text{vec}}(T - T_{ref})
\]

\(\alpha_{\text{vec}}\) takes different forms for different materials.

**Solving structural problems**
Equilibrium equations are finally used to solve structural problems involving stress-strain relations.

The equilibrium equation expressed in compact form for 3D stresses is

\[-\nabla \cdot \sigma = F\]  \hspace{1cm} (2.16)

Where, \(\sigma\) is stress tensor and \(F\) represents body forces.

**Constraints**
A constraint specifies the displacement of certain part of a structure. In structural analysis using COMSOL Multiphysics, it is possible to define constraints on all domain levels: points, edges, faces/boundaries, and subdomains.

Available boundary conditions (constraints) are:
- free
- fixed
- roller
- symmetry plane
2.3 Simulation Strategies and Tools Used

2.3.2 Treatment of Thermo-optic Effect using COMSOL Multiphysics and FIMMwave

Heat Distribution Analysis with COMSOL

In case where sensing involves intermediate/direct thermal effects in order to modify optical properties of the resonator structure, it would be necessary to model the heat flow through the structure. COMSOL Multiphysics comes with heat transfer module which allows such analysis.

Heat transfer is defined as the movement of energy due to a temperature difference. It can be characterized by conduction, convection or radiation. Both static and transient analyses are possible in COMSOL.

We limit ourselves to conduction analysis as the sensor basically involves this mechanism; heat transfer through diffusion due to temperature gradient.

To solve a particular heat conduction problem and find the temperature distribution inside a medium, it is necessary to specify the medium geometry and thermo-physical properties, the distribution of possible sources, the initial conditions and boundary conditions.

The Heat equation

The mathematical model for heat transfer by conduction is the heat equation:

\[ \rho C \frac{\partial T}{\partial t} - \nabla \cdot (\kappa \nabla T) = Q \]  \hspace{1cm} (2.17)

Where
- \( T \) is the temperature
- \( \rho \) is density
- \( C \) is heat capacity
- \( \kappa \) is thermal conductivity
- \( Q \) is heat source or heat sink
2.3 Simulation Strategies and Tools Used

The heat capacity $C$ describes the amount of heat energy required to initiate a unit temperature change with in a unit mass. Thermal conductivity $\kappa$ relates the heat flux vector $q$ and the temperature gradient through Fourier’s Law of heat conduction:

\[ q = -\kappa \nabla T \quad (2.18) \]

Heat source, $Q$, power per unit volume, describes heat generation within a domain.

**Boundary conditions**

The boundary conditions in conduction heat analysis provided in COMSOL are generally of two types:

In the first type, a temperature is imposed on a medium surface as:

\[ T = T_o \]

The second type of condition sets heat flux $q$ on a boundary as:

\[ -n.q = q_o \]

Where $n$ represents the surface normal, and heat flux $q$, as mentioned earlier is defined by Fourier’s Law of heat conduction as:

\[ q = -\kappa \nabla T \]

To summarize the available boundary conditions:

- Heat Flux \[ n.(\kappa \nabla T) = q_o \]
- Symmetry or insulation \[ n.(\kappa \nabla T) = 0 \]
- Prescribed temperature \[ T = T_o \]
- Zero temperature \[ T = 0 \]

**Projection of the Heat Analysis to Optical Modifications in Ring Resonator Structure**

Once the heat distribution in the structure has been analysed, the temperatures at the core and cladding can be used to estimate the respective refractive index changes in the materials. These changes can eventually be used in optical simulation tools, like
2.4 Summary

FIMMWAVE, in order to estimate the mode effective index change due to thermal effects. Finally, the corresponding resonance shifts in the ring can easily be predicted.

2.4 Summary

In this chapter, basic concepts underlying ring resonator based guided mode sensors are discussed. These sensors respond with shift in their resonance when either optical or mechanical changes are felt by the structures. It is indicated that optical modifications can arise due to bio/chemical interactions between the sensors and analytes, due to stress-optic effect in the presence of mechanical strain, or through thermo-optic effect. Dimensional change in ring resonator structures is another potential factor leading to resonance shift. In order to analyze these physical effects behind the sensing scheme, simulation tools are used. Fimmwave is used to study the change in mode index with the surrounding refractive index change. Mechanical / structural aspects and thermal distribution in the structures are analysed through COMSOL Multiphysics.
In this chapter the two hydrogen sensors specifically studied in this thesis and the related numerical simulations are discussed.

3.1 The Hydrogen Sensors
As pointed out in the introductory chapter, two ways of hydrogen sensing strategies are adopted in this thesis.

The basic structure, on which the sensors are built, is ring resonator on SOI platform. The SOI structure has silicon ring waveguide of thickness 220nm buffered from the substrate by a 2 μm thick lower SiO$_2$ cladding. Material used for top cladding is either SiO$_2$ or polymer bisbenzocyclobutene (BCB).

In order to couple the hydrogen gas environment to the ring resonator characteristics, two types of sensitive layers are used in this work. The first scheme makes use of palladium layer of nanometer scale on top of the ring structure. In the second scheme hydrogen sensing is achieved by WO$_3$ catalytic coating on the structure.

3.1.1 Hydrogen Sensing with Pd Coating
When Pd is exposed to hydrogen, the hydrogen molecules are adsorbed at Pd surface and dissociate into hydrogen atoms. In an inert atmosphere, such as nitrogen, hydrogen atoms can diffuse through the bulk material and palladium hydride (Pd-H) system is formed. This system exhibits different physical properties from the hydrogen free Pd. At higher
3.1 The Hydrogen Sensors

Hydrogen concentrations the hydride is observed to show hysteresis in its physical properties. The hysteresis free region is often called alpha phase where as the other region is called beta phase of the system. The concentration at which this phase transition takes place depends on temperature and thickness of the Pd film. Fast transitions are observed at low temperatures and higher thicknesses [9].

On adsorption of hydrogen the lattice parameter increases and Pd expands in volume. This further reduces volume density of free electrons leading to decrease in both real and imaginary parts of the refractive index [10]. Hence, both mechanical and optical changes take place. The sensor under consideration potentially makes use of both effects.

The hydrogen expansion coefficient of Pd is approximately 0.0655 per atomic hydrogen to Pd ratio [11]. For ratios below 0.03, the Pd –H system is purely in alpha phase that hysteresis free responses can be achieved [11]. Simulations on mechanical changes are run for low concentration change of 0 to 0.01 hydrogen to palladium atomic ratio. The strain developed in sensing layer can potentially modify the effective index of the ring waveguide mode through elasto-optic effect. In addition, the strain can physically stretch the ring.

The optical change with hydrogen concentration is dictated by the change in complex dielectric permittivity of Pd, expressed by simple empirical relation [10]:

\[ \varepsilon_{Pd-H} = h \times \varepsilon_{Pd} \]  

(3.1)

Where \( h \) is a nonlinear function of hydrogen concentration taking values less than 1. The value of \( h \) decreases with hydrogen concentration, \( h=1 \) corresponding to zero hydrogen concentration. This optical change in palladium can potentially lead to effective index change of the resonator modes through evanescent field interaction.

3.1.2 Sensing With Tungsten Oxide (WO₃) Catalytic Layer

Catalytic combustion is a flameless combustion process that uses solid catalyst to boost reaction by lowering the activation energy of chemical reaction. This mechanism allows reactions to take place at lower gas concentrations and lower temperatures without creating fires.
3.2 Simulation Results

The WO₃ catalytic layer converts hydrogen and oxygen into water with the release of heat. This heat is used to induce refractive index change in the resonator structure through thermo-optic effect. The resulting shift in the ring resonance is, in turn, used to sense hydrogen in the surrounding air.

The chemical reactions involved are [14]:

\[ \text{WO}_3 + H_2 \leftrightarrow \text{WO}_2 + H_2O \]
\[ \text{WO}_2 + \frac{1}{2}O_2 \leftrightarrow \text{WO}_3 \]

However, as it is common with exothermic reactions, a little activation or heating is required to initiate the chemical reaction. The rate of reaction, and hence, heat production is dependent on the catalyst temperature and hydrogen concentration at a given air flow. At high enough temperatures the reaction becomes diffusion limited and is controlled by the hydrogen concentration gradient between the surrounding air and the catalyst surface [12].

3.2 Simulation Results

3.2.1 Simulations Results for Pd Based Sensor

In order to make the best use of the mechanical and optical changes for the sensing goal, some structural considerations, particularly with thickness of the top cladding and the Pd layer, are necessary.

**Optimum Pd Thickness**

For optimum response of the Pd layer to surrounding hydrogen, and to achieve consistent optical and mechanical modifications with increasing as well as decreasing concentration of hydrogen, optimum thickness of the metal has to be determined. Furthermore, it is necessary to analyze the optical losses associated with Pd thickness and minimize them.

The simulation results show (Fig. 3.1) that propagation losses are low for higher Pd thicknesses at a given cladding thickness. The likely reason for this result can be the dominance in the reflective nature of the metal at thicknesses higher than the penetration depth. Though reduced loss is an important issue, adequate penetration of the evanescent
3.2 Simulation Results

field to the layer surface for better sensitivity has to also be considered. Another fact which has been mentioned in the beginning of this chapter is the hysteresis observed in physical and optical properties of Pd. Lower Pd thicknesses (often in the range 10nm to 100nm) are observed to give good response with hydrogen concentrations of interest, i.e., below lower explosive limit [9]. Taking these trade-offs into consideration, 10nm to 20nm thick sensing layers are chosen from the optical point of view. The choice of Pd thickness further depends on the sensing effect we would like to have from mechanical contributions. These issues are discussed in the mechanical simulation section.

Optimum Cladding Thickness

As indicated in the previous chapter, resonator sensitivity is enhanced with the quality factor as small change in effective index can lead to visible shift in resonance. The quality factor depends largely on the propagation loss of the waveguide among other parameters.

Palladium, as a metal, has high optical absorption coefficient of around 677448/cm (with imaginary part of the refractive index corresponding to -8.36) at wavelength of 1550nm. Due to this reason, the Pd layer has to be kept at reasonable distance from the core to minimize optical loss, and so to enhance the quality factor. However, situating it far from the core means that the interaction of the WGM evanescent field with the sensing layer, and hence, the sensitivity is reduced. Accordingly, optimum cladding thickness has to be chosen.

Figure 3.1 variation in optical loss of the sensor structure with palladium layer thickness for 700nm thick silica cladding a) 2d (RWG) simulation with FIMMwave, b) 1d (slab) simulation with CAMFR and FIMMwave

Optimum Cladding Thickness

As indicated in the previous chapter, resonator sensitivity is enhanced with the quality factor as small change in effective index can lead to visible shift in resonance. The quality factor depends largely on the propagation loss of the waveguide among other parameters.

Palladium, as a metal, has high optical absorption coefficient of around 677448/cm (with imaginary part of the refractive index corresponding to -8.36) at wavelength of 1550nm. Due to this reason, the Pd layer has to be kept at reasonable distance from the core to minimize optical loss, and so to enhance the quality factor. However, situating it far from the core means that the interaction of the WGM evanescent field with the sensing layer, and hence, the sensitivity is reduced. Accordingly, optimum cladding thickness has to be chosen.
3.2 Simulation Results

Simulations are done in FIMMWAVE for different SiO₂, and BCB buffer thicknesses to determine optimum regions. The BCB is chosen as an alternative cladding material for practical reason that it can easily be spin-coated on air-clad ring resonator and has a refractive index (1.54) close to SiO₂. Figure 3.2 shows the simulated Q values for different SiO₂ buffers for 10nm thick Pd layer.

![Figure 3.2: Simulated Q-factor for different silica buffer thicknesses, with 10nm thick Pd layer](image)

The evanescent field interaction with the sensing layer for different buffer thicknesses is then simulated by considering the fact that the complex dielectric permittivity of Pd changes with hydrogen concentration. The relation given by equation (3.1) along with typical \( h \) values for low enough hydrogen concentrations, 0.95 to 0.7, found from literature is used to calculate the permittivity at the corresponding hydrogen concentrations [10]. A change of \( h \) value from 1 to 0.95 roughly corresponds to 1% hydrogen to Pd atomic ratio [10]. Simulations in FIMMWAVE are then run to calculate the effective index at given hydrogen concentration. The average change in effective index calculated for \( h \) varied

![Figure3.3: Sensor sensitivity per 1% hydrogen in Pd for different buffer thicknesses](image)
3.2 Simulation Results

from 0.95 to 0.7 in step of 0.05 increases with decreasing buffer thickness. Effective index changes of $10^{-6}$ and $6 \times 10^{-5}$ are estimated per 1% hydrogen atom concentration in palladium for 700nm and 300nm thick buffers respectively. The corresponding sensitivity, i.e., shift in resonance, is summarized in figure 3.3. These simulations are run for 10nm thick palladium layer.

<table>
<thead>
<tr>
<th>BCB thickness(nm)</th>
<th>h</th>
<th>$n_{\text{eff}}$</th>
<th>Average change in $n_{\text{eff}}$ ( per 1% H in Pd)</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>1</td>
<td>2.37921049</td>
<td>6X10^{-5}</td>
</tr>
<tr>
<td></td>
<td>0.95</td>
<td>2.37924737</td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.9</td>
<td>2.37930379</td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.85</td>
<td>2.37935988</td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.8</td>
<td>2.37941663</td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.75</td>
<td>2.37947305</td>
<td></td>
</tr>
</tbody>
</table>

Table 3.1: Effective index change with hydrogen concentration in Pd layer for 300nm thick BCB buffer and 10nm thick Pd coating.

Results comparable to those calculated for SiO$_2$, with respect to quality factor and sensitivity, are found for BCB buffers as well. For instance, for 300nm thick BCB and SiO$_2$ buffers the simulated Q is around 1400 and 1524 respectively. Fabrication losses are expected to reduce these values. From these simulated values, the resonance BW for 300nm thick buffers corresponds approximately to 1nm.

With BCB cladding, which has refractive index a little higher than that of SiO$_2$, a small enhancement in sensitivity is observed; for instance, for BCB thickness of 300nm, the maximum change in effective index is around $6 \times 10^{-5}$ and the corresponding shift in resonance is around 40pm for 1% change in hydrogen concentration, which is not far from 37pm shift for similar thickness of SiO$_2$ cladding. For both claddings, the expected average shift in wavelength is just below 40pm.

From the simulated values for the sensor sensitivity, working with 300nm to 400nm thick buffers appears to be essential to have detectable response from the sensor. However, the wide resonance bandwidth in the range between 0.5nm to 1nm for these thicknesses is likely to pose problem to detect the expected small shifts. The quality factor of around 0.5nm for 400nm thick cladding looks better, but the sensitivity falls almost by half, approximately to 20pm per 1% hydrogen concentration in palladium.
3.2 Simulation Results

Mechanical simulation

Noting the elastic deformation experienced by Pd in the presence of hydrogen, the corresponding stress and strain in the sensor structure is studied using COMSOL Multiphysics. Later on, the simulated stress results are used to estimate the corresponding elasto-optic effect. The accompanying strain values are, on the other hand, used to estimate the change in ring diameter. The structural problem is treated both in two and three dimensions.

In two dimensional plane stress/strain analysis, the stress, $\sigma_z$, along the propagation ($Z$) direction is assumed not to change with position. However, it changes with stresses in X and Y directions [13]. In ordinary plane strain approximation, equation (3.2) represents stress changes in respective directions. The structure is not allowed to relax along Z direction, and hence, the stain component $\xi_z$ is zero.

$$
\sigma_x = \frac{E}{(1+\nu)(1-2\nu)}[(1-\nu)\ddot{\xi}_x + \nu\ddot{\xi}_y] - \frac{\alpha E \Delta T}{1-2\nu}
$$

$$
\sigma_y = \frac{E}{(1+\nu)(1-2\nu)}[\nu\ddot{\xi}_x + (1-\nu)\ddot{\xi}_y] - \frac{\alpha E \Delta T}{1-2\nu}
$$

$$
\sigma_z = \nu (\sigma_x + \sigma_y) - \alpha E \Delta T
$$

Where $E, \nu, \alpha$ are the Young’s modulus, Poisson’s ratio and thermal expansion coefficient respectively.

This 2d approximation, however, is valid for waveguides which are invariant along the propagation direction, and sufficiently long compared to the cross section. With our ring structure, which bends in the propagation direction, this approximation doesn’t seem to work well. It is, however, used to compare the results from 3d simulations.

The 3d stress/strain simulation, though ideal, requires the structures to be as small as possible to reduce computational complexity and to be able to handle the problem with available computer power. Accordingly, structures which are about 5-10% smaller than the real structure are used. The results are then compared with those from larger 2d structures.
3.2 Simulation Results

In real structures, the edges of the rest of the structure are far away from the waveguide that edge effects are not felt. Whereas in numerical models, where the structures are made smaller, care has to be taken on choosing the right window of calculation. In both the 3d and 2d simulations efforts have been made to minimize edge effects by keeping the ring away from the edges.

The boundary conditions used in the structural simulation are, the bottom of the buried oxide layer is fixed in all directions and it approximates the substrate. This is taking into account the relatively very large thickness of the substrate, around 700µm, and the thin layer of palladium used to induce stress in the structure. Interior boundaries are symmetry planes, and the rest of the surfaces are free to relax.

Figure 3.4 principal stress distribution for 6µmX4µm (width X length) silica clad structure with ring radius of 2.5µm. (a) Stress distribution in x direction, (b) Stress distribution in z direction.

Figure 3.5 principal stress distribution for 8µmX8µm (width X length) silica clad structure with ring radius of 2.5µm: (a) stress along y direction, (b) stress in z direction.
3.2 Simulation Results

The reduced edge effects with increasing structure size can be observed comparing the stress distributions plotted in figure 3.4 and 3.5. Less stresses are felt by the ring for larger structure. The possible large dimension (8µm X 8µm) which can be handled with the computational limit is used. The calculated stresses and strains are summarized in table 3.2. Though these values can’t accurately represent the real structure, along with 2d simulation results to be discussed later, can be used to give useful estimations and can lead to valuable conclusions.

![Image](image.png)

Figure 3.6: 2d Stress distribution simulation, 100µm wide structure with 60 µm wide Pd, the tiny dot in the middle represents the cross section of the silicon waveguide.

In 2d plane strain simulation the magnitude of stress around the waveguide region decreases with increasing structure size. Increasing the width of the top palladium layer further reduces this stress level. On the other hand, the 3d simulations show that as this positive or compressive stress felt around the core decreases with increasing structure size, the physical stretching near the ring edges increases. More importantly, stress and strain in the structure are increased with palladium thickness. Most of the simulation results discussed here use 500nm thick Pd layer on top silica clad for the reason that lower thicknesses resulted in insignificant structural changes. Results for BCB clad are to be compared later.

From the results of the approximate 3d and 2d simulations, summarized in table 3.2, it is observed that the 3d simulation appears to overestimate local stresses due to its implementation on smaller structure. Still these estimations are not very far from those
obtained from 2d simulations. The stresses calculated from both simulations are below 10 MPa.

<table>
<thead>
<tr>
<th>Simulation</th>
<th>Structure dimension (width X Length)</th>
<th>$\sigma_x$ (MPa)</th>
<th>$\sigma_y$ (MPa)</th>
<th>$\sigma_z$ (MPa)</th>
<th>Approx. Change in ring diameter (pm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3D</td>
<td>6µm X 4µm</td>
<td>22</td>
<td>12</td>
<td>25</td>
<td>300</td>
</tr>
<tr>
<td></td>
<td>8µm X 8µm</td>
<td>8</td>
<td>10</td>
<td>8</td>
<td>500</td>
</tr>
<tr>
<td>2D</td>
<td>Structure Width (µm)</td>
<td>$\sigma_x$ (MPa)</td>
<td>$\sigma_y$ (MPa)</td>
<td>$\sigma_z$ (MPa)</td>
<td>Approx. Change in ring diameter (pm)</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>5</td>
<td>2</td>
<td>7</td>
<td>Assumed not to relax</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>nearly zero</td>
<td>nearly zero</td>
<td>nearly zero</td>
<td>Assumed not to relax</td>
</tr>
</tbody>
</table>

Table 3.2 Summary of 2d and 3d simulated principal stress distribution and ring diameter change

Figure 3.8 Stress distribution along x direction for BCB clad 8µm X8µm structure,

In comparison with SiO$_2$ clad structures, the stress levels around the core drop to below 5Mpa for 400nm thick BCB buffer with Pd layer of 500nm. The corresponding physical expansion in the core further falls to around 100pm. This can be attributed to the fact that the very low Young’s modulus of BCB, 2.9 GPa, makes it to expand to itself and leave less stress for the surrounding structure.
3.2 Simulation Results

Using the stress distribution listed in table 3.2 the maximum stress induced refractive index change in the core is calculated with equations 2.10 and 2.11 given in the previous chapter. The corresponding effective index change for 1% hydrogen concentration is then estimated using FIMMWAVE. Comparing the resonance shift with and without stress, the net shift due to the stress effect is estimated to be about 5pm.

With respect to sensing contribution from physical stretching of the ring, as shown in table 3.2, the physical change in ring diameter is around 300pm. This corresponds to additional wavelength shift of 40pm. Consequently, considering all the optical (20pm shift for 400nm thick clad) and mechanical sensing effects from the palladium layer, a total of about 65pm resonance shift is expected for small change in hydrogen concentration (1% hydrogen to palladium atomic ratio).

It is worth noting that these results are obtained for 500nm thick Pd layer, almost no stress and physical expansions around the core are observed for thicknesses below 100nm. However, higher Pd thickness may not be practical from hysteresis point of view mentioned earlier in this chapter, and fabrication limitations to be discussed in the next chapter.

Finally, though considering the substrate as fixed is reasonable, treating the bottom of the buried oxide layer as symmetry plane rather than fixed would make the simulation more logical. An alternate 3d simulation with slight modification on boundary conditions is done and comparable results from earlier simulations are found. In this simulation, instead of approximating the substrate by fixing the bottom of the buried oxide layer, a 2µm thick substrate is used. This thickness is, in fact, ridiculously small compared to the actual substrate. However, the Young’s modulus of the substrate in the simulation is artificially set to a very large value, 10^6 times the actual, to account for the thickness and the expected negligible wafer bending. All the outer boundaries in this simulation are then set to be free. The resulting estimated stresses still lie in ten’s of MPa range.
3.2 Simulation Results

3.2.2 Simulation Results for WO3 Coated Sensor

In this sensing strategy, hydrogen combustion in the WO3 layer serves as a heat source to the SOI structure underneath it. Some of the heat generated on the top surface of the catalyst will be absorbed by the by-product water, and some will be absorbed in the catalyst itself depending on its heat conductivity and thickness. The remaining heat reaches the SOI sample surface. To verify the expected wavelength shift of the SOI ring, for a given change in temperature, heat transfer simulation is done in COMSOL. The SOI structure considered has 720nm thick top SiO2 cladding with the catalytic layer coated on it.

The boundary conditions taken into account for this simulation are:

\[ T = T_{\text{catalyst}} \] on top boundary of the SOI structure
\[ T = T_{\text{sample}} \] on the bottom boundary of the structure
\[ n \cdot k \nabla T = 0 \] (Thermal insulation) on lateral surfaces
\[ n \cdot (k_1 \nabla T_1 - k_2 \nabla T_2) = 0 \] (Continuous heat flow) on interior boundaries

Where \( n \) represents the surface normal, and \( \kappa \) is the thermal conductivity.

![Temperature distribution](image)

Figure 3.8 temperature distributions across 720nm thick silica clad SOI waveguide for 1°C temperature change on top of the sample

Taking into account the thermo–optic coefficients 1.8 X10^{-4} and 10^{-5} for Si core and SiO2 clad respectively, the refractive index changes in the two materials are calculated using the
3.3 Summary

The temperature distribution is found from heat transfer simulation in COMSOL. The calculated refractive index changes are $1.12 \times 10^{-4}$ and $7.5 \times 10^{-6}$ for Si and SiO$_2$ respectively. Later on FIMMAVE is used to calculate the corresponding effective index change of the guided mode.

The resulting effective index change is $1.2 \times 10^{-4}$ corresponding to resonance shift of about 78 pm per one degree temperature change on the sample surface.

Similar simulations have done to study the sensor sensitivity for lower cladding thicknesses. 10 to 15 pm improvements in resonance shift per 1°C change in temperature are predicted for 600 nm and 540 thick SiO$_2$ claddings.

3.3 Summary

Hydrogen sensing in this thesis is conceived to be implemented through design and fabrication of two separate sensors on SOI ring resonators. The first implementation links the change in optical and mechanical properties of hydrogen sensitive Pd coating to the ring resonator performance. The three physical phenomena involved in this scheme are evanescent field interaction, stress optic effect and physical expansion.

Simulations in FIMMAVE have been done to determine optimum Pd layer and buffer thicknesses. 10 to 20 nm Pd thicknesses are chosen based on the promised hysteresis free operation and fabrication issues related with thicker layers.

BCB or SiO$_2$ buffers of around 400 nm thickness are chosen taken into account the trade-off between evanescent field interaction and resonance bandwidth required for feasible sensor operation. About 20 pm resonance shift per 1% hydrogen to Pd atomic ratio is predicted from evanescent field interaction at around 400 nm thick buffers.

The mechanical simulations for the Pd sensor have been a bit challenge due to the computational limits to simulate larger 3d structures which would fairly approximate the real ones. Nevertheless, fairly logical approximate simulations both in 2d and 3d have been
done. These simulations have shown that the strain/stress from the Pd layer are well felt by the waveguide region only for thicker Pd layers. 5pm resonance shift due to stress-optic effect and 40pm shift due to physical stretching of the ring are predicted for 500nm thick Pd coating on 400nm thick SiO2 cladding. These stress/strain effects are observed to be lower for BCB cladding due to its very low Young’s modulus which makes it to relax without strongly affecting the surrounding structure.

Taking into account the difficulty to deposit thicker Pd layers (to be discussed in the next chapter), all the sensing effect is expected to come from evanescent field interaction for this sensor implementation.

In the second sensing scheme, the heat generated from combustion of hydrogen in oxygen with the aid of WO3 catalytic coating modifies the ring resonance through thermo-optic effect.

To estimate the expected resonance shift for a given temperature rise on the surface of the SOI sample, heat transfer simulation in COMSOL has been done. The temperature distribution from this simulation is used to calculate refractive index changes in SiO2 cladding and the Si core. FIMMWWAVE is then used to calculate the corresponding effective index change. The predicted resonance shift from these simulations is around 78pm /°C for the ring with 720nm thick SiO2 clad.
In this chapter, the fabrication of the two sensors studied in this thesis and the measurements results are presented.

4.1 Sensor Fabrication

The SOI ring resonator structures used for hydrogen sensor implementation are fabricated using CMOS tools at IMEC- Leuven. The structures are patterned on SOI wafer using 248nm deep UV optical lithography. This lithography technique gives better control over feature sizes of the structures. The structures are then formed by etching 220nm thick top Silicon layer on the wafer. The silicon layer is buffered from the substrate by 2µm thick bottom SiO2 cladding. The fabricated ring resonators of around 5µm radius used in this thesis are based on previous works in the photonics research group. The SOI rings are designed to operate in telecom wavelength range.

4.1.1 Depositing the Pd Hydrogen -Sensitive Layer

Post sensor fabrication steps on SOI samples are carried out at the University clean room. The two commonly used top claddings for the ring resonator structures are SiO2 and air. Air –clad structures are used in this thesis for Pd- based sensor fabrication because of the flexibility they provide to lay out different thicknesses of cladding material of choice. A polymer material, namely, bisbenzocyclobutene (BCB), known for its good planarization property [1], is used to achieve various cladding thicknesses for Pd-based sensor implementation. It has been demonstrated in previous work that thinner BCB layers can be achieved by mixing the so called B staged BCB with right proportion of mesitylene[2].
300nm to 600nm BCB layers are achieved spin-coating right mixtures of BCB and mesitylene at appropriate speeds, and following right curing cycles. The cladding layer is used to buffer the metallic palladium layer from the guiding region so as to reduce absorption losses without largely compromising the evanescent field interaction needed for the sensing effect.

The fabricated SOI structure has two ring resonators in series (spaced by 40 to 90µm). While Pd is deposited on one of the rings, the other is left free as reference for temperature effects.

![Diagram of Pd deposition process in the clean room](image)

Masks of slightly varied sizes and geometries are designed to cover one of the rings partly based on simulation results. The mask is patterned on positive photoresist (AZ5214) spin coated on SOI samples using 365nm optical lithography of appropriate dosage. Post exposure baking and flood exposure are later performed as image reversal steps before development to make the sample ready for Pd deposition. Oxygen plasma etching of only a few minutes is carried out on the samples to improve Pd adhesion to the BCB. Pd layer of a required thickness is deposited on the samples using electron gun (e-gun) evaporating
4.1 Sensor Fabrication

machine. Finally, lift-off process is carried out leaving the Pd on top of one of the rings. The processing steps for Pd deposition are pictorially summarized in fig. 4.1.

Deposition of very thin films of Pd (10-25nm) has been successfully achieved. However, the attempt to deposit thicker layers around 500nm has failed; the Pd layer is peeled-off during the final lift-off process due to the poor adhesion of Pd on BCB and the stress developed at the interfaces.

![Figure 4.2 a) Opening formed on one of the rings after lithography b) 10nm thick Palladium deposited on one of the rings](image)

Figure 4.2 a) Opening formed on one of the rings after lithography b) 10nm thick Palladium deposited on one of the rings
4.2 Measurement Results

4.1.2 Coating WO$_3$ sensitive layer on SOI rings

This part of the hydrogen sensor fabrication is done in collaboration with the Sensors research group Materia Nova, Cellule Capteurs in Mons, Belgium. While the SOI ring resonators are designed at Photonics research group at Gent university, and fabricated at IMEC- Leuven, the preparation of the WO$_3$ sensitive layer and coating are done in Mons.

The catalytic layer of a few microns thickness is coated on top of 720nm thick silica clad ring resonators using dip–coating technique.

4.2 Measurement Results

Measurement Setup

The sample to be characterized is seated inside a small gas chamber which has a circular opening on top of side of it. The chamber is sealed with a 2mm thick glass window during measurement. The gas chamber has two gas inlet valves connected to the input gases through flow controllers. A gas outlet valve on the other side of the chamber ensures continuous one directional gas flow.
4.2 Measurement Results

Input light from Tunics Plus tuneable laser is vertically coupled to the SOI rings through the glass window. The laser has tuning resolution of less than 10pm and it is tuneable in 1500-1640nm range. Coupling from optical fibre to the waveguide structures is achieved with grating couplers. The output light from the rings (through grating couplers) is collected by computer controlled Xenics (Xeva-511) infrared camera.

4.2.1 Measurement Results from the Pd Based sensor

Measurements with and without hydrogen in nitrogen environment have been made on several Pd coated samples. The palladium coatings are 10nm to 20nm thick, deposited on 300 to 500 nm thick BCB buffers.
4.2 Measurement Results

Figure 4.4 shows the profile and bandwidth of the resonance measured for 20nm coating deposited on approximately 300nm thick BCB buffer. The resonance bandwidth is about 0.6nm and rather distorted profile is observed due to the proximity of the highly lossy Pd layer to the waveguides.

![Resonance profile and bandwidth](image)

Figure 4.4 Resonance measured for a ring with approximately 300nm BCB buffer and 20nm Pd coating

Due to the fact that deposition of thicker Pd layers is not possible for the fabrication limitation mentioned earlier, the sensing effects from mechanical strain and stress can be neglected as predicted by the simulation results discussed in the previous chapter. Accordingly, all the sensing with 10nm to 20nm thick Pd coatings is expected to come from effective index change with hydrogen concentration. Taking into account the corresponding very small resonance shifts, and the observed distorted nature of resonance for lower BCB buffers, slightly higher buffers are preferred.

![Resonance shift](image)

Figure 4.5 Shift in resonance measured for 2.5% hydrogen in nitrogen on a) reference ring, b) Pd coated ring. The BCB and Pd are around 400nm and 20nm thick.
4.2 Measurement Results

Measurement results for samples with approximately 400nm thick buffers and 20nm Pd coatings appear to have better resonance bandwidth and smoother profile. Measured resonance shifts for the reference (without coating) and Pd coated rings at 2.5% hydrogen concentration in nitrogen are shown in Fig 4.5 a) and b) respectively. A slight blue shift of about 40pm, and approximately 10pm red shift is measured on the reference and Pd coated ring respectively. Such oppositely directed shift of the two resonances is to be attributed to thermal effects to be discussed later on.

![Figure 4.5](image.png)

Figure 4.6 resonance shifts measured from another sample at 2% hydrogen in nitrogen on a) reference ring b) coated ring. For around 400nm buffer and 20nm Pd coating

Figure 4.6 shows measurement results on another sample with about 400nm thick BCB buffer and 20nm Pd coating. Blue shifts are measured on both rings. However, a slightly higher shift is observed for the reference ring. The measured shifts are around 100pm and 60pm on the reference and coated ring respectively.

At this point, referring back to the simulation results would be necessary. Noting the decrease in dielectric permittivity, hence the refractive index, of Pd with increasing hydrogen, it can intuitively be expected that the guided mode would be more confined to the ring owing to the lower refractive index surrounding. This is further justified by the simulation results mentioned in the previous chapter [table 3.1]; which shows that the effective index increases with hydrogen concentration. Accordingly, the hydrogen sensing should be observed through red shift rather than blue shift in resonance.

However, remembering the high temperature sensitivity of SOI structures, the blue shifts can be ascribed to thermal effects. When the gases, both the hydrogen and nitrogen, come
4.2 Measurement Results

out of the bottles, they become less pressurized and their temperature would also drop leading to cooling of the sample. This, in turn, would lead to blue shift through thermo-optic effect.

Figure 4.7 resonance shifts measured at 0.5 % and 3% hydrogen concentrations in nitrogen a) and c) on reference ring, b) and d) on coated ring

The useful fact which will help us to verify the hydrogen sensing by one of the rings is the difference in magnitude of shift observed for the two rings. With the assumption that the two rings would respond equally to thermal effects and uniform gas flow over both rings, the net red shift of the coated ring can be attributed to hydrogen effect. The later assumption is less arguably valid considering the very small dimension and spacing of the rings. However, the first assumption is valid in case where Pd coating is highly conductive to heat. Pd as a metal is good heat conductor (with thermal conductivity of 71.8 W/ (m. k)), moreover, the coating is very thin. On this ground the assumption can be valid. The heat transfer simulation in COMSOL further consolidates this assumption; almost no heat is
4.2 Measurement Results

absorbed in the 20nm thick Pd layer. The slight tendency of red shift depicted in fig. 4.5 (b) also supports this fact.

Consequently, a net resonance shift of about 40pm is observed for 2% hydrogen concentration in nitrogen. This result appears to coincide with the predicted average shift of about 20pm per 1% hydrogen in palladium for 400nm thick BCB cladding, without forgetting that there may not be direct relation with the two concentration measures. It is however worth noting that the measurement with such small resolution would be largely susceptible to errors. Thermal instability and systematic errors are very likely to give wrong figures.

Besides the very small expected as well as experimentally observed resonance shifts from the Palladium sensor, other accompanying weak features are observed. Once the sensor has responded to a first exposure to hydrogen, very insignificant response is observed for the following exposures of increasing or decreasing concentrations. Figure 4.5 and 4.6 demonstrate two instances of this observation; negligible shift in resonance is measured three to four minutes after the removal of hydrogen. Due to this limitation, responses for different concentrations of hydrogen are compared from measurements taken after long time intervals. One such comparison is shown in figure 4.7. While nearly similar blue shifts of 80pm are measured on both rings for 0.5% hydrogen , blue shift of around 70pm and 10pm is measured on the reference and coated ring respectively for 3% hydrogen concentration. This corresponds to approximately 60pm red shift on the Pd coated ring. This observation further justifies sensitivity to hydrogen presence.

It can also be noticed from figures 4.5 and 4.6 that less significant shift upon removal of hydrogen are observed on both rings. More stable newly established thermal equilibrium is the likely reason with regard to the thermal response of the rings. More stable thermal equilibrium is to mean, temperature gradient between the sample with and with hydrogen is lower during hydrogen removal than during the first contact with hydrogen. Regarding the response of the coated ring for hydrogen removal, it can not show red shift because hydrogen is removed and the new thermal equilibrium is more stable, blue shift is not expected either since the cooling effect from hydrogen is removed.
4.2 Measurement Results

The poor response for increasing concentration after the first exposure can be due to the hysteresis effect often observed in palladium at higher hydrogen concentrations. Though operating at lower concentrations may solve this problem, the very low sensitivity of the sensor doesn’t allow observable effects at such low concentrations.

4.2.2 Measurement Results from WO₃ Coated Ring Resonator Sensor

In this section the measurement results from thermo-optic based sensing and the accompanying observations are discussed.

The graphs in figure 4.8 show measured shifts on resonance with increasing hydrogen concentration in air for a silica clad ring coated with WO₃ catalytic material. The silica buffer between the Si ring and the coating is about 720nm thick. The coating is a few hundreds of microns thick.

![Graphs showing resonance shifts](image)

Figure 4.8 a) and b) Measured resonance shifts for increasing hydrogen concentration. Measurement taken on a hot day (without external heating) with constant air flow of 0.5L/min into the gas chamber.

The measurement results shown in figure 4.8 are taken without using any external energy to enhance the exothermic reaction between hydrogen and oxygen. Issues related to the sensor response dependence on surrounding environment are discussed later in this section. Around 100pm shift towards longer wavelengths is measured for 1% hydrogen in dry air. It can be observed from fig. 4.8 (b) that the sensor response becomes less strong with increasing hydrogen concentration at 0.5L/min constant air flow. This effect is found out to
4.2 Measurement Results

be due to lower gas flow rates; the hydrogen concentration is varied keeping air flow at 0.5L/min in the chamber.

When air flow is increased to 1L/min, noticeable improvement is observed on the sensor response. Fig. 4.9 compares the responses for 2% hydrogen concentration at air flows of 0.5L/min and 1L/min; more than a hundred percent improvement, from 130pm to 270pm, is measured.

Unfortunately, measurements made on later days without using external heating were found to give almost no response. Change in the surrounding temperature and humidity are the likely reasons for deterioration in sensor response. While exothermic reactions are enhanced with temperature, water content in humid air has the tendency to suppress them. In an attempt to solve this issue, constant air flow is let through the gas chamber and the area around the sample is heated with the light from halogen lamp. Measurements are then made after waiting until roughly steady sample temperature is achieved. Steady heating is verified by repeatedly measuring the shift in ring resonance. Heating with the lamp obviously is not a proper option, but with the current challenge to connect temperature control inside the chamber, it is a useful option to make use of. The steady state resonance shift achieved with such heating is shown in figure 4.10. Considering around 80pm shift per 1 °C rise in temperature of SOI ring predicted from simulation results, the measured 2nm shift corresponds to about 20 °C rise in sample temperature.
4.2 Measurement Results

Figure 4.10 Resonance shift at roughly steady heating from halogen lamp

Figure 4.11 measured resonance shift at different hydrogen concentrations in air, sample temperature kept at about 40°C and 1.3L/min air flow.

After steady sample temperature of about 40 °C in constant flow of air is achieved, measurements for decreasing hydrogen concentrations are taken and the results shown in figure 4.11 are obtained. About 340pm shift is achieved for 1.4% hydrogen concentration. More noticeably, a shift of 1.2nm is achieved for 3% hydrogen concentration. The catalytic layer provides more than 10 °C temperature rise on the sample surface at 3% hydrogen concentration in air. Tailoring the air flow rate and elevating the operating temperature to a reasonable level, further enhancements in the sensor performance can be achieved.

Further interesting feature observed about the WO₃ coated sensor is its fast response for hydrogen concentration gradient. All the measurement results discussed are observed in less than a minute after change in hydrogen concentration in either direction.
4.3 Summary

The basic SOI ring resonators used for the two sensor implementations are fabricated at IMEC. Post fabrication processes needed for Pd deposition and the deposition itself are carried out at the University clean room. BCB is used as buffer material for Pd based sensor owing to the involved fabrication flexibility. Because of the stress developed at interfaces and the weak adhesion of Pd on BCB, deposition of thicker layers hasn’t been possible. The fabricated Pd based hydrogen sensors, thus, use 10 to 25nm thick Pd coatings.

The WO₃ hydrogen sensitive layer is coated on ring resonators using dip-coating technique in collaboration with Materia Nova, Cellule Capteurs sensors group in Mons.

Measurements on Pd based sensors have shown that hydrogen sensing is influenced by thermal effects due to the high temperature sensitivity of the SOI rings. Cold gases reaching the sample resulted in blue shifts in resonance. However, since the sensor employs a reference ring in addition to the Pd-coated ring, the net red shift due to hydrogen can be measured from the difference in blue shifts on the two rings. Lower blue shifts are observed on the coated ring indicating sensitivity to hydrogen. The average measured resonance shift for Pd – coated samples is about 40pm for 2 to 2.5 % hydrogen concentrations in nitrogen. All the sensing effect is expected to come from the evanescent field interaction.

Less sensitivity (slow response) to hydrogen removal and later exposures within reasonable time is the negative features observed on Pd based samples. The phase transition in Pd-H system usually observed at relatively high hydrogen concentrations is the likely reason for poor response to subsequent exposures. Thermal effects and, partly, effects from phase transition can be attributed to the weak response upon hydrogen removal.

Significant responses are measured from WO₃ coated SOI ring resonator hydrogen sensor. On the other hand, measurements have shown that the sensor sensitivity is dependent on environmental condition; largely on temperature. Keeping the sample at relatively high and constant temperature is necessary for improved response. Gas flow rate in the chamber is another factor which determines the sensor response. Fairly high flow rates give better
responses. This is due to the inherent enhancement of exothermic reactions with temperature and reactant supply rate. Around 340pm and 1.2nm resonance shifts are measured for 1.4% and 3% hydrogen in air respectively, at constant air flow rate of 1.3L/min and approximately constant sample temperature of about 40°C.

Sensitivity to much lower concentrations, and larger resonance shifts at a given concentration can be achieved operating at higher sample temperatures and with appropriate gas flow rates. Reducing the cladding thickness can also be considered while making sure that optical losses are not increased to unreasonable extent due the Tungsten oxide coating.

The larger resonance shifts observed with WO3 based sensor open the opportunity for inexpensive sensor implementation. The high sensitivity, fast response and implementation at integrated level of this SOI ring resonator based sensor, make it to comprise critical features expected from good hydrogen sensor. The heating requirement for efficient sensor operation can be achieved with optical means of heating. This avoids the need for electrical contacts and further ensures safe implementation of the sensor in potentially flammable hydrogen environment.
Chapter 5

Conclusions and Recommendations

5.1 Conclusions

Two SOI ring resonator based implementations of integrated optical hydrogen sensor have been analysed through numerical simulations and fabrications. Interesting outcomes have been achieved from this work. While sensitivity to hydrogen has been observed for both sensor implementations, the WO₃ coated sensor, which exploits thermo-optic effect, has shown impressive performance over the Pd coated one.

Simulations have shown that all the effects for Pd based sensor come from evanescent field interactions. This is due to the fact that mechanical effects are kept at insignificant level owing to thin Pd coatings used for practical reasons. Both from numerical predictions and measurements, the sensitivity of this sensor is found to be around 20pm per 1% hydrogen concentration in nitrogen environment. The low sensitivity of the sensor is to be attributed to the strong TE mode confinement in the HIC SOI waveguide.

Sensitivity to temperature and slow response to changing hydrogen concentration are negative aspects further observed about Pd based sensor implementation. This observation can be accounted to high temperature sensitivity of SOI structures and hysteresis effects commonly observed on Pd films.

On the other hand, far better performance has been achieved from WO₃ coated ring resonator hydrogen sensor. Over 1.2nm resonance shift is measured for 3% hydrogen in air, at temperature of 40 °C and 1.3 L/min air flow. This sensor has shown the potential to be
5.2 Recommendations

geared to a required level of sensitivity through proper choice of operating temperature and gas flow rate without the need for restructuring it.

The heating requirement for the WO$_3$ coated sensor can be achieved with controlled optical radiation, avoiding the need for electrical contacts which are unsafe in potentially flammable hydrogen environment.

Taking into account the current demand for ideal hydrogen sensor, this thermo-optically functioning integrated hydrogen sensor appears as a potential solution to fill the gap. The multiplexing capability, ultra-high compactness, compatibility with fibre networks and safety are among the interesting features which make the sensor an ideal candidate. Further more, the large resonance shift achieved with such sensing scheme along with CMOS compatibility provides the opportunity for inexpensive sensor implementation.

5.2 Recommendations

- Sensing with palladium coated structure can have potential application in areas involving nitrogen. The sensitivity of this sensor can be enhanced using TM modes which are more evanescent than currently used TE modes.

- To look for convenient alloys of palladium, in order to reduce the hysteresis effects and to improve adhesion of thicker Pd layers on SOI structures.

- Further study on efficient optical heating mechanism, reliability and life time for the WO$_3$ coated sensor. Integration of possible structures, such as gratings, that would make use of some of the input light for heating the catalyst.

- Considering tuning of other parameters such as coating and cladding thickness to further optimize sensitivity of the WO$_3$ based sensor
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