The study of long period gratings for improved refractive index sensing applications

Lim, Anthony

2013

Lim, A. (2013). The study of long period gratings for improved refractive index sensing applications. Doctoral thesis, Nanyang Technological University, Singapore.

<https://hdl.handle.net/10356/61307>

<https://doi.org/10.32657/10356/61307>

**NANYANG
TECHNOLOGICAL
UNIVERSITY**

**THE STUDY OF LONG PERIOD GRATINGS
FOR
IMPROVED REFRACTIVE INDEX SENSING
APPLICATIONS**

ANTHONY LIM

2013

ANTHONY LIM

School of Electrical & Electronic Engineering

2013

**THE STUDY OF LONG PERIOD GRATINGS
FOR
IMPROVED REFRACTIVE INDEX SENSING
APPLICATIONS**

ANTHONY LIM

School of Electrical & Electronic Engineering

A thesis submitted to the Nanyang Technological University
in partial fulfillment of the requirement for the degree of
Doctor of Philosophy

2013

Acknowledgement

This had been a long but enriching learning journey for me. In the process, many people had come into my life, inspired and motivated me towards this academic goal.

First, my greatest appreciations to my supervisor, Dr Tjin Swee Chuan who had accepted, patiently guided, consistently provided feedback to encouraged me all these years. My sincere thanks go to Dr Pita who had guided me and impressed on me the need to be detailed in my work. My appreciations also go to Dr Decoster of CNRS, Dr Jean Chazelas, Dr Jean-Pierre Huignard, Dr Myriam, all from THALES who had given me advices and guidance during my stay in NTU. I like to thank the NTU laboratory technicians for their support in equipment and parts during my laboratory work; namely Ms Yvonne Yee, Ms Seet, Mr Desmond Lim, Ms Debbie, Ms Poh Chee, Mr Chow

I also appreciate and wish to thank my supervisors, from past to present employment who had encouraged, inspired me to explore and finish this PhD program; they are namely Mr Frank Teo and Mr Tan Kian Hin, both from the DSO National Laboratory and Mr Fong Saik Hay from the ST Engineering. Also special thanks go to Mr Tan Seng Hua, DCE (A) and Mr Tan Kay Chuan (D/EIT/CC), both from Institute of Technical Education (ITE) who had been staunch supporter for life-long learning and had granted me time off to pursue and complete this academic endeavor.

To my parents, Mr Lim Gark Chew and Mdm Poh Ah Yeen who had silently stood behind me and supported me during all these years, I like to thank you.

Last but not the least, special thanks to my wife Sy Fei, my children Claire, Eugene and Gwyneth who had walked this journey with me and supported me all these years as I juggled between work, family and the PhD program.

This has been a long journey. However I hoped I had shared this life-long learning journey and the perseverance to complete with my children; I hope you can learn and emulate me in your learning journey.

<u>Table of Contents</u>	<u>Page</u>
Author's Works: Publications	viii
Conference	viii
Abstract	ix
List of Figures	xii
List of Tables	xvii
List of Acronyms	xviii

Chapter 1: Introduction

1.1 Introduction	1
1.2 Motivation	
1.2.1 Review of LPG for Ambient RI Sensing	6
1.2.2 Review of LPF as sensor for integration into Lab-On-A-Chip (LOC)	7
1.3 Research Objectives	8
1.4 Originality of Thesis	9
1.5 Organization of Thesis	10

Chapter 2: Background

2.1 Fundamental of Sensor	12
2.1.1 Type of sensors	12
2.2 Principle of Optical Sensors	14
2.2.1 Optical Fiber	14
2.2.1.1 Introduction	14
2.2.1.2 Mode Coupling in Optical Fiber	15
2.2.1.3 Phase Matching Condition	17
2.2.2 Fiber Braggs Gratings	22

2.2.2.1 Introduction	22
2.2.2.2 Phase-Matching Condition	22
2.2.2.3 Applications	25
2.2.3 Long Period Gratings	27
2.2.3.1 Introduction	27
2.2.3.2 Phase-Matching Condition	27
2.2.3.3 Mode Coupling in LPG	29
2.2.3.4 Characteristics Curves of Long Period Gratings	31
2.2.3.5 Coupling Coefficient	35
2.2.3.6 Transmission of Long Period Gratings	39
2.2.3.7 Effects of External Perturbation on Long Period Gratings	40
2.2.3.8 Effect of Temperature on Long Period Gratings	40
2.2.3.9 External Refractive Index effect on Long Period Gratings	43
2.2.3.10 Cladding Thickness Effect on Resonance Wavelength and Sensitivity	47
2.3 Development of LPG Sensors	49
2.3.1 Review of the development of LPG for Ambient RI Sensing	49
2.3.2 Review of LPG as Sensor for Integration into LOC	54

Chapter 3: Fabrication of Long Period Gratings

3.1 Fabrication of Long Period Gratings	59
3.1.1 Introduction	59
3.1.2 Fabrication Techniques	59
3.1.2.1 UV Amplitude Mask Technique	59

3.1.2.2 CO ₂ Laser Technique	63
3.1.2.3 Femtosecond Laser Pulse Technique	67
3.1.2.4 Electric Arc Technique	68
3.1.2.5 Ion Implantation Fabricated LPG	70
3.2 Challenges with Hydrogen-loaded LPG fiber	71
3.3 Annealing of LPG	75

Chapter 4: Improved RI sensitivity utilizing Long Period Gratings with Corrugations on Cladding

4.1 Introduction	79
4.2 Background	79
4.3 The Use of CO ₂ as the Ablation Source	84
4.4 Fabrication of the Corrugations on the Cladding of the LPG Sensor	88
4.4.1 Preparation of the hydrogen-loaded SMF	89
4.4.2 Setup of the CO ₂ laser system	89
4.4.3 Fabrication of the Corrugated Cladding LPG	91
4.5 RI Sensitivity of the Corrugated LPG	95
4.5.1 Preparation of the different RI solutions	95
4.5.2 Testing of the sensitivity of the Corrugated LPGs to different RI solutions	97
4.6 Summary	102

Chapter 5: Long Period Gratings with Controllable Refractive Index Sensitivity Using the RIE Process

5.1 Introduction	104
------------------	-----

5.2 Theory	107
5.2.1 Effect of Cladding thickness on Resonance Wavelength and Sensitivity	107
5.2.2 Chemical Etch process	108
5.2.3 Reactive Ion Etch process	109
5.3 Fabrication of the RIE-treated LPG Sensor	113
5.3.1 Preparation of the hydrogen-loaded SMF	113
5.3.2 Setup of the RIE system	113
5.4 RI Sensitivity of the RIE-treated LPG	115
5.4.1 Preparation of the different RI solutions	115
5.4.2 Testing of the sensitivity of the dry-etched LPGs to different solutions	115
5.5 Summary	119
 Chapter 6: Conclusions	
6.1 Conclusions	120
6.2 Recommendation for Future Research	
6.2.1 LPG Sensor	
6.2.1.1 Theoretical work on the corrugated long period gratings as an ambient RI sensor	124
6.2.1.2 Compensation for temperature effect on the corrugated long period gratings	124
6.2.1.3 Development of the LOC with the LPG integrated into the chip	125
6.2.2 Microfiber	125
 References	 126

Author's Works

Publications

1. Wen Bin Ji, Huan Huan Liu, Swee Chuan Tjin, Kin Kee Chow, and Anthony Lim, "Ultrahigh Sensitivity Refractive Index Sensor Based on Optical Microfiber", *IEEE Photonics Technology Letters*, Vol 24, No 20, Oct 15, 2012.
2. Anthony LIM*, Wen Bin JI, Swee Chuan TJIN, "Improved Refractive Index Sensitivity Utilizing Long Period Gratings with Periodic Corrugations on Cladding", *Journal of Sensors*, Volume 2012 (2012)
3. Josselin Pello, Trinh Le, Anthony Lim, Elhadj Dogheched, Myriam Kaba, Kantisara Pita, Didier Decoster, Jean-Pierre Huignard, Swee C. Tjin and Jean Chazelas, "Novel approach for planar Bragg grating characterization using prism coupling", *Optoelectronic Materials and Devices IV, Proc. of SPIE-OSA-IEEE Asia Communications and Photonics*, SPIE Vol. 7631, 76310A , 2009
4. M. Kaba, F. C. Zhou, , A.Lim, D. Decoster, "Evaluation of dispersive Bragg Gratings (BG) structure for the processing of RF signals with large time delays and bandwidth", *Proc. SPIE 6781, Passive Components and Fiber-based Devices IV*, 67812Q, November 21, 2007

Conference

5. Pello J, Le T, Lim A, Dogheche E, Kaba M, Pita K, Decoster D, Huignard J.P, Tjin, S.C, Chazelas J, "Novel approach for planar Bragg Gratings characterization using prism coupling", *Communications and Photonics Conference and Exhibition (ACP)*, 2009 Asia (Volume:2009-Supplement)

Abstract

Fiber optical-based sensor system offers many distinctive advantages over conventional electrical-based sensor system such as immunity to EMI, the ability to multiplex sensors into complex networks and low cost. One of these fiber optical-based sensors is the Long Period Gratings (LPG) that has the advantage of higher sensitivity to the ambient Refractive Index (RI) change compared to the Fiber Braggs Gratings (FBG) which makes it a suitable sensor for many applications in chemical, biological and environmental measurement and test.

Many different configurations of LPG on a Single Mode Fiber (SMF) had been developed with the highest sensitivity of 506.9nm/ RIU reported for a post-etched LPG with 81° tilted gratings written into it. There are other studies to introduce coatings to the cladding of the LPG using gold nano-rods, SiO₂ nano-particles to increase the sensitivity but it has trade-offs, one of which is the increase in the process steps and complexity with the additional materials coated onto the LPG cladding surface which increases the fabrication cost. In the first part of this study, **the LPG with Regular Corrugations on its Cladding** was developed and characterized with an improved sensitivity of 611nm/ RIU, compared to the other LPG sensors developed before. Using the CO₂ laser, corrugations on the LPG cladding was formed which encourages the interaction of the evanescent wave with the surrounding environment. With the control of the CO₂ laser beam power and speed, different corrugation depths and periods were fabricated and their effects on the LPG sensitivity were characterized. The effect of the corrugation periods on the resonance wavelength of the LPG was also studied. The study showed that the variation of corrugation depth changes the sensitivity of the sensor to the ambient RI while the changes in the corrugation period do not have any effect on the sensor sensitivity. On the other hand, the variation of the corrugation depth do not change the resonance wavelength significantly while the changes in the corrugation periods will caused a shift in the resonance wavelength. The experimental results show that this corrugated cladding LPG structure has good performances in terms of linearity and sensitivity to ambient RI changes

while it can also serve as a cost effective sensor. The innovative dual use of the corrugations to improve the LPG sensitivity, also served as a reservoir to collect and hold the specimen for testing. This sensor has the advantages of high sensitivity, small feature size, compact, ease of use and offers potential use in various chemical and biological sensing applications in the future. With the in-fiber corrugations that serve also as the collection reservoir, it can also be deployed as an onsite sensor to collect specimen for measurement and disposed thereafter due to its low cost.

In the second part of the study, **the LPG with Controllable Refractive Index Sensitivity** involves the innovative use of the semiconductor process, Reactive Inductive Etch (RIE) to accurately etch the cladding of the LPG sensor to the design requirements of the application. In past studies, reducing the cladding of the LPG using the chemical etch was the standard process used where the cladding is etched with the diluted 12% Hydrofluoric Acid (HF), achieving an etching rate of 20 μ m/min. This inhibited the development of an accurately designed LPG sensor on a 125 μ m SMF. However, the use of RIE with the smaller etch rate of 46nm/min allowed the development of LPG sensor with accurate RI ambient sensitivity. The effect of the different parameters in the RIE process, CF₄, O₂, Radio Frequency (RF) and chamber pressure on the etching of LPG cladding was studied. The results showed that the RIE process can effectively reduce the LPG cladding thickness with a resolution of 46nm and does not damage the functionality of the LPG. The study also showed that controlling the time of the RIE etch process, different LPG cladding thickness can be developed within the desired sensitivity. The characterization showed good system performances such as linear responses, good repeatability and low hysteresis, which is important for its use in industrial applications and where it has to operate in harsh environment. As a result, with the RIE process, fiber-based LPG sensor can be designed to the required sensitivity and integrated as a sensor into the silicon substrate platform to form the Lab-On-A-Chip (LOC) kit using the well-established RIE semiconductor process. Together with deposition, photolithography and etch processes, RIE can be used to fabricate the optical source and detector on the same silicon substrate platform

while it can also be simultaneously used to trim the fiber-based LPG to the designed sensitivity so that it can function as an onboard ambient RI sensor in the LOC system.

<u>List of Figures</u>	<u>Page</u>
Figure 1.1: Working Principle of an Optical Measurement System	2
Figure 2.1: Schematic of a sensor subsystem	12
Figure 2.2: Function integration and trends in sensor technology	13
Figure 2.3: Intensity profile of the fundamental guided mode in a SMF. It can be seen that the intensity profile extends partially into the cladding and extend into the cladding-air interface	15
Figure 2.4: β -plot showing the propagation constant of both the forward and backward propagating core and cladding modes where $\beta^{(1)}$ is the lowest forward propagating cladding mode	16
Figure 2.5: Perturbation in the core of the fiber can be induced by the Inscription of a uniform modulation of refractive index	17
Figure 2.6: Pictorial representation of diffraction of light rays passing through a fiber gratings	18
Figure 2.7: A simulation to show the exchange of energy between the two modes when the phase-matching condition is not met, $\delta \neq 0$ and (b) when phase- matching condition is met, $\delta = 0$	21
Figure 2.8: Transmission spectrum of a LPG with gratings period of $320\mu\text{m}$	22
Figure 2.9: A graphical representation of the Fiber Bragg Gratings	23
Figure 2.10: Characteristic curve of the FBG showing the relationship between the gratings period and the reflected wavelength	24
Figure 2.11: Mode coupling mechanism in the Fiber Bragg Gratings that shows large value of $\Delta\beta$ required small grating periods to achieve phase-matching conditions	25
Figure 2.12: A setup for the FBG sensor system	26

Figure 2.13: LPG characteristics curve of resonant cladding modes for a step index optical fiber LPG	28
Figure 2.14: Mode conversion of a fundamental core mode into the forward propagating cladding mode in a optical fiber with Long Period Gratings inscribed	29
Figure 2.15: β -plot of mode coupling mechanism in the LPG	30
Figure 2.16: Near field intensity distribution of $LP_{0,m}$ cladding modes with (a) $m=4$, (b) $m=5$, (c) $m=6$, (d) $m=7$	31
Figure 2.17: Characteristic curves for the first ten cladding modes in SMF-28	34
Figure 2.18: Different gradients of the slope show that different order of the cladding has different sensitivity to temperature change	42
Figure 2.19: Top graph shows the LPG resonance wavelength response to external index change	44
Figure 2.20: Experimentally measured core mode transmission losses for several refractive indices surrounding the LPG	45
Figure 2.21: Schematics diagram of the SLPGs and the corresponding mode coupling mechanism	50
Figure 2.22: Structure of the two tapered-type interferometer	51
Figure 2.23: Structure of the core-offset-attenuator-type interferometer	51
Figure 2.24: Schematic of the C-RLPGs	52
Figure 3.1 Setup configuration of amplitude mask technique for the fabrication of LPG	60
Figure 3.2: Evolution of the LPG using an amplitude mask after various UV exposures time: A to E represent different transmission spectrum after the 1 min exposure was initiated with E having the longest UV exposure	61
Figure 3.3 Transmission spectrum of a long period grating with a period of $\Lambda=320\mu\text{m}$	62
Figure 3.4 Setup for batch fabrication of long period grating just by using a simple amplitude mask	63

Figure 3.5:	Schematic setup of the LPG fabrication system using the CO ₂ laser	65
Figure 3.6:	The distribution of the optical power on the LPG fiber during the CO ₂ ablation which resulted in asymmetrical index change in the fiber	66
Figure 3.7:	Experimental setup for a LPG fabrication system using the femtosecond laser	67
Figure 3.8:	Setup of a LPG fabrication system using the electric arc system	69
Figure 3.9:	(a) Simulation, based on ion coordinates from SRIM, of 2.4 MeV H ⁺ ions implanted in to a silica fiber with a diameter of 120μm. (b) Setup of the Ion Implantation LPG fabrication	70
Figure 3.10:	Diagrams show the continual growth of the LPG with the grating period of 450μm spectrum immediately after UV-laser illumination has stopped. (a) spectrum immediately after the UV-laser is switched off. (b) spectrum after 2mins. (c) spectrum after 4mins. (d) spectrum after 6mins	71
Figure 3.11:	(a) Shift in resonance wavelength with time after UV illumination.(b) Growth in resonance wavelength dip with time after the UV Illumination	73
Figure 3.12:	Setup shows the exposed and unexposed region of the fiber during the UV illumination in the LPG fabrication process	74
Figure 3.13:	Shift in the resonance wavelength with the annealing at temperature 150°C	76
Figure 3.14:	Transmission spectrum of the 320μm LPG (a) before annealing (b) after annealing for 100°C for 20hrs	77
Figure 4.1:	A schematic of the LPG which shows the core mode confined in the fiber core while the cladding modes extends into the cladding-air interface	82
Figure 4.2:	Thermal gradient on glass substrate induced by CO ₂ irradiation	87
Figure 4.3:	Optimum energy deposition rate in inducing glass strip peeling	87
Figure 4.4:	Alignment of the LPGs onto the grid graph paper	90

Figure 4.5:	Setup of the CO ₂ laser system for fabrication of corrugations onto the cladding of the LPG	90
Figure 4.6:	Screen settings for power, speed in the Epilog Legend TT 7000 CO ₂ laser system	91
Figure 4.7:	(a) Corrugations period. (b) Corrugations depth	91
Figure 4.8:	Low frequency pulsing output from the CO ₂ laser head	92
Figure 4.9:	(a), (b): Measurement using the microscope: Side and top view respectively	94
Figure 4.10:	(a), (b): Using the SEM: Side and top view respectively	94
Figure 4.11:	The correlation between different concentrations of salt solutions with RI	96
Figure 4.12:	Setup of the corrugated LPGs for testing with different RI solutions	97
Figure 4.13:	The spectral response curve to different RI solutions	98
Figure 4.14:	The wavelength responses of the corrugated LPG (with period of $281.7\mu\text{m} \pm 3.4\mu\text{m}$, depth of $20.1\mu\text{m} \pm 0.4\mu\text{m}$) to different RI solutions	99
Figure 4.15:	Graph of RI vs Wavelength Change for different corrugation depths, keeping the corrugations period constant at $281.7\mu\text{m}$	100
Figure 4.16:	Graph of RI vs Wavelength Change for different corrugation periods, keeping the corrugations depth constant at $20.1\mu\text{m}$	101
Figure 5.1:	A schematic of the ion-etched LPG which shows the greater portion of the cladding modes field distribution extends into the cladding-air interface	105
Figure 5.2:	Transmission spectrum of the UV-irradiated LPG: shift in attenuation band with a reduction in cladding diameter	108
Figure 5.3:	Sensitivity versus the fiber diameter for LPG with gratings pitch of $360\mu\text{m}$ for ambient RI of 1.33	108
Figure 5.4:	Chemical etch process for removing cladding in a LPG	109

Figure 5.5:	A schematic of the RIE system	110
Figure 5.6:	Reaction of the free radicals with the SiO_2 substrate	110
Figure 5.7:	The selection of the F/C ratio for dry etch in the RIE	111
Figure 5.8:	Setup of the LPG on the RIE chamber	114
Figure 5.9:	(a), (b): SEM view of the LPG and RIE-etched LPG respectively	114
Figure 5.10:	Setup of the Ion-etched LPGs for testing with different RI solutions	116
Figure 5.11:	Wavelength Change (nm) for different cladding thickness of LPGs	117
Figure 5.12:	Linear trend for the different cladding thickness etched with the RIE process against sensitivity	118

<u>List of Tables</u>	<u>Page</u>
Table 1.1: Comparison of the Extrinsic and Intrinsic Fiber Optical sensors	3
Table 1.2: Comparison of the different fiber optical sensors' applications	3
Table 2.1: Sellmeier parameter for bulk glass samples of different composition	33
Table 2.2: Review of the different LPG-based ambient RI sensors	56
Table 2.3: Review of LPG structure on non-SMF or deposition of coating for improved sensitivity	58
Table 4.1: Equipment and materials list	89
Table 4.2: Matrix table for variation in speed and power with the Epilog Legend TT 7000 CO ₂ Laser and its effect on the cladding corrugations in the SMF fibers	93
Table 4.3: (a) Variation in the corrugations dimension formed with changes in the power and speed setting of the CO ₂ laser. (b) Corrugations period and depth	95
Table 4.4: Equipment and materials list	95
Table 4.5: Concentration of salt in water to achieve the different RI solutions	96
Table 5.1: Equipment and materials list	113
Table 5.2: Parameters for the dry etch of SiO ₂ fiber	113
Table 5.3: Equipment and materials list	115
Table 5.4: The correlation between the RIE exposure times, the resultant cladding thickness, average fiber thickness and the LPG sensitivity	118

List of Acronyms

EMI	electromagnetic interference
FBG	Fiber Bragg Gratings
LPG	Long Period Gratings
Mm	Millimeter
μm	Micrometer
nm	Nanometer
min	Minute
MHz	MegaHertz
RI	Refractive Index
UV	Ultraviolet
RIU	Refractive Index Unit
HF	Hydrofluoric Acid
PAH	Poly (allylamine) Hydrochloride
SMF	Single Mode Fiber
RIE	Reactive Inductive Etch
LOC	Lab-On-A-Chip
MEMs	Micro Electrical-Mechanical Systems
NEMS	Nano Electrical-Mechanical Systems
DWDM	Wavelength Division Multiplexing
CO_2	Carbon Dioxide
GeO_2	Germanium Dioxide
SiO_2	Silicon Dioxide
B	Boron
Ge	Germanium
H_2	Hydrogen
O_2	Oxygen

KrF	Krypton Fluoride
CF ₄	Carbon Tetrafluoride
EDFA	Erbium Doped Fiber Amplifier
HOF	Hollow Core Optical Fiber
TLS	Tunable Laser Source
OSA	Optical Spectrum Analyzer
DOE	Design of Experiments
SEM	Scanning Electron Microscope
Concentration _{Molar}	Molar Concentration of Salt Solution
Mass _{Molar}	Molar Mass of Salt
Volume _{water}	Volume of Water

Chapter 1: Introduction

1.1 Introduction

In an intelligent system, there is always a decision making subsystem where information are captured from external stimuli and evaluated. Thereafter a decision will be made based on programmed algorithm and the system performs specific tasks in relation to the external stimuli. The information is sensed from many different external sources via suitable sensors. These intelligent systems find many applications such as in civil engineering, biomedicine, aerospace, environment [1]. With its established technology, electrical subsystem that utilizes electrical-based sensor had been predominantly used for sensing in these applications. Despite their popularity, the electrical subsystem suffers from numerous disadvantages such as sensitivity to electromagnetic interference (EMI) and lack of multiplexing capability. To offset the EMI challenge, the normal copper electrical cables used to transmit the signal to the electrical sensor are replaced with shield copper cables which are more costly [1]. These challenges can be overcome with the adaptation of the optical system where the subsystem uses fiber optic sensors which has the advantages of immunity to EMI and also allows multiplexing to several sensors that can be read simultaneously.

Sensors are transducers that are used for measuring a physical quantity in the real world and then convert it into a signal so that it is easily readable by an instrument or processed in a subsystem. Many different types of sensors had been developed and they used different mechanisms to operate, such as acoustic, chemical, electromagnetic, ionizing radiation, mechanical, optical, and thermal [2]. The parameters that these sensors can measure are in many areas, such as pressure, heat, proximity, and reflective index etc. Compared to other sensor types, fiber optical sensors offer the advantage of immunity to EMI, has electrical isolation capability, compact and light with its small feature size, large dynamic bandwidth and passive [1]. It finds many applications in areas where safety consideration is a concern when the electrical-based sensors were used instead. The applications of fiber optic sensor can be increasingly found in areas like chemical, biological and environment detection and measurement [2]. The quest for more cost effective and efficient fiber optic

sensor devices serves as a motivating factor for more research and development efforts so as to increase the reliability of these sensors and to lower the fabrication cost. Technology advances in the optical industry had served as a catalyst that had driven the development of new optical products that sees its replacement of electrical sensors in civil engineering, biomedicine, aerospace, and environment [1] [2]. Despite these advantages, the implementation of fiber optic sensors in industries comes with a few concerns in the area of cost, fragility, reliability and complexity. There are also the challenges faced by the industry to develop and replace established product with different sensor types due to the consideration of reliability and accuracy in the product.

Incorporated as a fiber optic sensor, the measurand can be used for the detection of different parameters which includes pressure, flow, liquid level, displacement, vibration, rotation, magnetic field and acceleration. The working principle can be illustrated in Figure 1.1 where the transmitting light beam from the optical source is altered by the measurand in the measurement zone. The alteration of the light property can be in the form of intensity, phase, polarization, wavelength or spectral distribution and time-of-flight [2] changes.

Figure 1.1: Working Principle of an Optical Measurement System

There are fundamentally two classifications of fiber optic sensors and they are intrinsic or extrinsic sensor. For the extrinsic sensor, the measurement zone is in another transmitting medium, like air or liquid. The light from the Optical

Source leaves the feed fiber and its properties are changed in another transmitting medium and then continued to the detector by means of the return fiber. In the intrinsic sensor, the properties of light are changed while they are transmitting inside the optical fiber. The differences in performance of both the intrinsic and extrinsic fiber optic sensors and its use in different applications are tabulated in Table 1.1 and Table 1.2 respectively [2].

Extrinsic Sensor	Intrinsic Sensor
Applications – temperature, pressure, liquid level, flow	Applications – rotation, acceleration, strain, acoustic pressure, vibration
Less sensitive	More sensitive
Easily multiplexed	Cannot be multiplexed
Ingress/ egress connection problems	Reduce connection problems
Easier to use	More elaborate signal demodulation
Less expensive	More expensive

Table 1.1: Comparison of the Extrinsic and Intrinsic Fiber Optic Sensors [2]

Sensor Type	Applications	Industry
Chemical sensor	Remote spectroscopy, Ground water and soil contamination monitoring	1) PHARMACIA BIOTECH (SWEDEN) 2) FIBERCHEM 3) THE QUANTUM GROUP
Temperature sensor	Large temperature range, -40 ⁰ C to 1000 ⁰ C	US-small companies, Japan-HITACHI and SUMITOMO
Strain sensor	Used Fiber Bragg Gratings (FBG) technology. Sensed as little as 9 microstrain	NRL and UNITED TECHNOLOGY RESEARCH
Biomedical sensor	Spectroscopic biomedical sensor. Can sense CO ₂ , O ₂ and ph simultaneously. Flow monitoring by Laser Dopplerimetry	
Electrical and magnetic sensor	Inherent dielectric nature, small size and safe, hybrid.	ABB CORPORATION RESEARCH CENTER
Rotation sensor	Based on Sagnac effect. Laser gyroscope or fiber optics gyroscope	HONEYWELL, LITTON, NORTHROP, ALLIED SIGNAL etc
Pressure sensor	Based on moving diaphragm. 0 to 70000 Torrs	
Image sensor	Allow low light imaging, night vision	

Table 1.2: Comparison of the different fiber optic sensors' applications [2]

The optical fiber has been a target of intensive research and development into an optical sensor. It is used extensively in the modern world and in many different applications. It was first demonstrated by Daniel Colladon and Jacques Babinet in Paris in the early 1840s with the guiding of light by refraction [3]. Using the optical fiber, the first in-fiber Bragg Gratings was demonstrated by Ken Hill in 1978 [3]. The gratings were then fabricated using a visible laser that propagated along the core of a photosensitive fiber from both ends of the fiber. The standing wave formed within the fiber causes a periodic variation of the refractive index inside the core, and hence formed the first Bragg Grating. In 1989, Gerald Meltz and colleagues demonstrated the fabrication of a fiber Bragg grating using the transverse holographic inscription technique which offers more flexibility since the laser illumination is from the side of the fiber [3]. The periodic refractive index variation in the fiber is created from the interference pattern in the intersection zone formed by two beams originating from a single ultraviolet laser light source. Thereafter, other fiber optic device, such as the Long Period Gratings (LPG) was developed and fabricated onto the optical fiber sensor platform.

For strain, pressure and temperature measurement, the two most commonly implemented optical fiber sensors are the Fabry-Perot optical fiber sensors and the fiber grating-based sensors. A Fabry-Perot optical fiber sensor uses the concept of Fabry-Perot interferometer, in which interference occurs at the partially reflecting end face of an optical fiber and an external mirror [1]. The Fabry-Perot optical fiber sensors offers high sensitivity but are only a single point sensor; they cannot be multiplexed to form multiple sensor network to sense several external stimuli simultaneously or position at several locations at the same time. In addition, to achieve good interference signal, precision alignment between the fiber and the external mirror is critical. On the other hand, fiber-based gratings sensor operates based on the changes on the gratings that are inscribed in the optical fiber core and hence it will not require any precise alignment. With the fiber-based gratings sensor, it can be multiplexed into a complex sensor network if required [4].

Another important application of optical fibers sensor is in the area of chemical and biomedical sensing. In these areas, fiber-based gratings sensors are used. There are two main types of fiber-based gratings sensors, namely short period or Fiber Bragg Gratings (FBG) and the transmission-based or Long Period Gratings (LPG). The FBG sensor operates on the principle of the change in the optical properties of the back reflection of light within the core of the fiber, which will result in changes of the back reflected wavelength. FBG sensors have been implemented in various types of perturbation monitoring such as strain, temperature [5, 6, 7] and chemical [8, 9, 10]. The principle of the LPG however, is based on the change of the optical property of the forward propagating cladding modes. An LPG consists of a uniform modulation of the refractive index, with grating period between $100\mu\text{m}$ and $1000\mu\text{m}$, inscribed within the core of the optical fiber. This modulation of refractive index results in the coupling of the fundamental forward propagating core mode into discrete forward propagating cladding modes which are sensitive to changes of the refractive index surrounding the fiber.

The advantages of LPG over the FBG include the elimination of back reflection as the cladding modes in the LPG are forward propagating. The back reflection, if it occurs will induce source oscillation which will in turn affect the accuracy of the signal output from the sensor [10]. To overcome this back reflection, an optical isolator is usually used to prevent this transmission back to the light source. In terms of fabrication cost, the LPG requires a less costly metallic amplitude mask for direct-writing, as compared to the relatively expensive quartz phase mask used in the fabrication of the FBG. Hence as a sensor, the LPG offers a relatively cost effective option compared to the FBG system. Another advantage in the use of the LPG as a sensor is the relatively simpler fabrication of sensitive sensor for use in chemical or refractive index sensing applications. Using an FBG for sensitive chemical or refractive index sensing can be very complex as it will requires the etching of the cladding to within a few microns from the core to allow the evanescent field of the guiding mode [11, 12] to propagate into the surrounding medium. This etching process reduces the structural strength and integrity of the sensor and degrades the FGB sensor's performances under harsh environment conditions. On the other

hand, the LPG has inherent higher sensitivity for the ambient refractive index detection as the trailing portion of the cladding modes extends out of the cladding-air interface. This allows the cladding modes to interact with the ambient refractive index change which makes it more sensitive, compared to the FBG. Hence the structural integrity of the LPG is better, compared to the FBG for use in general sensitive ambient RI sensing. With the advantages of higher sensitivity to ambient refractive index change without compromising structural integrity, developing the LPG as a fiber sensor has attracted much interest over the years.

1.2 Motivation

1.2.1 Review of LPG for Ambient RI Sensing

An important characteristic for the LPG to be deployed as a chemical or biomedical sensor is its ambient Refractive Index (RI) sensitivity. In these applications, with the sensitivity of the LPG to the change in the ambient RI, the characteristic in the shift of the cladding modes detected can be used to identify the change in the RI detected. This change in the ambient RI can be attributed to different materials or solutions that the LPG is immersed in. With this attribute, the LPG can also be used as a concentration detector to identify the amount of dilution for a given material/ solution. As an ambient RI sensor, the relatively lower cost of the LPG, compared to the FBG that requires etching for improved sensitivity, the LPG offers as a good sensory platform that can be deployed in many chemical and biological sensing applications [13]. Many different configurations of LPG on a SMF had been developed and papers published [14, 15, 16, 17, 18, 19]. The standard LPGs are direct-written by UV exposure to produce the gratings which couple light from the core mode to various forward propagating cladding modes in the single mode fiber (SMF). As a result, at discrete wavelengths, a series of attenuation bands in the fiber transmission spectra are created. This sensor works on the principle of changes in the wavelength of these forward propagating modes with the variation in the external environment parameters such as ambient RI, temperature or strain. From the development work on different configurations of LPG to achieve the sensitive ambient RI sensor that will be covered with more details in Chapter 2, the highest sensitivity of 506.9nm/ RIU was obtained for a

post-etched LPG with 81° tilted gratings written in it [21]. The introduction of external coatings of gold nano-rods, sol-gel, SiO_2 nano-particles to the cladding of the LPG further increased the LPG sensitivity [22, 23, 24, 25, 26] but it has other trade-offs, one of which is the increase in the process steps and complexity with the additional materials coated onto the LPG cladding surface. Hence the motivation for this study is to design and develop the LPG sensor with higher sensitivity for ambient RI detection but has low fabrication cost by utilizing solely a different configuration of LPG and not to include additional external coating. This is found in the development of the LPG with Regular Corrugations on its Cladding. This sensor has an improved sensitivity of 611nm/ RIU compared to the other LPG sensor developed.

1.2.2 Review of LPF as sensor for integration into Lab-On-A-Chip LOC

The summary of the development work on developing the LPG as a sensitive sensor are tabulated below: A. Ladicicco had also reported using chemical etch to improve RI sensitivity; exposing the LPG cladding to a diluted 12% hydrofluoric acid to achieve an etching rate of $20\mu\text{m}/\text{min}$ [27]. The highest sensitivity for the LPG without any introduction of coating on the cladding is reported with a RI sensitivity of 506.9nm/RIU using the 81° tilted LPG, followed by wet-etch [21]. For the LPG coating of gold nano-rods, sol-gel, and SiO_2 nano-particles, wet etch is always used to reduce the cladding thickness, followed by deposition of different nano-based particle. In all these chemical etch processes to improve the sensor RI sensitivity, the etch rate of $20\mu\text{m}/\text{min}$ is high due to the nature of the wet etch process and when used to remove the cladding on a single mode fiber (SMF) of $125\mu\text{m}$, it is not easy to control the amount to be etched to the μm resolution accurately. As a result, using the chemical etch method, the sensitivity of the LPG which depends on the amount of cladding thickness removed cannot be fabricated accurately to any designed value due to the high etch rate from the wet etch process. The motivation for this study is to develop LPGs sensitivities that can be accurately controlled. This will allow the LPGs to be used as a sensitive sensor and be integrated into the Lab-On-A-Chip (LOC) module. This LPG with the sensitivity that can be designed and controlled to the μm resolution accurately can be found in the

development of the LPG with Controllable Refractive Index Sensitivity Using the RIE process.

1.3 Research Objectives

Fiber optic-based sensor system offers many distinctive advantages over conventional electrical-based sensor system such as immunity to EMI, the ability to multiplex sensors into complex networks and low cost. One of these fiber optic-based sensors is the LPG that has the advantages of higher sensitivity compared to the FBG, coupled with the low cost of fabrication that makes it a good candidate in applications that requires the measuring sensor to be disposed after used. Hence it finds good applications in chemical, biological and environmental measurement and test.

The objective of this study is based on the review the sensitivity of the developed LPG as the sensitive sensors and to use it as a baseline to develop a higher sensitive LPG-based sensor using a different configuration that will not increase its insertion loss while still maintaining the low fabrication cost. The first novel LPG sensor, known as the LPG with Regular Corrugations on its Cladding had been developed and characterized. The LPG with Regular Corrugations on its Cladding has improved sensitivity compared to the other LPG sensor with a sensitivity of 611nm/ RIU. The corrugations serve also as a reservoir to collect the specimen which allows this sensor to be used in many chemical and biological applications as a low cost sensor as part of the disposable RI measurement kit. Because the improvement in sensitivity is due to the corrugations fabricated on the cladding of the LPG, there is no additional material implanted onto the LPG sensor and hence it will not increase the insertion loss of this sensor. The method of using CO₂ to irradiate on the cladding to create the corrugation will ensure the low fabrication cost of this sensor.

The second objective is also to develop a novel LPG sensor with high sensitivity that can be designed and fabricated accurately to meet different application needs; such as in the design and integration of LPGs onto the Lab-On-A-Chip (LOC) platform. The LPG with Controllable Refractive Index

Sensitivity using the RIE Process had been developed and characterized. This will allow the fiber optic LPG to be designed and integrated as a sensitive sensor into silicon substrate platform to form the LOC kit using the well-established RIE semiconductor process; the development of optical source and detector on the silicon substrate platform using the semiconductor processes, such as deposition, photolithography, etch had been researched extensively. Using the RIE, the etch process can be used to fabricate the optical source and detector while it can simultaneously be used to trim the fiber optical LPGs to the designed sensitivity needed to make a complete LOC system. This process is more efficient as there is no need to fabricate a waveguide-based LPG sensor using a multiple of semiconductor processes.

The characterization of both the LPG sensors showed good system performances such as linear responses, good repeatability and low hysteresis, which is important for its use in industrial applications where it has to operate in harsh environment.

1.4 Originality of Thesis

For the LPG with Regular Corrugations on its Cladding, from the best of the literature searches, there is no similar work to fabricate corrugations on LPG to improve the ambient RI sensitivity. The corrugations had improved the sensitivity of this sensor to 611nm/ RIU which is the highest reported ambient RI sensitivity so far. In addition, the corrugations also serve as a reservoir for collection of specimen when this sensor is used for biological, chemical or environmental applications. This will allow onsite collection of specimens onto the LPG sensor and thereafter the LPG sensor can transport back to the laboratory for measurement and analysis.

The Long Period Gratings with Controllable Refractive Index Sensitivity is unique as it uses the semiconductor process to improve and tune its sensitivity accurately to suit the application needs. From literature searches, there had been no report in the use of semiconductor technology on fiber optics as they belong to different technology platforms. However with this work, it will be possible to design, fabricate and integrate the LPG-based sensors accurately

with the use of the RIE process which the process can be simultaneously used to fabricate other functional devices for the LOC applications.

1.5 Organization of Thesis

This section gives an outline of the content of this thesis. It introduces the mechanism for ambient RI detection of the LPG-based sensors. It gives the motivation for the development of the LPG with Regular Corrugations on its Cladding as a highly sensitive ambient RI sensor. It also covers the motivation for the development of the LPG with Controllable Refractive Index Sensitivity Using the RIE Process to design LPG sensor with accurate sensitivity for integration onto the Lab-On-A-Chip (LOC).

Chapter 2 will outline the theoretical concept of LPG-based sensor and mode coupling between the forward and backward propagating modes. For maximum power transfer between the two modes, phase matching condition must be met. The chapter also covers an overview of the short period grating or otherwise known as the Fiber Braggs Gratings (FBG) as a reference. In the FBG, when the phase matching condition is satisfied, the forward propagating fundamental core mode is coupled to the backward propagating core mode. Comparing with the FBG, the LPG theoretical operation mechanism, characteristic curves and coupling coefficients will be introduced. It traces the development of different LPG configurations and its reported sensitivity. It also includes the recent developments where fabrication of LPG structure on non-SMF or deposition of coating were made to improve sensitivity

Chapter 3 will cover the fabrication techniques of the LPG through the use of the ultra-violet laser radiation on the photosensitive optical fiber via the amplitude mask. Besides the amplitude mask, other techniques such as using the CO₂ laser, the femtosecond laser, electric arcing and the Ion Implantation will also be covered and discussed. In addition, the annealing of LPG fabricated on hydrogen-loaded SMF will also be covered.

Chapter 4 will cover the experimental results and discussions on the novelty of the LPG with Regular Corrugations on its Cladding as the ambient RI sensor. It

is characterized to have the highest ambient RI sensitivity so far (see Table 1.3). A full characterization on this sensor was carried out and the results will be presented in this chapter.

Chapter 5 focuses on the LPG with Controllable Refractive Index Sensitivity Using the RIE Process which is the semiconductor process technology. Characterization results will be presented that will include sensitivity, repeatability, hysteresis, stability will be discussed.

Chapter 6, the last chapter of this thesis, is a summary of the work done for this thesis, along with recommendations for future work which will encompass the theoretical work of integrating the two sensors developed, individually as the highly sensitive and low cost sensors for chemical and biological applications.

Chapter 2: Background

2.1 Fundamental of Sensors

2.1.1 Type of sensors

The term sensor is often used as a super-ordinate term to describe any component that generates a usable response from a measured physical parameter, as illustrated in Figure 2.1. In its simplest form, a sensor consists merely of a naked sensor element, for instance in a pressure sensor element made of silicon and mounted on a substrate, no more than a few millimeters in exterior dimension. The term “measured physical parameter”, refers to the principle of physical or chemical conversion resulting in a usable response, which is commonly in electrical domain. The measuring parameter is hence converted to an internal signal based on a physical measuring principle of the sensor element.

Figure 2.1: Schematic of a sensor subsystem [28].

There are different types of sensors that can be used for measuring different physical parameters; this includes process sensors, industrial sensors, specialized sensors (extra-corporeal and intra-corporeal) or mass sensor [28]. It can manifest the physical measurement principles in the form of mechanical (ultrasonic, resonant), thermal, chemical, electrical (resistive, capacitive, piezo-resistive, piezo-electric), magnetic (microwave) and radiation (optics) [28] [29]. The trend is developing towards a multi-sensing sensor subsystem which is smarter, lower in cost, decentralized, self-testing and embedded (small form size). It is developing towards integrating the sensor subsystem towards a

MEMS or NEM (micro or nano electrical-mechanical systems) technology with an increasing use of silicon in the measuring elements, for calorific and chemical parameters [28]. There is also an increasing application of silicon as a substance for measuring elements for various physical parameters, besides pressure and acceleration, it can be also be used for force, humidity, and temperature sensing applications [28]. The utilization of silicon allows further reduction of sensor-element size by application of micromechanics technology. Figure 2.2 shows the functional integration and trends in sensor technology.

Figure 2.2: Function integration and trends in sensor technology [28]

Compared to other sensor types, the optical sensor offers the advantage of immunity to Electromagnetic Interference (EMI), electrical isolation, compact and light weight with small feature size, large dynamic bandwidth and passive [29]. It finds applications in many areas where undesired electrical interferences can affect safety consideration which will be a concern and

priority, such as in flight where electrical interference can affect instrumentation and navigational system. The applications of the optical sensors as a sensitive sensor are also increasingly found in areas like chemical, biological and environment monitoring and measurement. In the development of integrated optical sensor, fiber Bragg grating arrays (for strain gauges and temperature measurement), and miniaturized optical solid-state resonators on semiconductor platform (e.g. neodymium YAG crystals, miniaturized spectrometers, wavelength tunable semiconductor lasers) had been studied and developed.

2.2 Principle of Optical Sensors

2.2.1 Optical Fiber

2.2.1.1 Introduction

In a perfect or ideal situation where the physical dimension remains homogenous along the longitudinal axis of the fiber, the refractive index along the optical fiber will be constant and hence the fundamental guided core mode will propagate unaffected in the core of the optical fiber. However due to manufacturing process which causes fiber imperfection, perturbation due to the refractive indices long the fiber length occurs which may cause the fundamental mode to be couple into other higher order radiation modes which is not desirable. In the case of the Fiber Bragg Gratings (FBG) and the Long Period Gratings (LPG), periodic perturbations of the refractive index along the fiber length are formed by exposing the fiber core to an intense optical interference pattern. This results in the FBG that has good ambient RI discrimination when its cladding is etched while the LPG has inherently better RI sensitivity compared to the FBG. Hence both these sensing devices have potential for use in the sensing applications.

In the development of this chapter, an overview of the mode coupling mechanism in the optical fiber will be discussed, as this will form the fundamental concept of the Bragg Grating technology. Although the LPG is the focus of this study, an overview on the concept of the FBG will also be presented, as a reference and comparison. This will provide a more holistic picture of the two basic types of gratings for comparison and allows the reader

to appreciate the LPG as the sensor of choice. Various characterizations of the LPF properties will be included in this chapter. These will be the characteristic curves, the differential propagation constant, coupling coefficient and the modal overlap between the fundamental guided mode and the cladding modes over the region of the LPG.

2.2.1.2 Mode Coupling in Optical Fiber

The propagation of light along an optical fiber can be described in terms of a set of guided electromagnetic waves that are commonly referred to as modes of the optical waveguide. Each guided mode is a distribution of electrical and magnetic fields, with the fields repeated along the optical fiber in periodic intervals. The fundamental core mode, as shown in Figure 2.3 below is tightly confined in the core of the Single Mode Fiber (SMF). For a thinned cladding fiber, the electric field, in the form of the evanescent wave, which follows the properties of the wave equations will extend past the cladding-air interface and into the ambient environment outside of the fiber.

Figure 2.3: Intensity profile of the fundamental guided mode in a SMF. It can be seen that the intensity profile extends partially into the cladding and extend into the cladding-air interface [30]

For a monochromatic light with radiation frequency, ω , a mode travelling in the direction of z along the fiber has a time and z dependency given by [30]:

$$e^{j(\omega t - \beta z)} \quad (2.1)$$

The factor β is the z component of the wave propagation constant in the medium given by $\beta = nk$ where k is the propagation constant in vacuum and n is the refractive index of the medium. For the guided mode to propagate and be

confined along the core of the fiber, β has a discrete range of value given by $n_2k < |\beta| < n_1k$ where n_1 and n_2 are the core and cladding refractive indices respectively [30].

Another common graphical representation to describe β in both forward and backward propagating guided and radiation modes can be shown by the β -plot in Figure 2.4 [31]. The propagation constant, β is written as $\beta = \frac{\omega n}{c}$ where n is the effective refractive index of the medium through which the mode propagates and $n_2 < n < n_1$.

Figure 2.4: β -plot showing the propagation constant of both the forward and backward propagating core and cladding modes where $\beta^{(1)}$ is the lowest forward propagating cladding mode [31]

The fiber also allows the propagation of an infinite continuum of radiation modes which are not trapped in the core but are still guided along the fiber. These radiation modes are caused by the optical beam falling outside the fiber acceptance angle which causes the waves to be refracted out of the core. Due to the finite radius of the cladding thickness, some of radiation modes were trapped in the cladding, giving rise to cladding modes [30]. Like the guided core mode, the electric fields of the cladding modes are not confined completely within the cladding thickness but will extend partially onto the cladding-core interface and also extend into the cladding-air interface. As these cladding modes propagate along the fiber, they may get coupled to each

other resulting in an energy transfer from the fundamental guided mode to discrete cladding or lossy modes.

2.2.1.3 Phase Matching Condition

In the fiber, perturbation in the core can be induced by the inscription of a uniform modulation of refractive index (RI). For the core and cladding modes to interact and transfer significant amount of energy in the region of the RI modulation, a phase matching condition must be fulfilled between the 2 modes. The coupled mode equations will be used to show this phase- matching relationship. In the fiber gratings shown in Figure 2.5 below, a uniform modulation of refractive index is inscribed in the fiber core. This modulation of refractive index is created with the inscription of a fiber grating. This RI modulation creates a perturbation to the core propagating mode which is travelling in the fiber core.

Figure 2.5: Perturbation in the core of the fiber can be induced by the Inscription of a uniform modulation of refractive index

The fiber grating is an optical diffraction gratings and the relationship between the incident and the diffracted wave can be shown pictorially in Figure 2.6.

Figure 2.6: Pictorial representation of diffraction of light rays passing through a fiber gratings [32].

From the figure above, its operations can be described by the following grating equation [32]

$$n_2 \sin \theta_2 = n_1 \sin \theta_1 + m \frac{\lambda}{\Lambda} \quad (2.2)$$

where θ_2 is the angle of the diffracted wave, n_2 and n_1 are the refractive indices of the mediums respectively, and m denotes the diffraction order. This equation calculates the diffracted angle, θ_2 , into which the constructive interference occurs while it can also be used to determine the wavelength in which the fiber gratings can most efficiently couple light between the two modes.

In the energy transfer between the 2 modes in the RI modulation region, we denote the two modes to be a_1 and a_2 travelling in the z direction, which is along the fiber axis and with β_1 and β_2 as the discrete propagation constant respectively. Starting with the lossless unperturbed fiber, the relationship can be described by [33]

$$a_1(z, t) = A_1(z) e^{j(\omega t - \beta_1 z)} \quad (2.3a)$$

$$a_2(z, t) = A_2(z) e^{j(\omega t - \beta_2 z)} \quad (2.3b)$$

where ω is the angular frequency, $A_1(z)$ and $A_2(z)$ are the complex normalized amplitudes that are dependent of z .

In the case where the RI modulation is present, the energy transfer between the two modes will be such that $A_1(z)$ and $A_2(z)$ are functions of the propagation distance, z with the amplitude $A_1(z)$ and $A_2(z)$ following [33]

$$\frac{dA_1(z)}{dz} = \kappa_{12}A_2(z)e^{-j\Delta z} \quad (2.4a)$$

$$\frac{dA_2(z)}{dz} = \kappa_{21}A_1(z)e^{-j\Delta z} \quad (2.4b)$$

where κ_{12} and κ_{21} are the cross-coupling coefficients that determine the amount of coupling between the two modes, Δ is the phase mismatch between the propagating modes in the presence of a perturbation with period Λ and can be denoted by [33]

$$\Delta = \beta_1 - \beta_2 - \frac{2\pi}{\Lambda} \quad (2.5)$$

Δ has a strong dependence on the β_1 and β_2 as these propagation constants are functions of the optical wavelength. For synchronized transfer of power between the modes, the value of Δ can be assumed to be ideally zero which will result in the following [33]:

$$\beta_1 - \beta_2 = \frac{2\pi}{\Lambda} = \Delta\beta \quad (2.6)$$

where $\Delta\beta$ is the differential propagation constant. This is known as the phase-matching condition.

From equations (2.3a) and (2.3b), assuming $z=0$, $a_1(0)=A$ and $a_2(0)=0$, when a_1 interacts with the perturbations to form a propagating polarization wave that result in a_2 . The generated mode, a_2 , reacts to the perturbations to alter the energy in a_1 . This continuous exchange of energy between a_1 and a_2 will depend on the differential propagation constant $\Delta\beta$ and the period of the RI modulation. When the phase matching condition is met, the power transfer between the two modes is then a function of the coupling coefficient. On the

other hand, if the phase-matching condition is not met, the magnitude of interaction between the two modes is negligible.

The power residing in the two modes can be obtained from the complex modal amplitude, $A_1(z)$ and $A_2(z)$ which is expressed as [33]

$$A_1(z) = A \frac{2\kappa_{12}}{\sqrt{4\kappa^2 + \Delta^2}} e^{j\frac{\Delta z}{2}} \sin\left[\frac{1}{2}(\sqrt{4\kappa^2 + \Delta^2})z\right] \quad (2.7)$$

$$A_2(z) = A e^{j\frac{\Delta z}{2}} \left\{ \cos\left[\frac{1}{2}(\sqrt{4\kappa^2 + \Delta^2})z\right] - j \frac{\Delta}{\sqrt{4\kappa^2 + \Delta^2}} \sin\left[\frac{1}{2}(\sqrt{4\kappa^2 + \Delta^2})z\right] \right\} \quad (2.8)$$

where κ_{12} is the cross coupling coefficient from mode a_1 to mode a_2 and $\kappa^2 = |k_{12}|^2$.

Hence the power of the two modes, $A_1(z)$ and $A_2(z)$ can be shown as [33]

$$p_1(z) = |A_1(z)|^2 = \frac{P_0 \sin^2\left[\kappa z \sqrt{1 + \left(\frac{\delta}{\kappa}\right)^2}\right]}{1 + \left(\frac{\delta}{\kappa}\right)^2} \quad (2.9)$$

$$p_2(z) = |A_2(z)|^2 = P_0 \left\{ \frac{\cos^2\left[\kappa z \sqrt{1 + \left(\frac{\delta}{\kappa}\right)^2}\right] + \left(\frac{\delta}{\kappa}\right)^2}{1 + \left(\frac{\delta}{\kappa}\right)^2} \right\} \quad (2.10)$$

where $\delta = \frac{\Delta}{2}$ is termed the detuning parameter and P_0 is the incident power and is denoted by $P_0 = |A|^2$

From the above 2 equations, the frequency and coupling magnitude are functions of the detuning ratio, $\frac{\delta}{\kappa}$ [33]. The plot in the figure below shows the variations in the normalized incident power $P_1(z)$ and $P_2(z)$ as functions of κz for different values of detuning ratio.

Figure 2.7: A simulation to show the exchange of energy between the two modes when the phase-matching condition is not met, $\delta \neq 0$ and (b) when phase-matching condition is met, $\delta = 0$ [33]

From the Figure 2.7, when the phase matching condition is satisfied, $\delta = 0$, the exchange of power between the two modes will be at the maximum. When this condition is not met, the power transfer will be reduced. Hence at the phase-matching condition, the above two equation will be reduced to the following [33]:

$$P_1(z) = |A_1(z)|^2 = P_0 \sin^2[\kappa z] \quad (2.11)$$

$$P_2(z) = |A_2(z)|^2 = P_0 \{\cos^2[\kappa z]\} \quad (2.12)$$

From the above equations, it can be seen that the magnitude of coupling is dependent on the coupling coefficient, κ and the length of RI modulation, z .

From the Figure 2.7(b), for maximum coupling to occur, $\kappa z = \frac{m\pi}{2}$, where m is an integer. The power transfer can further be developed from the equations above to the following [33]:

$$C = \frac{P_1(z)}{P_0} = \sin^2 \kappa z. \quad (2.13)$$

This equation determines the power that can be transferred between modes and the coupling coefficient; in the case of the LPG transmission, κ will determine the depth of the resonance dip as shown below in Figure 2.8.

Figure 2.8: Transmission spectrum of a LPG with gratings period of 320um.

In summary, the coupling and transfer of power between two forward propagating modes in an optical fiber has been discussed. To allow this transfer of power, the phase-matching conditions need to be satisfied. When this condition is met, the power varies sinusoidal along the direction of the RI modulation. This is the primary condition that governs the operations of the FBG and LPG.

2.2.2 Fiber Braggs Gratings

In this section the principle of operations of a FBG, phase-matching condition and its applications will be discussed:

2.2.2.1 Introduction

Fiber Gratings can be divided into short period gratings or long period gratings. To give a complete picture of the fiber gratings, an overview of the fiber Bragg Gratings will be presented here for comparison.

2.2.2.2 Phase-Matching Condition

Fiber Bragg Gratings (FBG), demonstrated by Ken Hill in 1978 was based on the phase-matching between the forward propagating and backward

propagating LP_{01} guided core modes in the presence of a periodic modulation of refractive index in the core of the optical fiber [34]. The Figure 2.9 shows the schematic of the FBG.

Figure 2.9: A graphical representation of the Fiber Bragg Gratings [34]

From the phase-matching equation, the propagation constant for β_1 and β_2 in Equation (2.6) can be written as [34]

$$\beta_1 = n_{core}^{eff} \kappa \quad (2.14)$$

$$\beta_2 = -\beta_1 = -n_{core}^{eff} \kappa \quad (2.15)$$

where n_{core}^{eff} is the effective refractive index in the core of the fiber and $\kappa = \frac{2\pi}{\lambda}$ is the free-space propagation constant. β_2 has the negative sign as it is travelling in the counter-propagating direction. Inserting these into Equation (2.6) yields [34]

$$2\left(\frac{2\pi}{\lambda} n_{core}^{eff}\right) = \frac{2\pi}{\Lambda} \quad (2.16)$$

which can be simplified into the Bragg equation or phase-matching equation for fiber Bragg Gratings [34].

$$\lambda_{ref} = 2n_{core}^{eff} \Lambda \quad (2.17)$$

where λ_{ref} and Λ are the reflected Bragg wavelength and grating period respectively. From Equation 2.17, it can be seen that the Bragg Wavelength is directly proportional to the grating period and the effective refractive index of the core. Changes in either of these will cause the reflected wavelength to shift. The characteristic curve of the FBG can be obtained from the differentiation of the equation where

$$\frac{d\lambda_{ref}}{d\Lambda} = 2n_{core}^{eff} \quad (2.18)$$

This gives a linear relationship between the reflected wavelength and the grating period which can be represented pictorially in the Figure 2.10 below. An incremental change in the grating period will cause an approximate similar magnitude of incremental change in the Bragg wavelength.

Figure 2.10: Characteristic curve of the FBG showing the relationship between the gratings period and the reflected wavelength [34].

Figure 2.11: Mode coupling mechanism in the Fiber Bragg Gratings that shows large value of $\Delta\beta$ required small grating periods to achieve phase-matching conditions [34]

Using the β plot shown in Figure 2.11, it can be seen that for an FBG under phase-matched condition, only a small grating period can result in large $\Delta\beta$ [34]. Typical grating period for optical communication applications at 1550nm wavelength lies between 524nm and 559nm.

2.2.2.3 Applications

FBG has found many applications in communication, sensing network, true-time delay etc. In the optical communication, it has been deployed as an optical filter in Dense Wavelength Division Multiplexing (DWDM) network, dispersion compensation, tunable filter, fiber amplifier [35]. Beside its application in optical communication system, the FBG has also been utilized as a sensor. In sensing applications, it had been used to sense external perturbation dynamically such as temperature, strain, and vibration, etc. As any of these external perturbations will cause the FBG reflected wavelength to shift, compensation circuit are often used to hold the other parameters constant. With the FBG sensor response to the perturbation in wavelength encoded mode, the amount of perturbation can be collated by measuring the amount of shift in the reflected Bragg wavelength with respect to the original transmitted spectrum. The basic setup for a FBG Interrogating system is shown below in Figure 2.12.

Figure 2.12: A setup for the FBG sensor system

Here the interrogator source is from the Broadband Source where the range of wavelength is swept and sent to the FBG via the circulator. The circulator functions as the directional coupler as it allows optical signal to flow from Broadband Source to the FBG while allowing the reflected signal to be transmitted from the FBG back to the Optical Spectrum Analyzer.

From the Bragg Equation (2.17), it can be seen that the reflected wavelength is dependent on the effective refractive index of the core and serve to function as an ambient RI sensor. Any change in the cladding-air interface of the FBG will change the effective refractive index of the core and will cause a shift in the reflected wavelength. There are several studies on improving the sensitivity to FBG RI sensors. One of these methods is to chemically etch the fiber cladding to reduce the cladding thickness. This will allow the fundamental core mode to extend out of the cladding-air interface hence causing the effective refractive index of the fiber to change resulting in a shift of the reflected wavelength. However the challenge here is for an etched SMF to maintain structural integrity; with core diameter of $9\mu\text{m}$ and cladding diameter of $125\mu\text{m}$, etching the cladding from $125\mu\text{m}$ to near $9\mu\text{m}$ will impact the structural strength of the fiber thus making this sensitive FBG sensor unsuitable for use in mechanically harsh environment.

This shortcoming of the FBG can be overcome with the use of the Long Period Gratings. In the LPG, the core modes propagating in the fiber core are

coupled into discrete cladding modes that will travel and get dissipated in the cladding. As the cladding modes penetrates into the medium surrounding the fiber, any change in the refractive index of external medium will result in changes of the cladding modes. This change in the optical characteristic of the LPG can be in the form of wavelength shift in the cladding modes. Decreasing the cladding thickness of the LPG will improve its sensitivity. Because of the inherent sensitivity in LPG, cladding thickness need not be reduced dramatically to improve its sensitivity; hence the structural integrity of the fiber is still maintained, thus making it more durable.

2.2.3 Long Period Gratings

2.2.3.1 Introduction

In this section, the principle of operations of a LPG will be discussed. This will cover the phase matching condition, mode coupling, the LPG characteristics and the coupling coefficient.

2.2.3.2 Phase-Matching Condition

In the LPG, the gratings will cause the transfer of energy between the fundamental core mode and the cladding modes, following the fulfillment of the phase-matching condition. From Equation 2.6, and with $\beta_1 = n_{core}^{eff}(\lambda)$ and $\beta_2 = n_{cl,m}^{eff}(\lambda)$, the phase-matching condition can be written as [33]:

$$\lambda_{ref} = [n_{core}^{eff}(\lambda) - n_{cl,m}^{eff}(\lambda)]\Lambda \quad (2.19)$$

where λ_{ref} is the resonance wavelength, n_{core}^{eff} is the effective refractive index of the core mode and $n_{cl,m}^{eff}$ is the effective refractive index of the m th cladding mode. The wavelength dependence of the effective refractive indices is due to material and waveguide dispersion. Differentiating Equation (2.19) yields [33]:

$$\frac{d\lambda_{ref}}{d\Lambda} = \frac{[n_{core}^{eff}(\lambda) - n_{cl,m}^{eff}(\lambda)]}{\{1 - \Lambda \frac{d[n_{core}^{eff}(\lambda) - n_{cl,m}^{eff}(\lambda)]}{d\lambda_{ref}}\}} \quad (2.20)$$

From Equation (2.20), it can be seen that the resonance wavelength of a cladding mode, as a result of the coupling from the core mode, can be estimated with a particular period. The first step of estimation involves the calculation of the effective refractive indices of the fundamental core mode and the various resonant cladding modes of the fiber for a specific wavelength. A set of grating periods $A^{(m)}$ obtained, that will meet the matching condition, is given by Equation (2.19). This step is repeated for several other wavelengths. The plot is shown in Figure 2.13 and depicts the mode couplings of the core mode to the azimuthally symmetric cladding modes with uniform refractive index perturbations perpendicular to the direction of the propagation [36]. From the plot, a grating period can be chosen to allow mode coupling to take place at the required wavelength. This is known as the LPG characteristic curve, shown in Figure 2.13 [36].

Figure 2.13: LPG characteristic curve of resonant cladding modes for a step Index optical fiber LPG. Lower order modes are to the right [36]

2.2.3.3 Mode Coupling in LPG

The principle of mode coupling in LPG, as proposed by Vengsarkar *et al.* in 1996 is illustrated in Figure 2.14 below [37].

Figure 2.14: Mode conversion of a fundamental core mode into the forward propagating cladding mode in a optical fiber with Long Period Gratings inscribed [37].

The fundamental core mode, LP_{01} travels in the fiber and is perturbed by the presence of the gratings in the core. The difference between the propagation constant of the guided core mode and the phase matching vector of the grating gives rise to the propagation constant of one or more cladding modes at resonant wavelengths. The cladding modes are guided in the cladding and along the cladding-air interface and get attenuated rapidly due to the bends in the fiber and absorption by the cladding material. Hence at the phase-matching condition, the energy of the cladding modes is dissipated at the cladding. This results in the series of resonant dips or bands in the LPG transmission spectrum as shown in Figure 2.8. As the cladding modes wave profile extend out significantly more into the cladding-air interface, any change in the external medium RI surrounding the cladding will affect the resonant wavelength. This is because with the change in the surrounding medium RI, the effective refractive index in the m th cladding modes will be affected, as denoted by the term, $n_{cl,m}^{eff}(\lambda)$ in Equation 2.19. This property makes the LPG as an attractive sensor for refractive index sensing for chemical and biomedical applications.

The β plot below can also be used to illustrate the operating principle of the LPG [37].

Figure 2.15: β -plot of mode coupling mechanism in the LPG [37]

From the β plot shown in Figure 2.15, the forward propagating guided core mode with the propagation constant β_{01} couples light to the azimuthally symmetric cladding modes with propagation constants that falls in the range of $\omega n_3/c < \beta^{(m)} < \omega n_2/c$. $\Delta\beta^{(m)}$ is the differential propagation constant and Λ is the grating period as defined earlier. From the $\Delta\beta^{(m)}$ relationship, the gratings period for a LPG has to be larger than fiber Bragg Gratings. This is normally chosen to be in the range of 100 μm to 1000 μm . In a typical LPG, different cladding modes will satisfy the phase-matching condition with different resonant wavelength. This spectral dependence of the propagation constant of the core and cladding modes result in a highly wavelength-selective response in the LPG as shown in Fig 2.8. Here it shows the coupling of the fundamental core mode to different discrete cladding modes which results in distinct resonance dips in the transmission spectrum. The resonance dips have different peak losses and spectral bandwidth due to the dissimilar coupling coefficient that are functions of the modal overlap. The Figure 2.16 below shows the intensity distribution measured by the near-field technique for a group of cladding modes [38]

Figure 2.16: Near field intensity distribution of $LP_{0,m}$ cladding modes with (a) $m=4$, (b) $m=5$, (c) $m=6$, (d) $m=7$ [38]

For the standard fiber, Corning SMF-28, the differential effective refractive index between the core and cladding is from 10^{-3} to 10^{-2} . For the resonant wavelength to occur in the IR communication wavelength (1520nm to 1620nm), the gratings periods will need to be in the hundreds of micron range. For the LPG, it does not have any reflected spectrum. The only slight reflection is due to Fresnel reflection that occurs at the fiber end as a result of the refractive index mismatch between the fiber and air interface [31].

2.2.3.4 Characteristics Curves of Long Period Gratings

This section will cover a few parameters to have a better understanding of the characteristic curve of the LPG. This curve will depict the coupling of the fundamental mode to the various discrete cladding modes. The affecting parameter, coupling coefficient that is dependent on the refractive index and the modal overlap between the core mode and the cladding modes will also be covered.

From the phase matching Equation (2.19), this can be written as

$$\delta n_{eff} \Lambda = \lambda_{ref} \quad (2.21)$$

where $\delta n_{eff} = [n_{core}^{eff}(\lambda) - n_{cl,m}^{eff}(\lambda)]$

Differentiating with respect to Λ , this can be shown as [39]

$$\frac{d\Lambda}{d\lambda} = \frac{\delta n_{eff} - \lambda \left[\frac{dn_{eff}}{d\lambda} \right]}{(\delta n_{eff})} \quad (2.22)$$

With group index of the core mode ($n_{core, g}$) and the cladding mode ($n_{cl, g}$) defined as follows [39]:

$$n_{core, g} = n_{core}^{eff} - \lambda \frac{dn_{core}^{eff}}{d\lambda} \quad (2.23)$$

$$n_{cl, g} = n_{cl}^{eff} - \lambda \frac{dn_{cl}^{eff}}{d\lambda} \quad (2.24)$$

Equation (2.22) and Equation (2.23), Equation (2.24) can be rewritten as below [39]:

$$\frac{d\lambda}{d\Lambda} = \frac{(\delta n_{eff})^2}{\delta(n_{core, g} - n_{cl, g})} \quad (2.25)$$

The equation above suggest that the slope of the resonant wavelength versus grating period is dependent on the differential effective refractive index, group index of core mode and group index of cladding mode, $n_{eff}, n_{core, g}, n_{cl, g}$ between the core and the cladding modes. These parameters are dependent on the physical properties of the fiber. This wavelength versus gratings period curves are termed as the characteristic curve of an LPG. These curves are dependent on the fiber physical properties where the LPG is inscribed [39].

In order to determine the resonance wavelength of an LPG from the period of the gratings, the spectral dependency of the effective refractive indices of the core and the cladding will have to be determined. The effective refractive indices of the core and cladding are dependent on the material variation of the fiber. A quick technique to determine the core and cladding refractive indices with wavelength, if the Germania concentration in the core is known, is to approximate the refractive index $n(\lambda)$ as a function of the wavelength for a particular material, using the Sellmeier equation [40]

$$n^2(\lambda) = 1 + \sum_{i=1}^M \frac{A_i \lambda^2}{\lambda^2 - \lambda_i^2} \quad (2.26)$$

where A_i and λ_i are material dependent parameters. The parameters in the Sellmeier equation for bulk glass samples of different composition are summarized in Table 2.1 [41] [42].

Composition	A_1	A_2	A_3	$\lambda_1 (\mu m)$	$\lambda_2 (\mu m)$	$\lambda_3 (\mu m)$
Quenched SiO_2	0.696750	0.408218	0.890815	0.069066	0.115662	9.90059
13.5% GeO_2 : 86.5% SiO_2	0.711040	0.451885	0.704048	0.064270	0.129408	9.425478

Table 2.1: Sellmeier parameter for bulk glass samples of different composition [41][42]

For general application, the use of 3 terms ($M=3$) is sufficient to obtain a refractive index resolution up to 10^{-5} . The Table 2.1 listed the first three parameter (A_1, A_2, A_3) of the Sellmeier equation for a 13.5% GeO_2 fiber. With the cladding of most standard optical fiber made of quenched silica and with the core doped with specific concentration of GeO_2 , it is sufficient interpolate the first three terms to estimate the refractive index of the material.

For example, the Sellmeier parameters for a quenched sample of 6.75% GeO_2 and 93.25% SiO_2 can be approximated by averaging the values give in Table 2.1. Figure 2.17 shows the cladding modes from $m=1$ to $m=10$ [37]. The dashed curve is the cut-off wavelength or the minimum wavelength at which coupling can occur to a cladding mode for a given grating period. This cut-off wavelength is calculated for each grating period by using the phase-matching condition and increases with periodicity of the gratings. From the figure, the resonance wavelength can be estimated at values larger than this cut-off wavelength. For a fixed gratings period, the coupling wavelength is a function of the cladding mode order. The wavelength separation between the resonant wavelengths increases with the order of the corresponding cladding modes. Hence using Figure 2.17, one can estimate the resonant wavelength for a given period.

Figure 2.17: Characteristic curves for the first ten cladding modes in SMF-28. The dashed curve corresponds to the cutoff wavelength (λ_{cut}) for different gratings period [41].

From Figure 2.17, it can be observed that for $m > 9$, the slope of the curve approached infinity at the wavelength $> 1500\text{nm}$. This can be explained in terms of the differential refractive index difference between the guided mode and the cladding mode that operates in the positive dispersion region of the spectrum [41]. For $m < 9$, the slope of the curve is positive and termed as the normal region while for $\frac{d\lambda_{res}}{d\Lambda} = \infty$, the slope is termed as the turning point for the m th cladding mode. The wavelength which this occurs is termed the equalization wavelength, λ_0 . Beyond this, the slope becomes negative and the region is termed as the anomalous region [41, 42, 43].

The polarity of the slope will determine the direction of the resonance wavelength shift with a change in the refractive index or the gratings period. In the normal region where $\frac{d\lambda_{res}}{d\Lambda} > \infty$ and with Equation (2.23), the resonance wavelength will shift to the higher value as the core refractive index is increased during the grating fabrication process. On the other hand, the resonant wavelength will shift to a lower value when the effective

refractive index of the m th cladding mode increases. The reverse will occur when $\frac{d\lambda_{res}}{d\Lambda} < 0$ which is in the anomalous region. In the anomalous region, a smaller grating period is required compared to operating in the normal region for the same wavelength shift. Most of the fabrication of LPG is operated in the normal region due to the ease of fabrication of larger grating periods. The amplitude mask used for this project work is designed in the normal region.

2.2.3.5 Coupling Coefficient

For the LPG, the resonant dips have different peak losses due to dissimilar coupling coefficient that are functions of the modal overlap. The LPF characteristic curves described the effect of external influences, such as fabrication process, refractive index of external medium or the changes in the grating period, all of which have an effect on the spectral location of the resonance wavelength in the transmission spectrum. In addition to the resonance wavelength change, the degree of loss in the resonance wavelength dip in the LPG spectrum will also be influenced by external influences which are related to the coupling coefficient [44, 45, 46].

From Equation (2.9), the coupling between two modes travelling in the same direction is a strong function of the detuning ratio $\frac{\delta}{\kappa}$ where δ is the detuning parameter and κ is the coupling coefficient of the gratings. The detuning parameter is dependent on the proximity of the operating wavelength to the phase-matching wavelength of the gratings. The coupling coefficient is a function of the index change and the modal overlap between the guided core mode and the cladding mode over the region of the perturbation, as shown in Figure 2.14. For maximum power transfer, the value of the detuning ratio has to be minimum and ideally tends towards zero.

The coupling coefficient can be defined using the approach proposed by A.W. Snyder and J.D. Love [35]. Let the periodic modulation of refractive index in the core of the optical fiber be represented by the function, $\delta n(z)$. Hence the modified refractive index profile in the fiber core, $n(z)$ can be represented [46]

$$n(z) = n_1 + \delta n(z) \quad (2.27)$$

where n_1 is the unperturbed core refractive index. Assuming a rectangular profile of periodic modulation in refractive index, it can be developed into [39]

$$\delta n(z) = \Delta n \text{rect}\left\{\frac{z}{\Lambda}\right\} \quad (2.28)$$

where Λ is the grating period, Δn is the peak change in the refractive index.

For a 50% duty cycle, the $\text{rect}\left\{\frac{z}{\Lambda}\right\}$ can be denoted as [39]

$$\text{rect}\left\{\frac{z}{\Lambda}\right\} = 1 \quad \text{for } q\Lambda < z < \left\{q + \frac{1}{2}\right\}\Lambda \quad (2.29a)$$

$$\text{rect}\left\{\frac{z}{\Lambda}\right\} = 0 \quad \text{for } \left\{q + \frac{1}{2}\right\}\Lambda < z < \{q+1\}\Lambda \quad (2.29b)$$

where q is an integer

The periodic variation in the index can then be expressed in terms of Fourier series by [39]

$$\delta n(z) = \Delta n \left[A_0 + \sum_{N=1}^{\infty} A_N \cos\left(\frac{2\pi N}{\Lambda} z\right) \right] \quad (2.30)$$

where A_N are the Fourier coefficients. The coupling coefficient κ can be denoted by [48]

$$\kappa = \sqrt{\kappa_{12}\kappa_{21}} \quad (2.31)$$

where κ_{12} and κ_{21} are cross-coupling coefficient and the subscript 1 and 2 represent the guided and cladding modes respectively. The cross-coupling coefficient is a function of the order of the m th mode and is given by [47]

$$\kappa^m = \frac{k |A_n|}{2} \frac{\left| \int_0^a \Delta n E_{core}(r) E_{cl}^m(r) r dr \right|}{\sqrt{\int_0^\infty (E_{core}(r))^2 r dr \int_0^\infty (E_{cl}^m(r))^2 r dr}} \quad (2.32)$$

where $\kappa = \frac{2\Pi}{\lambda^m}$. The $E_{core}(r)$ and $E_{cl}^m(r)$ are the electric field distribution of the fundamental guided core mode and the circularly-symmetric cladding mode of order m respectively.

Defining the overlap integral, η^m as [47]

$$\eta^m = \frac{\left| \int_0^a \Delta n E_{core}(r) E_{cl}^m(r) r dr \right|}{\sqrt{\int_0^\infty (E_{core}(r))^2 r dr \int_0^\infty (E_{cl}^m(r))^2 r dr}} \quad (2.32)$$

which the coupling coefficient can be written as [47]

$$\kappa^m = \frac{\Pi |A_n|}{\lambda^m} \eta^m \quad (2.33)$$

where the coupling coefficient is assumed to be constant across the spectral width of the resonance dip of order m and centered at λ^m . From the equation, the coupling coefficient is directly proportional to the order of the cladding mode, the change in the refractive index in core and the overlap integral. It is however, inversely proportional to the resonant wavelength. With the direct proportion to the overlap integral, the modal distribution of the cladding mode will have a strong influence on the magnitude of the coupling. Hence the

coupling of the guide mode occurs only to circularly-symmetric cladding modes; for other cladding modes, the overlap integral is zero.

With the amplitude mask used in the fabrication of the LPG to be rectangular metallic mask and with a 50% duty cycle, the Fourier series of the rectangular refractive index modulation with a grating period of Λ can be expressed as [47]

$$\delta n(z) = \Delta n \left[\frac{1}{2} + \frac{2}{\pi} \sum_{N=1}^{\infty} \frac{1}{N} \sin\left(\frac{N\pi}{2}\right) \cos\left(\frac{N\pi}{2} z\right) \right] \quad (2.34)$$

Comparing Equation (2.34) with Equation (2.30), the Fourier coefficients ($N > 1$) are given by [47]

$$A_N = \frac{2}{N\pi} \sin\left[\frac{N\pi}{2}\right] \quad (2.35)$$

For $N=1$. The Fourier coefficient is $A_1 = \frac{2}{\pi}$. For $A_2=0$, and for a LPG with a perfect rectangular refractive index modulation of 50% duty cycle, the second order interaction between the guided and the cladding mode do not exist. Thus for first order interaction, this will reduce to [47]

$$\kappa^m = \frac{2}{\lambda^m} \eta^m \quad (2.36)$$

The unit for the coupling coefficient is expressed in per unit length (cm^{-1}) with the overlap integral having a dimension-less units. For an LPG, the refractive index modulation created during the direct-writing process makes the coupling coefficient a function of the transmittance function that is dependent on the amplitude mask parameters and the peak refractive index. Hence to obtain the desired decoupling coefficient, the core must be accounted during the analysis as it will account for the overlap between the core and the cladding modes [47].

2.2.3.6 Transmission of Long Period Gratings

From Equation (2.13), the coupling strength between two modes had been discussed. The coupling ratio C , from one mode to the cladding mode of order m to the initial power in the fundamental guide mode is approximated by [40]

$$C = \frac{P^m(L)}{P_{core}(0)} = \frac{\sin^2[\kappa^m L \sqrt{1 + (\frac{\delta^m}{\kappa^m})^2}]}{1 + (\frac{\delta^m}{\kappa^m})^2} \quad (2.37)$$

where L is the length of the grating, κ^m and δ^m are the coupling coefficient and detuning parameter for the corresponding cladding mode respectively. The detuning parameter at the wavelength λ is defined as [40]:

$$\delta^m = \frac{1}{2}[\beta_{core} - \beta^m - \frac{2}{\Lambda}] = \frac{\pi}{\Lambda}(\frac{\lambda^m - \lambda}{\lambda^m}) \quad (2.38)$$

At the phase-matching wavelength λ^m , Equation (2.38) is 0. For maximum power transfer from the guided mode to the cladding mode, the magnitude of the detuning ratio $\frac{\delta^m}{\kappa^m}$ should be kept at 0. The normalized power T transmitted by the fundamental guided mode through the gratings is given by [40]

$$T = \frac{P_{core}(L)}{P_{core}(0)} = \frac{\cos^2[\kappa^m L \sqrt{1 + (\frac{\delta^m}{\kappa^m})^2}] + (\frac{\delta^m}{\kappa^m})^2}{1 + (\frac{\delta^m}{\kappa^m})^2} \quad (2.39)$$

At the phase-matching wavelength, Equation (2.38) and Equation (2.39) will be reduced to [40]:

$$C = \sin^2(\kappa^m L) \quad (2.40)$$

$$T = \cos^2(\kappa^m L) \quad (2.41)$$

Hence at the phase-matched wavelength, Equation (2.40) gives the maximum value with $C=1$ which is the coupling efficiency and Equation (2.41) gives the minimum power in the transmitted guided mode for a fixed grating length.

From Equation (2.40), the coupling strength $(\kappa^m L)$ is maximum when $(\kappa^m L) = \frac{\pi}{2}$ and will oscillate between 0 and 1, when $(\kappa^m L) < \frac{\pi}{2}$ or $(\kappa^m L) > \frac{\pi}{2}$.

The effect is apparent during the fabrication of LPG such that during the exposure of the UV-laser, the resonant dip in the transmission spectrum increases initially for $(\kappa^m L) < \frac{\pi}{2}$. The loss is at maximum when $(\kappa^m L) = \frac{\pi}{2}$. For

$(\kappa^m L) > \frac{\pi}{2}$, the power in the cladding mode starts to couple back into the

fundamental core mode, leading to an increase of the guided mode transmitted power and a reduction in the coupling ratio. More details on the fabrication process will be discussed in chapter 3.

2.2.3.7 Effects of External Perturbation on Long Period Gratings

Similar to the FBG, the original applications of LPG even till today, is in optical fiber-based devices for optical communications such as EDFA gain equalizer, optical filter, mode coupler, etc. However in the recent years, more focus has been placed in LPG applications for sensing and monitoring. This is due to the sensitivity of LPG to external perturbations such as temperature, strain and refractive index of surrounding medium. This section will discuss the LPG spectral response to temperature, strain and refractive index of surrounding external medium.

2.2.3.8 Effect of Temperature on Long Period Gratings

The dominating effect of temperature change on the transmission spectrum of the LPG is in the shift of the resonance wavelength. Using the chain rule of derivatives, the sensitivity to a temperature T for a grating of length L can be obtained from equation 2.23 [49].

$$\frac{d\lambda_{res}}{dT} = \frac{\delta\lambda_{res}}{d(\delta n_{eff})} \left[\frac{dn_{core}^{eff}}{dT} - \frac{dn_{cl,m}^{eff}}{dT} \right] + \Lambda \frac{d\lambda_{res}}{d\Lambda} \frac{1}{L} \frac{dL}{dT} \quad (2.42)$$

Equation (2.42) shows that there are two factors affecting the temperature sensitivity of the LPG resonance wavelength, namely,

1. Material made of the fiber due to the change in the differential refractive index of the core and cladding arising from the thermo-optic effect.
2. Waveguide contribution due to the change in the grating periodicity due to the thermal expansion of the fiber.

For the material contribution, Bhatia reported that the material contribution is dependent upon the composition of the fiber and the order of the cladding mode [49]. For the same type of fiber, the order of the cladding mode determines if the material contribution dominates. For a fixed resonant wavelength, Equation (2.23) shows that the coupling to a higher cladding mode (lower cladding effective refractive index) requires smaller grating periods. For grating periods greater than 100μm, in standard germanosilicate fiber, the coupling will be to the lower order cladding modes and the material contribution will dominate. On the other hand, the material contribution becomes negligible when core mode is coupled to higher order cladding modes using grating periods that are less than 100μm [49,50].

For the case of waveguide contribution, the magnitude and sign are also dependent on the coupled order of the cladding mode. When the coupling is to the lower cladding modes, $\frac{d\lambda_{res}}{dT}$ will be positive while for the higher cladding modes, it will be negative [49, 50]. Figure 2.18 shows different wavelength shift of four resonance dips of an LPG [49].

Figure 2.18: Different gradients of the slope show that different order of the cladding has different sensitivity to temperature change. The location of the bands A, B, C and D are 1608.6nm, 1332.9nm, 1219.7nm and 1159.6nm, respectively at 31.2°C. The dashed line (E) is the shift for a Bragg grating at 1550nm with a temperature coefficient of 1.3nm/100°C [51]

By appropriate choice of grating period, it is possible to compensate the change due to material contribution with waveguide contribution, and yield a temperature independent LPG sensor. Bhatia has demonstrated a near-temperature insensitive LPG sensor, with grating period of 40 μm , which exhibits a temperature sensitivity of only. 1.8pm/°C [49]. For a normal LPG, fabricated with standard germanosilicate fiber and having grating period around 200 μm to 500 μm , the temperature sensitivity is approximately 30pm/°C [52].

The temperature sensitivity of a LPG sensor is dependent not only on the orders of the cladding mode but also the material of optical fiber used. LPG fabricated on standard germanosilicate fiber exhibits a temperature sensitivity of 3nm/100°C to 10nm/ 100°C. On the other hand, LPG fabricated with B-Ge co-doped fiber exhibits a temperature sensitivity of 275nm/ °C [53]. Hence for a LPG temperature sensor, B/Ge co-doped fiber would be a better choice.

2.2.3.9 External Refractive Index effect on Long Period Gratings

One of the characteristic of LPG is its inherent capacity to measure the refractive index of the external medium without the need to etch away the cladding region to get to the core. The reason is evident from the phase-matching condition of Equation (2.23). It depicts that the effective refractive index of the m th cladding mode is dependent on the difference between the refractive indices of the cladding and the external medium surrounding the LPG perturbation region. Thus under the condition that the external medium refractive index is lower than that of cladding, any changes in the external medium index will cause a change in the spectral location of the LPG's resonant wavelength. The response of the cladding modes to the presence of the external medium can be explained by the evanescent fields of the cladding modes that penetrate beyond the cladding-medium interface.

If the external medium refractive index increases, the effective refractive index of the m th cladding mode increases. According to Equation (2.23), this will render the shift of the LPG resonance wavelength to a lower spectral location, i.e. a blue shift, provided the final refractive index of the external medium is lower than that of cladding [54]. Patrick et al. also showed that changes in the external medium refractive index also affects the depth of the LPG resonant dips and is more prominent when the external medium's refractive index is closer to that of cladding, see Figure 2.19. Figure 2.19 shows that when the external medium's index increases beyond a value of 1.4 and approaches that of cladding, which is approximately 1.46, the depth of the LPG resonance dip reduces drastically. This characteristic of a significant change in the resonance dip with small changes in the external medium index makes the LPG a highly sensitive refractive index sensor. This is valid if the external medium's refractive index falls within the region that is close to the cladding refractive index.

Figure 2.19: Top graph shows the LPG resonance wavelength response to external index change. Bottom graph shows the effect of external index on the peak loss of the LPG resonance dip [52].

$$\frac{d\lambda_{res}}{dn_{ext}} = \frac{d\lambda_{res}}{dn_{cl,m}^{eff}} \left[\frac{dn_{cl,m}^{eff}}{dn_{ext}} \right] \quad (2.43)$$

Equation (2.43) describes the change in the resonance wavelength resulting from the change in external medium refractive index denoted by n_{ext} . This sensitivity is dependent on the order of the coupled cladding mode. Higher index sensitivity can be achieved with higher order of the cladding modes as these modes are less bound to the fiber waveguide itself [50, 52].

The above criterion is possible when the external medium refractive index is lower than that of cladding. In this situation, the phase-matching condition in the LPG maintains its validity. As the external medium refractive index increase beyond that of the cladding, the LPG transmission spectrum responds differently. In the case where the external medium refractive index matches that of cladding, the discrete cladding modes are no longer guided along the fiber. This is evident in the absence of the resonance dip in the LPG transmission spectrum as shown in Figure 2.19 and Figure 2.20.

Figure 2.20: Experimentally measured core mode transmission losses for several refractive indices surrounding the LPG [50]

It has been experimentally demonstrated that, from Figure 2.20, when the external medium refractive index matches that of cladding, $n_{ext} = n_{cl}$ the LPG resonance dip disappears. The reason is that the core will see an infinitely thick cladding region and without the cladding-air interface, there is an absence of guided region and thus the cladding modes cease to exist [50, 53, 54]. In this situation, the cladding modes will be converted to radiation modes due to the lack of total internal reflection at the cladding-medium interface, resulting in a diminutive loss in the transmission spectrum [50, 53, 54].

Another interesting phenomenon appears in the LPG transmission spectrum as the external medium's refractive index increases above that of the cladding. Figure 2.20 depicts the reappearance of the LPG transmission dip and a slight right shift in the spectral location, which is due to the difference in the nature of Fresnel and total internal reflections. Duhem and co-workers suggested that, due to a discontinuity of the indices between the cladding and the external medium, the radiation modes ceased to exist [54]. With this discontinuity, a

part of the cladding mode energy is reflected at the cladding-air interface. This ratio of reflection is dependent on the Fresnel coefficients, which is dependent on the external medium refractive index. Hence the cladding modes that exist in the cladding region will be depicted as attenuated cladding modes or leaky modes because of the absence of total internal reflection but rather Fresnel reflection occurring at the cladding-medium interface [55]. In this case, lacking a confining structure to contain the cladding mode within the cladding region, the phase-matching condition is no longer valid.

At the higher refractive index, the external medium and the cladding form a discontinuity at this interface, which means the cladding is no longer infinite and the radiation mode description becomes invalid. As mentioned earlier, then at the cladding-air interface, the Fresnel reflection coefficients are dependent on the external medium's refractive index. With the external medium's higher refractive index, the portion of energy reflected of the attenuated cladding modes will be higher. These attenuated cladding modes are only able to be guided by the abrupt cladding-air discontinuity if they succeed at being continually amplified down the length of the cladding in the direction of propagation. The difference between the guided cladding modes and the guided attenuated cladding modes is that the latter's resonance wavelength do not shift with the value of the external medium refractive index due to the lack of optical confinement by the external medium.

The effect on the LPG transmission spectrum with different external medium's refractive indices will be characterized by a change in the depth of the resonance dip. It can be observed from Figure 2.20 that when the external medium index increases from 1.4601 to 1.6716, the depth of the LPG resonance dip increases steadily. This is due to the increase in the Fresnel reflection coefficients that yields an improved reflection at the cladding-air interface, resulting in an increase in energy in the attenuated cladding modes. With higher energy attenuated cladding mode, more efficient coupling occurs between guided core mode and the attenuated cladding modes, which explains the increase in depth of the LPG resonance dip with respect to the increase of external medium's index beyond that of cladding [54].

In summary, the concept of mode coupling between the fundamental guided core mode and other modes has been discussed through the use of coupled mode theory. The mode coupling between the fundamental mode and other modes form is the key point in the operational principle for the LPG. For total exchange of energy between two modes, the phase matching condition has to be met. In a LPG, the fundamental forward guiding core mode will be coupled into numerous discrete cladding modes. The cladding modes are dependent on the differential refractive index between the core and cladding modes' effective refractive index and the grating period. This dependency has been discussed using the LPG characteristic curves. Another important parameter to the LPG that has been discussed is the LPG coupling coefficient. The coupling coefficient determines the strength in the exchange of power between the fundamental mode and the cladding modes.

2.2.3.10 Cladding thickness effect on Resonance Wavelength and Sensitivity

The resonance wavelengths of the LPG, λ_{ref} is determined by the phase-matching condition given in (2.23) where

$$\lambda_{ref} = (N_{o1} - N_{om})\Lambda \quad (2.44)$$

with N_{o1} , and N_{om} , as the mode refractive indices of the LP_{01} and LP_{0m} , modes respectively and Λ is the pitch of the grating. In practice,

For the calculation of the mode index of the cladding mode, the fiber core will be treated as a perturbation of the fiber without the core. From perturbation theory, this can be derived into [111]:

$$N_{om}^2 \approx \overline{N}_{om}^2 + \delta N_{om}^2 \quad (2.45)$$

With [111]

$$\delta N_{0m}^2 \approx (n_{core}^2 - n_{cl}^2) \frac{\int_0^a \varphi_{0m}^2(r) dr}{\int_{-\infty}^{+\infty} \varphi_{0m}^2(r) dr} \quad (2.46)$$

where $\psi_{0m}(r)$ is the mode field and n_{core} and n_{cl} are the refractive indices of the core and the cladding, respectively. The mode index for the coreless fiber, N_{0m} can be found from the asymptotic expression [111],

$$u = u_\infty [1 - (1/V)], u \equiv (2\pi / \lambda_0) \rho (n_{cl}^2 - N_{0m}^2)^{1/2} \quad (2.47)$$

To give
$$\overline{N}_{0m}^2 \approx n_{cl}^2 - \frac{u_\infty^2 \lambda_0^2}{4\pi^2 \rho^2} \left[1 - \frac{\lambda_0}{\pi \rho (n_{cl}^2 - n_{ext}^2)^{1/2}} \right] \quad (2.48)$$

where u_α is the m th root of the Bessel function J_0 . With $\psi_{0m}(r) = J_0(u_\alpha r / \rho)$, the step-index fiber can be developed into

$$\delta N_{0m}^2 \approx (n_{core}^2 - n_{cl}^2) \left(\frac{a}{\rho} \right)^2 \left[\frac{J_0^2(u_\infty a / \rho) + J_1^2(u_\infty a / \rho)}{J_0^2(u_\infty) + J_1^2(u_\infty)} \right] \quad (2.49)$$

When the cladding radius is changed from ρ to ρ_0 , the resonance wavelength of the LPG is shifted from λ , to λ_0 . Substituting Equation (2.47) (with the third term ignored) and Equation (2.48) into Equation (2.44) and assuming that N_{01} , is independent of the cladding radius, we obtain an explicit expression for the wavelength shift $\delta \lambda_0 \approx \lambda - \lambda_0$ [111]

$$\begin{aligned} \delta \lambda_0 \approx & \frac{u_\infty \lambda_0^2 \Lambda}{8\pi^2 n_{cl}} \left(\frac{1}{\rho^2} - \frac{1}{\rho_0^2} \right) - \frac{n_{cl} \Delta n \Lambda}{J_0^2(u_\infty) + J_1^2(u_\infty)} \\ & \times \left\{ \left(\frac{a}{\rho} \right)^2 \left[J_0^2\left(\frac{u_\infty a}{\rho}\right) + J_1^2\left(\frac{u_\infty a}{\rho}\right) \right] \right. \\ & \left. - \left(\frac{a}{\rho_0} \right)^2 \left[J_0^2\left(\frac{u_\infty a}{\rho_0}\right) + J_1^2\left(\frac{u_\infty a}{\rho_0}\right) \right] \right\} \quad (2.50) \end{aligned}$$

where $\Delta n = (n_{core}^2 - n_{cl}^2) / 2n_{core}^2$ is the relative index difference between the core and cladding.

From Equation (2.44) and Equation (2.48), with the external index change from n_{ext} to $\overline{n_{ext}}$

$$\delta\lambda_0 \approx \frac{u_\infty^2 \lambda_0^3 \Lambda}{8\pi^2 n_{cl} \rho^3} \left[\frac{1}{(n_{cl}^2 - n_{ext}^2)^{1/2}} - \frac{1}{(n_{cl}^2 - \overline{n_{ext}}^2)^{1/2}} \right] \quad (2.51)$$

From Equation (2.51), with the decrease in cladding radius, the sensitivity increases.

2.3 Development of LPG Sensors

2.3.1 Review of the development of LPG for Ambient RI Sensing

LPG sensors had been deployed extensively in chemical and biomedical applications due to its ambient Refractive Index (RI) sensitivity. In these applications, with the sensitivity of the LPG to the change in the ambient RI, the characteristic in the shift of the cladding modes detected can be used to identify the change in the RI detected. This change in the ambient RI can be attributed to different materials or solutions that the LPG is immersed in. With this attribute, the LPG can also be used as a concentration detector to identify the amount of dilution for a given material/ solution. As an ambient RI sensor, the relatively lower cost of the LPG, compared to the FBG that requires etching for improved sensitivity, the LPG offers as a good sensory platform that can be deployed in many chemical and biological sensing applications [13]. Many different configurations of LPG on a SMF had been developed and papers published [14, 15, 16, 17, 18, 19]. The standard LPGs are direct-written by UV exposure to produce the gratings which couple light from the core mode to various forward propagating cladding modes in the single mode fiber (SMF). As a result, at discrete wavelengths, a series of attenuation bands in the fiber transmission spectra are created. This sensor works on the principle of changes in the wavelength of these forward propagating modes with the variation in the external environment parameters such as ambient RI, temperature or strain.

The development works of the LPG in different configurations, as an RI sensor are as follows:

A multiple LPG sensor system that can simultaneously perform measurements of external refractive- index (RI) and temperature was reported using a sandwiched structure of long-period gratings (LPGs) by A Ping Zhang and team [14].

Figure 2.21: Schematics diagram of the SLPGs and the corresponding mode coupling mechanism [14]

Shown in Figure 2.21, the LPGs were fabricated in the sequence of LPG-A.1, LPG-A.2 and LPG-B. The period of LPG-A.1, LPG-A.2 were selected to be $643\mu\text{m}$ with an estimated center-to-center gratings separation distance of $47.5\mu\text{m}$ separation. LPG-B gratings period was selected to be $428\mu\text{m}$ which caused the loss dip to occur at a longer wavelength region without affecting LPG-A pair. The LPG-A pair with co-directional coupling between the guided mode and a low-order cladding mode function as a temperature sensor with low RI sensitivity, whereas the LPG-B performed as an RI sensing element which couples the guided mode to a higher order cladding mode. The sensitivity of 27.77nm/RIU was reported for this sandwiched coupled sensor [14].

An RI sensor based on the fiber Mach–Zehnder interferometer was realized by concatenating two single-mode fibers tapered separated in two locations and separated by a middle section as reported by Zhaobing Tian and co-worker [15].

Figure 2.22: Structure of the two tapered-type interferometer [15]

In this study, as shown in Figure 2.22, when the second tapered structure follows within centimeters of the first one, the attenuation of the cladding mode in the tapered region is negligible, and the cladding mode energy can be coupled back from the cladding to the core in the second tapered region. Due to the phase difference between the core and cladding modes within the two tapered regions, an MZ interferometer is created after the first taper. The separation of the interference spectrum is dependent on the n_{eff} of the middle section of the fiber. Using this MZ interferometer, a sensitivity of 17.1nm/RIU was reported [15]. In the later study, Zhaobing Tian and co-workers also fabricated another version of Mach–Zehnder interferometers through the alignment of two fibers' cores-offset with improved sensitivity of 33.3nm/ RIU as shown in Figure 2.23 [16].

Figure 2.23: Structure of the core-offset-attenuator-type interferometer [16]

The Michelson interferometer with a single core offset attenuator with a layer of approximately 500nm gold coating was also demonstrated [16]. The performance of the Michelson interferometer showed an improvement with minimum insertion loss of 0.01 dB and maximum extinction ratio of over 9 dB compared to the earlier configuration of concatenating two single-mode tapered fibers with minimum insertion loss of 3 dB and maximum interferometric extinction ratio over 20 dB [16]. The RI sensitivity for this core-offset-attenuator-type interferometer has similar sensitivity to the long period pair Mach-Zehnder interferometric sensor

Tao Zhu and team next reported a fiber Mach-Zehnder interferometer in 2011, which is realized with the use of two cascaded special long period gratings with rotary refractive index modulation (C-RLPFG) [17].

Figure 2.24: Schematic of the C-RLPGs [17]

The CO₂ laser beam was programmed to produce the cascaded long period fiber gratings (C-LPFG) profile, shown in Figure 2.24 in which the two LPFGs are fabricated with identical specifications (370 μ m, 30 periods, respectively) and the center-to-center grating's distance of $L=56.1$ mm (the unperturbed fiber length between the two LPFGs is 45mm) [17]. With a sensitivity of 58nm/ RIU, the sensitivity is 4 times higher than that of M-Z interferometer formed by using normal long period fiber gratings [17].

Recently, there are also several papers on the LPG sensors reported with improved ambient RI sensitivity where the LPG cladding is chemically etched,

the LPG is tilted during fabrication, fabrication of complex LPG claddings structures and the deposition of higher index materials on the claddings of the LPG [20, 21, 22, 23, 24, 25, 26].

To improve the sensitivity, one technique is to reduce the cladding thickness using wet etch, which is a common method. Jinhua Yan and team reported an LPG sensor with improved sensitivity of 262.38nm/ RIU by etching the cladding down to 98.5 μ m using hydrofluoric acid (HF) [20]. His grating device consists of two LPG sections, one of which is post etched by hydrofluoric acid (HF) solution after the formation of the LPG and shows an improved RI sensitivity of 3.6 times over the un-etched LPG [20].

Using similar etching method, Edward Davies and team reported the highest ambient sensor so far, a sensitivity of 506.9nm/ RIU for a post-etched LPG with 81⁰ tilted gratings written in it [21]. In the study, it was estimated that HF acid of 10% concentration will cause a thickness of 0.12 μ m of the cladding to be removed per minute [21].

Chien-Hsing Chen and team demonstrated a long period gratings sensor written into a large mode area photonic crystal fiber, using a CO₂ laser with an improved sensitivity of 167.5nm/ RIU with the gold nano-rods but the sensor is insensitive to temperature change [22].

Jian Yang and team reported a greatly improved LPG of 5980nm/ RIU with the use of a cladding-radius-reduced LPG sensor with a high- RI overlay [23]. The fabrication process of this sensor involved the thinning of the cladding thickness which increases effective refractive index difference between different orders of the cladding modes. As a result, the dependence of the cladding-mode effective index on the ambient RI is enhanced [23].

Edward Davies also reported the study of using coating with higher index materials on reduced cladding thickness to enhance the LPG RI

sensitivity; his work involved the use of sol–gel derived titanium oxide coatings and obtained a sensitivity of 1067.15nm/ RIU for his sensor [24].

Sergiy Korposha reported a LPG with a sensitivity of 1927 nm/ RIU over a RI range of 1.3233–1.4906. This LPG was fabricated with the nano-assembled mesoporous coating of alternate layers of poly (allylamine hydrochloride) (PAH) and SiO₂ nano-spheres for use as a fiber-optic refractometer. Different PAH/SiO₂ film thicknesses were deposited onto LPG with different sensitivity, reaching a maximum of 1927nm/ RIU [25].

X Yu and team reported the highest RI sensitivity of 243 902nm/ RIU with the use of a tunable long-period grating formed in a short length of PCF by use of the periodic mechanical pressure. Here the measurand are infiltrated into the air channels of the treated single mode photonic crystal fiber (PCF) for measurement [26].

From the various development works above, with the use of different configurations of LPG to achieve the sensitive ambient RI sensor, the highest sensitivity of 506.9nm/ RIU was obtained for a post-etched LPG with 81° tilted gratings written in it [21]. The introduction of external coatings of gold nano-rods, sol-gel, SiO₂ nano-particles to the cladding of the LPG further increased the LPG sensitivity [22, 23, 24, 25, 26] but it has other trade-offs, one of which is the increase in the process steps and complexity with the additional materials coated onto the LPG cladding surface.

2.3.2 Review of LPG as sensor for integration into Lab-On-A-Chip LOC

The development work on improving the LPG as a sensitive sensor had been ongoing: A. Iadicicco had reported using chemical etch to improve RI sensitivity; exposing the LPG cladding to a diluted 12% hydrofluoric acid to achieve an etching rate of 20µm/min [27]. The highest sensitivity for the LPG without any introduction of coating on the cladding is reported with a RI sensitivity of 506.9nm/RIU using the 81° tilted LPG, followed by wet-etch [21].

For the LPG coating of gold nano-rods, sol-gel, and SiO₂ nano-particles, wet etch is always used to reduce the cladding thickness, followed by deposition of different nano-based particle. In all these chemical etch processes to improve the sensor RI sensitivity, the etch rate of 20µm/min is high due to the nature of the wet etch process and when used to remove the cladding on a Single Mode Fiber (SMF) of 125µm, it is difficult to control the amount to be etched to the um resolution accurately. As a result, the sensitivity of the LPG which depends on the amount of cladding thickness removed cannot be fabricated accurately to any designed value due to the high etch rate from the wet etch process. This has been a challenge in using the wet etch method to improve the LPG sensitivity.

A summary of the work published on utilizing the different LPG configurations as the ambient RI sensor, the development of unique LPG structure on non-SMF and the deposition of coating for improved sensitivity can be found in Table 2.2 and 2.3 respectively.

Review of LPG as sensitive ambient RI sensor				
1. Different LPG configuration				
S/N	Year	Structure/ Process/ Methodology	RI sensitivity details	Published work
A	2005	Cascaded LPG - low sensitivity but measured RI and temperature effect simultaneously	27.77 nm/ RIU	"Sandwiched Long-Period Gratings for Simultaneous Measurement of Refractive Index and Temperature", <i>A. Ping Zhan, Li-Yang Shao, Jin-Fei Ding, and Sailing He</i> [14]
B	2008	LPG based on fiber Mach–Zehnder interferometer with concatenating two single-mode fiber tapers separated by a middle section	17.1nm/ RIU	"Refractive Index Sensing With Mach–Zehnder Interferometer Based on Concatenating Two Single-Mode Fiber Tapers", <i>Zhaobing Tian, Scott S.-H. Yam, Jack Barnes, Wojtek Bock, Patricia Greig, James M. Fraser, Hans-Peter Loock, and Richard D. Oleschuk</i> [15]
C	2008	LPG utilizing Mach–Zehnder and Michelson interferometers with core-offset	33.3nm/ RIU	"Single-Mode Fiber Refractive Index Sensor Based on Core-Offset Attenuators", <i>Zhaobing Tian, Scott S.-H. Yam, Member, and Hans-Peter Loock</i> [16]
D	2011	Based on a fiber Mach-Zehnder interferometer, with two cascaded special long period gratings with rotary refractive index modulation (C-RLPFG)	58nm/ RIU	"Highly Sensitive Refractive Index Sensor Based on Two Cascaded Long Period Gratings with Rotary Refractive Index Modulation", <i>Tao Zhu, Yanen Fan, Ming Deng, and Yunjiang Rao</i> [17]

Table 2.2: Review of the different LPG-based ambient RI sensors

Review of LPG as sensitive ambient RI sensor				
2. Fabrication of LPG structure on non-SMF or deposition of coating for improved sensitivity				
S/N	Year	Structure/ Process/ Methodology	RI sensitivity details	Paper
A	2007	Two LPG sections, one of which is post etched by hydrofluoric acid solution - measures RI and temperature simultaneously.	262.38 nm/ RIU	"Simultaneous Measurement of Refractive Index and Temperature by Using Dual Long-Period Gratings With an Etching Process", <i>Jinhua Yan, A. Ping Zhang, Li-Yang Shao, Jin-Fei Ding, and Sailing He</i> [20]
B	2009	Tilted fiber grating, (81°) wet-etched for 6 hours	506.9nm/ RIU	"Refractive Index Sensitivity Enhancement of 81° tilted Bragg Gratings by Cladding etching", <i>Edward Davies, Pouneh Saffari, Chengbo Mou, Kaiming Zou and Lin Zhang</i> [21].
C	2009	LPG sensor fabricated in a large mode area photonic crystal fiber. Coated with gold nanoparticle.	167.5nm/ RIU	"Laser-induced long-period fiber grating sensor modified with gold nano-rods", <i>Chien-Hsing Chen, Shan-Chien Chen, Yi-Chun Chen, Hao-Teng Hu, Tai-Huei Wei, Wei-Te Wu, Jian-Neng Wang and Jaw-Luen Tang</i> [22]
D	2007	LPG wet-etch, then overlay with high RI material onto cladding to sense ambient RI	5980 nm/ RIU	"Optimization of Cladding-Structure-Modified Long-Period-Grating Refractive-Index Sensors",

		change.		<i>Jian Yang, Li Yang, Chang-Qing Xu, and Yingfu Li [23]</i>
E	2009	Sol-gel titanium and silicon oxide coatings coated LPG	1067.15 nm/ RIU	“Sol–gel derived coating applied to long-period gratings for enhanced refractive index sensing properties”, <i>Edward Davies, Reeta Viitala, Mikko Salomäki, Sami Areva, Lin Zhang and Ian Bennion [24]</i>
F	2011	LPG coated with a nano-assembled mesoporous coating of alternate layers of poly (allylamine hydrochloride) (PAH) and SiO ₂ nanospheres	1927 nm/ RIU	“Refractive index sensitivity of fibre optic long period gratings with SiO ₂ nanoparticle based mesoporous coatings”, <i>Sergiy Korposha, Seung-Woo Lee*, Stephen W Jamesb and Ralph P Tatamb [25]</i>
G	2008	LPG written using CO ₂ and with measurand infiltrated into the air channels of a single mode photonic crystal fiber (PCF).	243 902 nm/ RIU	“Highly Sensitive Photonic Crystal Fiber-Based Refractive Index Sensing Using Mechanical Long-Period Grating”, <i>X. Yu, P. Shum, G. B. Ren [26]</i>

Table 2.3: Review of LPG structure on non-SMF or deposition of coating for improved sensitivity

Chapter 3: Fabrication of Long Period Gratings

3.1 Fabrication of Long Period Gratings

3.1.1 Introduction

This chapter covers the various techniques used in the LPG fabrication process. These techniques include the use of UV-laser amplitude mask [55], the CO₂ laser [57], the femtosecond laser [58], the electric arcing [59] and the Ion Implantation Fabricated LPG [72]. These techniques had been used to fabricate LPG on different types of optical fibers; single mode fiber (SMF), multimode fiber (MMF), photonics crystal fiber (PCF) and hollow core optical fiber (HOF). With the SMF, for improved photosensitivity, hydrogen-loaded fiber or the doped-SMF has been used. As the LPG produced in the projects here are fabricated using the hydrogen-loaded SMF, a section will be allocated to discuss the problems encountered using this process. The annealing of the hydrogen-loaded LPG fiber to stabilize its optical properties will also be covered.

3.1.2 Fabrication Techniques

There are several techniques, which have been employed in the fabrication of long period gratings for ambient refractive index (RI) sensing. The techniques that will be presented in this section include the amplitude mask technique, the CO₂ laser technique, the femtosecond laser technique, the electric arc technique and the Ion Implantation technique.

3.1.2.1 UV Amplitude Mask Technique

The UV-laser amplitude mask technique has been the most popular technique used for the fabrication of long period gratings. This technique involves direct UV writing onto the fiber via an amplitude mask [55]. Its advantages include low cost, quick setup, in-situ fabrication and ease of monitoring. This is the main technique used for the experiments conducted in this project. In using this fabrication technique, one requirement has to be fulfilled; that is the SMF has to be photosensitive prior to the UV exposure.

Photosensitivity is defined as the property of the photo-conductive or electro-optic material, here refers to the fiber, to exhibit and retain differential

modulation in its core's refractive index as a result of exposure to spatial distributions of intense electromagnetic radiation [60]. Photosensitivity was first discovered in 1978 at the Canadian Communication Research Center by Ken Hill and his co-workers [61], leading to the birth of Fiber Bragg Gratings (FBG).

Several techniques can be used to enhance photosensitivity in a SMF, such as hydrogen loading [62], Germanosilicate fiber co-doped with boron [63], flame brushing [64] and strain application [65]. The most commonly used photosensitive fibers are the hydrogen-loaded (H_2) fiber and germanosilicate fiber co-doped with boron (B/Ge) fiber. H_2 -loaded fiber is less costly and exhibit higher photosensitivity than B/Ge co-doped fiber. The advantage of using B/Ge co-doped fiber for fabricating an LPG is its smaller resonance wavelength drift after laser inscription as compared to an LPG fabricated with H_2 -loaded fiber. The higher resonance wavelength drift in H_2 -loaded fiber is due to the out-diffusion of H_2 molecules immediately after inscription. This phenomenon can be reduced with the use of annealing of the fiber after the UV writing. This problem will be discussed at a later part of this chapter. The fabrication setup using UV-laser amplitude mask technique is shown in Figure 3.1.

Figure 3.1 Setup configuration of amplitude mask technique for the fabrication of LPG [55].

Amplitude masks are fabricated when a rectangular transmittance function is imprinted onto a metal plate using a UV laser. The rectangular function has a duty cycle of 50% and the length of the mask can vary from 1cm to 5cm. For

the LPG writing process, either a pulsed 248nm (KrF Excimer) laser or 244nm (Continuous wave frequency doubled Argon-ion) laser can be used. The portion of the photosensitive fiber to be UV illuminated has to have its acrylic jacket removed, the length of which is dependent on the intended grating length to be fabricated. The reason for the removal of the acrylic jacket is that acrylic absorbs UV and will prevent the UV light from penetrating into the fiber core to create the modulation profile. The stripped fiber will then be placed in front of the amplitude mask, as shown in Figure 3.1. The fiber is supported on both sides of the bare region to prevent bending of the optical fiber. For the in-situ fabrication and monitoring, the light from a Tunable Laser Source (TLS), with a wavelength range between 1520nm to 1620nm, is launched from one end of the fiber while an Optical Spectrum Analyzer (OSA) is connected on the other end of the fiber to monitor the growth of the LPG during UV illumination.

Figure 3.2: Evolution of the LPG using an amplitude mask after various UV exposures time: A to E represent different transmission spectrum after the 1 min exposure was initiated with E having the longest UV exposure [37]

Figure 3.2 depicts the increase of the long period grating resonance dip at the highest wavelength within the spectral range of the optical source (point E). As observed, the resonance dip shifts toward the longer spectral wavelength while the peak loss increases as the UV illumination is continued. The spectral shift can be attributed to the increase in the guided mode effective refractive index

and in the coupling coefficient. This shift of the resonance dip during the writing process is a function of the order of the corresponding cladding mode which the fundamental core mode is coupled to as shown in Figure 3.3. The typical wavelength shift of highest order cladding mode during fabrication process can range from 40nm to 100nm. Additional observation from Figure 3.3 shows that each resonance dip has a different peak loss due to distinct coupling coefficients for the corresponding cladding modes.

Figure 3.3 Transmission spectrum of a long period grating with a period of $A=320\mu\text{m}$. Here the discrete spiky loss dips at 1532nm and 1618nm corresponds to the coupling of the fundamental core mode to discrete cladding modes.

The UV illumination stops when the complete power transfer condition is satisfied for the resonance dip with the longest coupling wavelength displayed in the optical spectrum of the TLS. In other words, this is when the peak loss of the resonance dip reaches its maximum. If the UV illumination does not stop after achieving maximum peak loss, the peak loss will start to decrease, resulting in a reduction of the resonance dip. Experimentally it was found that the duration of the fabrication process ranges from 5mins to 20mins depending

on the type of photosensitive fiber used. A point to note is that if hydrogen-loaded photosensitive fiber is used, the duration of the UV illumination to achieve a similar magnitude of resonance dip will increase with the increased period of the storage of the hydrogen-loaded fiber, after the hydrogen-loading process. This is due to the out-diffusion of the hydrogen atom from the fiber, during storage, making it less photosensitive; hence a longer UV illumination period is required.

A major advantage of the amplitude mask technique is that a number of long period gratings with the same grating period can be fabricated simultaneously. This can be accomplished by placing more than one single fiber in front of the amplitude mask, as shown in Figure 3.4. All the fibers are placed parallel to each other.

Figure 3.4 Setup for batch fabrication of long period grating just by using a simple amplitude mask.

Although only one fiber is monitored during the writing process, if the transverse profile of the UV radiation is assumed invariant and if all the fibers are placed parallel to each other, the imprinted gratings in the batch of fibers can be expected to have similar transmission spectra.

3.1.2.2 CO₂ Laser Technique

An alternative technique for the fabrication of long period grating is to use CO₂ laser, which is a non-photochemical process as compared to UV Amplitude

Mask technique. Davis and co-workers first demonstrated this technique of using CO₂ laser to fabricate LPG in 1998 [57]. In their experiment, a computer-controlled mechanical stage with a translation accuracy of 0.1μm pulls the fiber through a fixture so that it is in alignment with the 145μm Gaussian focal spot of the CO₂ laser beam. The power in the focused beam was 0.4W. A 3.9g weight was attached to the fiber over a pulley to provide a constant tension. As the fiber was moved to each new position by the translation stage, the computer opens a shutter for a programmed pulse duration allowing the CO₂ beam to be incident on the fiber. During this fabrication process, the spectral transmission of the fiber was monitored using a TLS covering the range from 1450nm to 1600nm and monitored with an OSA. A camera mounted over the fiber provides visual verification that energy flux is sufficiently low so that no physical deformation of the fiber would occur. After fabrication, the grating transmission was characterized with the use of a TLS and a power meter.

With the present cost of a CO₂ laser system reduced drastically and the flexibility of fabricating long period grating on standard non-photosensitive optical fiber, this technique has gained popularity. In the amplitude mask technique, which requires a UV laser, photosensitive fiber is required in the fabrication of the LPG. In the CO₂ laser technique, non-photosensitive fiber can be used instead to fabricate LPG as this technique is based on thermal shock and not based on the bleaching of germanium-oxygen-deficient center or GODC band. The wavelength of the CO₂ laser, which is 10.6μm, is highly absorbed by the silica fiber, and converted to heat in the fiber. The heated portion then experiences stress relief and densification in the fiber [57]. One reason that leads to the popularity of this technique is simpler setup required; no amplitude mask is required during the fabrication process and hence will lead to a lower cost of fabrication. In the Amplitude Mask technique, the grating period in the LPG is dependent on the slit period of the amplitude mask. This can result in some inflexibility as one has to change the amplitude mask if another LPG of different grating period is required.

In the CO₂ laser technique, the period of the laser ablation can be programmed by the CO₂ system. It does not require amplitude mask and hence offers lower

setup time in fabricating LPG with different grating periods which will increase the fabrication cost.

Figure 3.5: Schematic setup of the LPG fabrication system using the CO₂ laser [57]

As shown in Figure 3.5, the LPG fabrication system using the CO₂ laser technique, will focus the beam onto the optical fiber through an objective lens. A computer was programmed to control the shutter, which when open, the CO₂ laser will lase and focus onto the bare fiber. During the fabrication process, the translation stage will move by distances matching that of the required grating period and the shutter will open and close accordingly. With the combination of shutter control and lateral motion control, the user is able to fabricate LPG with different grating periods a non-uniform or chirp gratings

One characteristic of a CO₂ fabricated LPG is that the refractive index change across the entire cross-section of the fiber is asymmetric. The reason is that when the CO₂ laser beam is focused on the fiber, with the silica glass having strong absorption around the wavelength of the CO₂ laser, the CO₂ light intensity at the exit side of the fiber, shown in Figure 3.6 is weaker than the light incident on the input side of the fiber. This results in an asymmetric index change across the entire cross-section of the fiber with the refractive index change on the incident side being larger than that on the escape side [115].

Figure 3.6: The distribution of the optical power on the LPG fiber during the CO₂ irradiation which resulted in asymmetrical index change in the fiber [115].

Long-period gratings fabricated by exposure to CO₂ laser pulses have been shown to be exceptionally stable. Band rejection characteristics are unchanged in resonant strength and wavelength even when subjected to temperatures up to 1200°C. These long-period gratings have very long shelf-life. Corresponding UV- induced gratings, on the other hand, would be erased in seconds at these high temperatures. Thus CO₂ laser-induced long-period gratings are excellent candidates for both long-lifetime and high-temperature applications.

One problem associated with LPG fabricated using CO₂ laser is the induced azimuthally asymmetric refractive index change in the fiber cladding due to one-sided exposure. The same cladding mode propagating in the cladding region will experience different refractive index at different cross-sectional cladding region.

This can give rise to polarization-dependent loss (PDL) problem when it is used for optical communication applications, such as making of gain-flattened erbium-doped fiber amplifiers [66]. Despite this drawback, CO₂ fabricated LPG

finds its way into numerous applications both in communication [67] and optical sensing [68].

3.1.2.3 Femtosecond Laser Pulse Technique

In 1999, Yuki Kondo and co-workers reported a novel technique for fabricating LPG using focused irradiation of an infrared femtosecond laser [58]. In this technique, a periodic structure of the refractive index was induced in the core of a single-mode fiber using femtosecond laser which the focused irradiation caused a densification of the glass during irradiation. This resulted in the index change of the fiber core where the laser is focused in. In addition, a non-linear reaction through a multiphoton process and conventional heating by a multiphoton transition can occur during laser irradiation [58].

Figure 3.7: Experimental setup for a LPG fabrication system using the femtosecond laser [58].

The experimental set-up for the fabrication technique is shown in Figure 3.7. The most important advantage with this LPGs fabrication technique is the LPG inherent high resistance to thermal decay below 500°C, which shows that they have superior aging characteristic. By comparison, with the thermal degradation of long-period gratings fabricated using UV light irradiation at temperatures below 100°C, annealing of the grating at an elevated temperature for a short time to remove the unstable part of the UV-induced index, is required for the thermal stability of gratings performance. On the other hand, the LPGs fabricated by focused irradiation of femtosecond laser pulses have thermal stability since the mechanism of the index change induced by the focused irradiation of femtosecond pulses is different from that of the UV-induced index change. In the UV-induced grating using Ge-doped silica glass, the photo-excitation of oxygen-vacancy-defect states forms the GeE' centers, which accounts for the refractive index change [58]. The defects related to GeE' centers relax even at temperatures below 100°C and hence the grating performance degrades thermally. Compared with LPGs fabricated using UV light irradiation, those fabricated by focused irradiation of femtosecond laser pulses have a higher temperature resistance and a superior aging characteristic. This technique does come with its drawbacks such as the requirement of expensive femtosecond laser and the asymmetric refractive index induced across the cross-section of the fiber. Like the LPG fabricated using CO₂ technique, which is a non-photochemical process, LPG fabricated using the femtosecond laser has the similar modal birefringence in the fiber cladding. Hence the LPG fabricated using femtosecond laser has higher PDL comparing to LPG fabricated using UV-laser [69].

3.1.2.4 Electric Arc Technique

A common way for connecting two bare optical fibers together is by fusion arc splicing. G. Rego and co-workers demonstrated the technique of LPG fabrication using electric arc technique [59]. In this technique, LPG gratings are created with the fusion arc sparking over a length of the fiber, which creates the index change and hence the modulation profile that corresponds to the grating period. Electric arc technique is similar to CO₂ laser technique in that local

heating of the fiber takes place in the process of grating inscription by an electric arc discharge. Electric arc fabricated LPG also has comparable thermal stability result with LPG fabricated with CO₂ laser. The main advantage of electric arc technique is its simplicity as compared to the rest of the techniques mentioned above, which requires the use of expensive laser equipment. Using this technique, LPG can be fabricated using any type of optical fiber, because their spectral characteristics will mainly be defined by the intrinsic properties of the silica glass itself.

Figure 3.8: Setup of a LPG fabrication system using the electric arc system [59]

Figure 3.8 illustrates the experimental setup of the electric arc fabrication system. The setup and fabrication process is similar to that of the CO₂ laser technique. The bare fiber is clamped between two fiber holders so that the fiber will be kept straight throughout the process. The fiber holders are mounted on a translation stage, with a 0.1 μ m resolution, with the stage providing the lateral movement required. Instead of a CO₂ laser source, a pair of electrical probe was used to produce the electric arc discharge to create corrugations on the fiber.

The reason for the induced refractive index change in the region of the fiber exposed to electric arc can be explained by the fast local heating-cooling process [59]. The fast local heating-cooling process anneals periodically residual stress in the fiber region, which is exposed to electric arc, and creates

new stress in the fiber. This will result in a change in the glass density of the exposed region of the fiber and hence a change in refractive index. G. Rego also found that the LPG fabricated by electric arc technique exhibits good thermal stability. The LPGs are able to operate linearly up to a temperature of 900°C without any irreversible degradation of their spectra [59]. With such simple and low cost fabrication technique and good thermal stability, the LPGs fabricated by this technique provide a good potential to be used as disposable sensors in high temperature environments.

3.1.2.5 Ion Implantation Fabricated LPG

B. Rout and team reported the ion beam implantation using 2.4 MeV H⁺ ions was used to fabricate long period gratings in multimode optical fibers [72]. The Bragg Gratings was formed in the centre of the core of the fiber. The ion beam was implanted through a mask on the fiber with a period of 1.1mm to produce a periodic a refractive index modulation, of up to 1%, along the axis of the core of the fiber. The mode scrambling, from the fundamental mode, was monitored in situ as the implantation was done by observation of the light transmitted through the fiber as shown in Figure 3.9.

Fig. 3.9: (a) Simulation, based on ion coordinates from SRIM, of 2.4 MeV H⁺ ions implanted in to a silica fiber with a diameter of 120 μm. Dark Indicates higher H ion concentration (b) Setup of the Ion Implantation LPG fabrication [72].

It can be seen that LPG can be fabricated using numerous techniques. The technique that was used in this project is the amplitude mask technique using a KrF Excimer laser which was described in Section 3.1.2.1. It was shown that the fabrication of LPG using hydrogen-loaded fiber involves more steps which include the hydrogen-loading process and the annealing of the LPG after the UV illumination. These two processes will be discussed in the next two sections.

3.2 Challenges with Hydrogen-loaded LPG fiber

It was mentioned earlier that the most common technique for the fabrication of LPG is the UV-laser illumination amplitude mask technique. All the LPG sensors used in this work were fabricated with hydrogen-loaded optical fiber using the UV-laser illumination amplitude mask technique that is described in Section 3.1.2.1. Although the use of hydrogen-loaded fiber is more cost effective method and induced a greater refractive index change when compared to B/Ge co-doped fiber, it was observed that the LPG spectrum is unstable immediately after the fabrication process.

(a)

(b)

(c)

(d)

Figure 3.10: Diagrams show the continual growth of the LPG with the grating period of $450\mu\text{m}$ spectrum immediately after UV-laser illumination has stopped. (a) spectrum immediately after the UV-laser is switched off. (b) spectrum after 2mins. (c) spectrum after 4mins. (d) spectrum after 6mins.

Figure 3.10 depicts the development of the LPG, with the grating period of $450\mu\text{m}$, transmission spectrum minutes after the lasing of UV-laser had stopped. Observations from the figure show that the LPG resonance wavelength continued to shift towards the longer wavelength spectrum after the UV-laser illumination. In addition to the wavelength shift, the coupling strength of the grating continued to increase for the first 4mins and thereafter decreased beyond that. Figure 3.11 depicts the shift of resonance wavelength with respect to time.

Figure 3.11: (a) Shift in resonance wavelength with time after UV illumination. (b) Growth in resonance wavelength dip with time after the UV illumination [70]

From the plot in Figure 3.11, the resonance wavelength shifted linearly with time, while the resonance dip increased for the first 5mins and then decreased after that. The reasons for the observed phenomenon can be due to the diffusion of hydrogen in the core and the cladding in the fiber [56].

After UV-laser illumination, there are clearly defined sections of the hydrogen-loaded fiber that were exposed and unexposed to the UV laser during the inscription of the LPGs as shown in Figure 3.12, which correspond to the refractive index modulation of the LPG.

Figure 3.12: Setup shows the exposed and unexposed region of the fiber during the UV illumination in the LPG fabrication process

The hydrogen concentration in the exposed core section, after the UV illumination was much less than that in the unexposed core section since most of the hydrogen atoms were involved in the reaction that caused the change in refractive index. In addition, the large increase in temperature, because of UV absorption in the core of the fiber, also accelerated the diffusion of hydrogen out of the exposed region. Therefore, this resulted in a large difference in hydrogen concentration between the exposed core region and the unexposed core section and between exposed the core section and the exposed cladding region immediately after UV-laser illumination. In achieved equilibrium, the diffusion of hydrogen from both the unexposed core region and the cladding region to the exposed core region as well as into its surroundings will occur. Since refractive index of the fiber is a function of the hydrogen concentration [56], the increase in hydrogen concentration in the exposed core region due to diffusion will lead to an increase in the refractive index. This will lead to an increase in the amplitude index modulation in the coupling coefficient and hence an increase in the coupling strength of the grating. This explains the increase of the resonance dip for the first 5 minutes after the fabrication process, as shown in Figure 3.11 (b). The increase in the amplitude index

modulation also meant an increase in the effective refractive index of the core, n . In addition, since most of the reaction has taken place in the core of the fiber, as the germanium defects in the cladding region is negligible, the change in the refractive index in the cladding region is negligible and therefore the effective refractive index of the cladding can be assumed to be unchanged. Hence from the phase matching equation, there will be an increase in the resonance wavelength. This explains the observed shift of the resonance wavelength towards longer wavelength minutes after UV-laser illumination stopped as shown in Figure 3.11(a).

This phenomenon can pose potential problem as it will be difficult to fabricate LPGs with the desired characteristics. However for the diffusion of hydrogen out of the hydrogen-loaded fiber, if the LPG is kept at very low temperature prior to UV illumination, the trend of the change of resonance wavelength and resonance dip immediately after UV-laser illumination will be different. One method of stabilizing the LPG spectrum after fabrication is by heat-annealing and UV pre-exposure which is done before the actual LPG fabrication step [71]. In this method, before the writing of LPG, the hydrogen-loaded optical fiber is first pre-exposed to a uniform intensity UV-laser source. Through annealing after the LPG fabrication, the removal of hydrogen molecules will help to reduce the LPG resonance wavelength shift. This is because of the non-existence of hydrogen molecules to drift from the unexposed region to the exposed region in the fiber core after LPG fabrication, which will cause changes in the induced refractive index. Using this method, Hiroyuki Kawano and co-workers (71) found that the LPG resonance wavelength and transmission dip after the completion of the LPG writing process is more stable than with the typical procedure without UV pre-exposure.

3.3 Annealing of LPG

Despite continuous shift of the resonance dip in the LPG spectrum immediately after fabrication, this change will be reduced over time. This happens when the hydrogen concentration in the fiber reaches a state of equilibrium [70]. At equilibrium the hydrogen concentration will be uniform throughout the fiber but

still larger than that in the atmosphere. Thus this concentration imbalance will cause the hydrogen to diffuse out from the fiber into the atmosphere. The diffusion of the hydrogen out of the fiber will then reduce the average refractive index in the core and the cladding and forces the resonance band to move back to the lower wavelength (70). The process of hydrogen diffusing out of the fiber at room temperature is very slow and usually takes hundreds of hours to complete. A prolonged waiting time will not be economical and feasible during commercial production of LPGs.

To accelerate this process of hydrogen diffusion out of the fiber, the LPG fiber has to be placed in an oven and annealed at a high temperature immediately after fabrication. Results have shown the effect of annealing LPG at a temperature of 150°C with the resonance wavelengths stabilized to their final values after a few hours as shown in Figure 3.13.

Figure 3.13: Shift in the resonance wavelength with the annealing at temperature 150°C [37]

There is another reason for annealing the LPG. As the LPGs are used as components in systems, be it communication systems or sensor systems, it might be required to operate at temperatures higher than room temperature. In some cases, where a LPG is used as sensor in an engine for example, the working temperature may be above 100°C. Ergodan and co-workers (73) had shown that for FBG written in germanium-doped fiber, a portion of the UV-

laser-induced refractive index change was unstable and decayed over time depending on the operating temperature. This instability arises from the subtle reduction in the concentration of index-raising defect sites. In an LPG, this decay of a part of the induced refractive index modulation will cause the resonance wavelength to shift further into the shorter wavelength. In addition, the coupling strength of the grating will be reduced. For example, the resonance wavelength of a LPG with maximum coupling strength or resonance dip will reduce with time, resulting in a weaker coupling strength or a shallower dip. Therefore it is important that such unstable defects are removed from the gratings prior to use. Annealing of the LPG helps to remove these defects. The annealing temperature for the LPG has to be higher than the temperature in which the LPG will be operating at in the system. Hence annealing the LPG serves two main purposes – removal of residual hydrogen to make the performance stable and removal of unstable UV sites in the gratings.

For this work, the LPGs were annealed at 80°C to 100°C and over duration of 12 hrs. It was observed that after annealing, the LPGs resonance wavelengths had shifted to the shorter wavelength side. This is attributed to the removal of hydrogen and unstable UV sites which will then cause the reduction of the effective refractive index in the core of the fiber, as predicted using the phase matching equation, $\lambda = (\delta n_{core}^{eff} - \delta n_{cl,m}^{eff})$. In addition to wavelength shift, the coupling strength will be reduced as well. The experimental results plotting the LPG spectra before and after annealing is shown in Figure 3.14.

Figure 3.14: Transmission spectrum of the 320μm LPG (a) before annealing (b) after annealing for 100°C for 20hrs

The shift of the resonance wavelength and the change in the coupling strength of the LPG is dependent on the annealing temperature and duration. A LPG with the desired resonance wavelength of coupling strength can be tuned by controlling the annealing temperature for a fixed duration. On the other hand, annealing at a fixed temperature for a variable duration can also be used to achieve the same effect. Care has to be taken if a LPG is to be fabricated to yield the maximum coupling strength or resonance dip. As the change in the coupling strength follows a sine function, the coupling strength can also decrease through annealing. Hence, the LPG will have to be over coupled such that $\kappa L > \frac{\pi}{2}$ during the fabrication phase. This is to ensure a higher probability of achieving LPG with high coupling strength after annealing.

From the discussion, numerous LPG fabrication techniques have been presented in this chapter. They are UV-laser amplitude mask technique, CO₂ laser technique, femtosecond pulse laser technique and Ion Implantation technique. The most common technique used today is the UV-laser amplitude mask technique, which is also the same technique used to fabricate the LPG used for this project. In addition to the fabrication techniques, the problem experienced pertaining to the stability of LPG using H₂-loaded optical fiber after fabrication was also highlighted and presented. One recommended method of stabilizing the LPG spectrum after fabrication is by UV pre-exposure method. In the last part of this chapter, it was discussed that to achieve long term performance stability, it is recommended that the LPG be annealed at 100°C immediately after the fabrication process. Starting from next chapter onwards, the focus will be on the results and discussions on the two novel LPG-based ambient RI sensors that were developed and characterized. These two novel LPG-based ambient RI sensors developed the Improved Refractive Index Sensitivity Utilizing Long Period Gratings with Periodic Corrugations on Cladding and the Long Period Gratings with Controllable Refractive Index Sensitivity Using the Reactive Ion Etcher Process.

Chapter 4: Improved RI sensitivity utilizing Long Period Gratings with Corrugations on Cladding

4.1 Introduction

In this study, a novel Long Period Gratings (LPG) sensor will be introduced as a sensitive ambient RI sensor. This fiber optical LPG has a unique structure where periodic corrugations are imprinted on the cladding of the LPG. The work here focuses mainly on two main portions; the fabrication of the corrugated LPG as a sensitive ambient RI sensor and the performance characteristics of this sensor. The experimental results show that this LPG structure has good performances in terms of linearity and sensitivity; from literature searches, the sensitivity is the highest compared to the other LPG developed, without any external coatings [14, 15, 16, 17]. Hence it served as the high sensitivity and cost effective sensor developed so far. It can be deployed to collect specimen onsite for characterization in the lab as the corrugations can also serve as the reservoir for the specimen collection to be tested.

There will be six sections pertaining to this chapter. The section 4.2 covers the background behind the development of this corrugated LPG sensor. The section 4.3 gives the use of the CO₂ as the irradiation source. The section 4.4 covers fabrication of the corrugation on the cladding of the optical fiber LPG sensor and this will include the parameters influencing the corrugations on the cladding of the LPG sensor, the Design of Experiments (DOE) and the experimental setup. The section 4.5 focuses on the setup, experimental results and discussions of the LPG as a sensitive ambient RI sensor. The summary in section 4.6 rounds up the fourth chapter.

4.2 Background

In the last few years, Long Period Gratings (LPG) had been established as an effective sensor element for a wide range of external environmental measurement, ranging from strain, bending, and temperature to ambient Refractive Index (RI). Its small feature size, added with its high sensitivity makes it an attractive sensor choice for miniaturization in technology trend [28]. As an ambient RI sensor, the low cost and ease of disposal also offers LPG as

a good sensing platform that can be utilized in many chemical and biological sensing applications [74]. Many papers based on different configurations of LPG had been published [75] [76] [77] [78] [79]. The standard LPGs are direct-written by UV exposure where the gratings couple light from the core mode to various cladding modes in the single mode fiber (SMF). As a result, a series of attenuation bands in the fiber transmission are created at discrete wavelengths. This sensor works on the principle of changes in the attenuation bands with variation in the external environment parameters such as temperature, strain and ambient refractive index. Recently, there are also several papers on improving the RI sensitivity; the chemical etch on the LPG cladding, tilted LPG, fabrication of complex structure and the deposition coating on LPG [80] [81] [82] [83] [84] [85]. Another group of LPG sensors are formed with corrugations fabricated on its cladding. These corrugations are non-periodic due to the inherent nature of the process, and are formed through wet etching [86] [87] [88] [89]. These corrugated sensors had been used in only strain sensing applications. As an ambient RI sensing, the corrugations formed on SMF cladding, using the wet etch technique with the imprint lithography had a sensitivity of only 26.7nm/ RIU [90] which is the highest reported sensitivity for a corrugated-cladding LPG as an ambient RI sensor. The objective of this study is to develop and study the sensitive LPG sensor with corrugations formed on its cladding through the use of the CO₂ laser.

LPG fabrication by the UV radiation is the most common method for gratings fabrication. The LPGs fabricated for this study are created using this method. After the LPGs are fabricated, periodic corrugations are formed on the cladding of the LPG using CO₂ laser. In the UV direct-writing process of the LPG, radiation from the UV laser is incident onto the photosensitive fiber via an amplitude mask to create refractive index modulation in the fiber core. The amplitude mask consists of typical modulation depth of 10^{-4} , period between 100μm to 500μm and length of 2cm to 4cm. With the direct-writing process, the fiber's core index modulation is responsible for the light coupling from the propagating core mode to the co-propagating cladding modes. With the co-propagating cladding modes dissipated along the cladding of the fiber, the transmission spectrum consists of a series of cladding modes that are

dependent on the effective index of the coupled modes and the grating pitch. The phase matching between the core mode and the m th forward-propagating cladding modes is achieved at the resonant wavelength and denoted in equation (1) [91].

$$\lambda_{res} = (n_{core}^{eff} - n_{cl,m}^{eff})\Lambda \quad (4.1)$$

where λ_{res} the resonant wavelength, Λ is the grating period, n_{core}^{eff} and $n_{cl,m}^{eff}$ are the effective refractive indices of the fundamental core mode and the m th cladding mode, respectively.

As an ambient RI sensor, the variations in the ambient RI surrounding the cladding of a LPG can be expressed as [92]

$$\frac{d\lambda}{dn_{ext}} = \frac{d\lambda}{dn_{cl,m}^{eff}} \frac{dn_{cl,m}^{eff}}{dn_{ext}} \quad (4.2)$$

where n_{ext} and $n_{cl,m}^{eff}$ are the refractive indices of the surrounding material and m th effective cladding mode, respectively. The term $dn_{cl,m}^{eff} = dn_{ext}$ is distinct for each cladding mode, and hence, a LPG depends strongly on the order of the coupled cladding modes. For a given LPG and cladding modes, the wavelength shift resulted from the ambient refractive index changes may be positive or negative depending on the local slope of the phase matching curve $d\lambda / d\Lambda$ [93]. As shown in Figure 4.1, the core mode is tightly confined in the fiber core while the cladding modes extends into the cladding-air interface which allows the evanescent wave to react with the ambient medium and results in its sensitivity to ambient RI change. This sensitivity to ambient refractive index changes can be either enhanced or suppressed by manipulating the fiber parameters [94]; the variations in the fiber radius will affect the cladding mode field distributions in the cladding-air interface which will change the optical power related to cladding modes evanescent wave in the external medium.

Figure 4.1: A schematic of the LPG which shows the core mode confined in the fiber core while the cladding modes extends into the cladding-air interface.

From past studies, it had been established that the LPG cladding thickness will affect its sensitivity for use as an ambient RI sensor [20, 21, 27]. With a smaller cladding thickness along the length of the LPG inscribed-fiber, the cladding modes will extend a greater portion into the cladding-air interface. This will allow the evanescent wave to react with the ambient medium, changing the effective refractive index of the cladding and results in its sensitivity to ambient RI change.

In the case of the corrugated cladding, the change in the ambient RI will affect the resonance wavelength. Based on coupled mode analysis, the approximate spectral location for the resonance wavelength associated with the m th cladding mode and the LP_{01} mode in a long period gratings can be estimated by [98]:

$$\delta_{core-cl} + \kappa_{core} / 2 = 0 \quad (4.3)$$

where $\delta_{core-cl}$ is the detuning parameter and can be defined as

$$\delta_{core-cl} \equiv \frac{1}{2} (\beta_{core,01} + \beta_{cl,m} - \frac{2\pi}{\Lambda}) \quad (4.4)$$

with the factor of $\frac{1}{2}$ arising because of the assumption that only the core mode is substantially affected by the increased average index in the core.

To reflect the effect of the corrugated cladding, another term, $\bar{\kappa}_{cl}$, which is the average self-coupling coefficient of cladding mode is added to Equation (4.3) to give

$$2\delta_{core-cl} + \kappa_{core} - \bar{\kappa}_{cl} = 0 \quad (4.5)$$

where

$$\bar{\kappa}_{cl} = \frac{\omega \epsilon_0}{4} \int_{A\alpha} 2n_{ext}^{(0)} \Delta n_{core-cl}(r) e_{core}(r) e_{cl}(r) dA \quad (4.6)$$

where $e_{core}(r)$ is the field due to the core mode, $e_{cl}(r)$ is the field due to the average cladding mode, $\delta_{core-cl}$ is the detuning parameter

Using the definition of detuning parameter, the above resonance condition can also be modified into

$$\lambda_{res} = \frac{\Lambda(n_{core}^{eff} - \bar{n}_{cl}^{eff})}{1 + (\bar{\kappa}_{cl} - \kappa_{core})\Lambda / 2\Gamma} \quad (4.7)$$

where

Λ is the period of the corrugated LPG

n_{core}^{eff} and $\bar{n}_{cl}^{eff}$ are the effective index of the fiber core and averaged effective index of the fiber cladding respectively.

κ_{core} and $\bar{\kappa}_{cl}$ represent the self-coupling coefficient of the core mode and average self-coupling coefficient of the cladding mode respectively.

From Equation (4.7), it can be seen that the resonance wavelength will shift to a shorter wavelength if the averaged effective index of the fiber cladding increases. In this project, instead of a reduced constant cladding thickness along the length of the LPG gratings, the effect of the corrugations on the sensitivity of the LPG as an RI sensor will be studied.

4.3 The Use of CO₂ as the Irradiation Source

The use of laser to remove glass has been a common practice and when this is used, the optical absorption of the material to the laser beam is a significant concern. As glasses absorb laser energy in a way which is dependent highly on the irradiation wavelength, the mechanism of photon absorption plays a key role in laser machining of glass materials. When the wavelength of a laser beam is above 5 μm , laser energy is highly absorbed by glasses, such as borosilicate, fused silica, and soda lime. With the CO₂ laser radiation at wavelength of 10.6 μm , it is strongly absorbed by a Si-O vibration mode which can lead to the depletion of the glass [99].

With the radiation from the CO₂ laser, the deposited thermal energy heats the glass to different temperatures and causes the glass to vaporize, melt, peel off, and crack. Although there are different processes involved in the removal of glass with CO₂ laser, the combination of the laser power and the scanning speed will determine the laser energy delivered to the glass surface and this will affect the glass finishing after the laser irradiation. The total energy deposited on the glass surface per unit area is related to the pulse energy and the number of pulses at the area. Pulse energy is related to the initial output of laser power and number of pulses is a parameter which is determined by the laser beam scanning speed. The combination of the two parameters, the pulse energy and the scanning speed, dictates the energy that will be incident with a single scan of the laser beam. If the incident energy rate of a single scan is defined as the amount of energy incident on the substrate surface per unit area per unit time, it shows the energy transferred by the moving laser along the scanning path, i.e., the incident energy of the laser beam when traversed per unit area in 1s. For the single scan of laser beam, the value of incident energy rate is equal to that of the total incident energy at per unit area in 1s. This incident energy rate may be approximated by dividing the product between pulse energy and pulse frequency of the area scanned in 1s, shown in Equation (4.8). The total incident energy per unit area on the glass surface during a single scan can be calculated as shown in Equation (4.9) [27].

$$R_E = \frac{(W.\tau).K}{2\omega.L_o} \quad (4.8)$$

where,

R_E – Incident Energy rate of a single scan along the beam path, J/ (cm² S).

W - Average power of a single pulse.

τ - Pulse width, i.e., time duration of pulse duty cycle, s.

$(W.\tau)$ -Total photon energy (E_p) of a single pulse, i.e., pulse energy, J.

K - Pulse number in 1 s, i.e., pulse frequency of laser beam, Hz (s⁻¹).

$(W.\tau).K$ - Total incident energy in an area scanned in 1 s, J.

ω - Laser beam radius on the glass surface, cm.

L_o - Scanning distance of laser beam, cm, for 1 s.

$(2\omega.L_o)$ - Area scanned by laser beam, cm², in 1s.

$$E_T = \left[\frac{(W.\tau).K}{2\omega.L_o} \right] \left[\frac{L_o}{v} \right] \quad (4.9)$$

which can be simplified to

$$E_T = \left[\frac{(W.\tau).K}{2\omega.v} \right] \quad (4.10)$$

where

E_T - Total energy deposited per unit area on the glass surface, J/cm².

v - Scanning speed of laser beam, cm/s.

$\frac{L_o}{v}$ - Scanning time of a single scan used to scan a length of L_o , s.

Under the varied incident laser energy, the high absorption coefficient of the CO₂ laser irradiation causes substantial heating of the thin surface layer and its deposition amount determines the degree of the temperature increase at the laser irradiation area. A steady-state model may be used to explain the laser caused thermal heating. In the thermal analysis of laser irradiation of the glass substrate, which is the same material made with the SMF fiber, the following assumptions were made

- (i) The CO₂ laser beam is regarded as a surface heating source.
- (ii) On the surface of the glass without laser heating, the superficial heat irradiation is negligible.
- (iii) The thermal properties of the glass are isotropic and temperature-independent.
- (iv) The heat conduction in the glass substrate is negligible, as the deposited energy is on the thin surface layer and it is far greater compared to heat loss.
- (vi) The convection and thermal radiation in the surrounding environment are considered into the laser energy absorption efficiency of the glass
- (vii) The heating phenomena due to phase changes are neglected.
- (viii) Inertia effects are negligible during stress development.

Based on the above assumptions, the mathematical heat transfer model can be established and expressed by Equation (4.11) [99]

$$Q = (A \cdot L_o \cdot \rho) C_p \Delta T \quad (4.11)$$

where,

Q - Heat added to glass from laser irradiation for 1 s (J). Its value is the same as the energy amount deposited into glass by laser irradiation for 1 s.

A - Vertical plane area; the plane is perpendicular to the laser scanning direction and through beam propagation axis in the absorption layer inside glass, cm².

ρ - Mass density of glass, g/cm³.

$(A \cdot L_o \cdot \rho)$ - Mass of the glass heated by laser irradiation.

C_p - Specific heat of glass, J/(g°C)

ΔT - Temperature rise caused by laser irradiation, °C.

The Figure 4.2 shows the thermal gradient on glass induced by CO₂ irradiation. The strain point represents the point of elastic limit and softening point

represents the point of plastic deformation. With the focused CO₂ irradiation on the glass and the Gaussian temperature distribution in the laser beam, the average temperature in the glass surface layer will be at least above the strain point. As glass is cooled after the irradiation, the inside does not cool as fast as the outside of the glass. The inside stays soften or molten and expands while the outside is starting to get stiff and begins to contract. If the glass is cooled too fast, the expansion and contraction are frozen into place and this creates stress in the glass. The glass will eventually break to relieve this built up stress. For the corrugation on the glass to develop, cracking needs to occur at the region with a temperature between the strain point and the softening point [99].

Figure 4.2: Thermal gradient on glass substrate induced by CO₂ irradiation [99]

Figure 4.3: Optimum energy deposition rate in inducing glass strip peeling [99]

As the laser beam energy distribution can be computed as a Gaussian distribution, the center region of the irradiated area gets heat transferred more rapidly than the other regions. Hence the center region will get the highest power density; it has the highest temperature change and thus will generate the greatest expansion than the other regions of the glass substrate. Conversely upon cooling, the center region will experience the greatest amount of contraction. With the larger magnitude of expansion and contraction actions, the greatest force will occur in the center of the Gaussian distribution region. The bending moments is greater along the length of the glass strip than that across the width; the force on the longitudinal axis of the glass strip will be greater than the transverse axis. Thus laser irradiation leads to the removal of the glass material in the form of continuous strip (laser peeling) and corrugations, which can be formed on glass substrate surface. Thus, from experimental studies shown in Figure 4.3, the energy deposition rate may be used as a guide to facilitate the selection of the laser-induced peeling parameters for corrugation formation on the glass surface [99]. Laser irradiation at whatever combination of laser power and scanning speed, as long as the R_E , Incident Energy rate of a single scan along the beam path from the Equation (4.8) is located in the range 3.0-6.0 J/(cm².s), the temperature rising inside glass would be above the strain point and in the softening region of the glass. As a result, glass strip peeling should be able to occur and corrugations will be formed on the glass surface.

4.4 Fabrication of the Corrugations on the Cladding of the LPG Sensor

The fabrication of the corrugations on the cladding of the LPG covers many steps and is described in the following sections:

- Preparation of the hydrogen-loaded SMF in Section 4.4.1
- Setup of the CO₂ laser system to produce the corrugations on the cladding in Section 4.4.2.
- Fabrication of the corrugations on the LPG. Here the CO₂ parameters to control the corrugations' depth and period are discussed in Section 4.4.3.

4.4.1 Preparation of the hydrogen-loaded SMF

S/N	Processes	Equipment/ material
1	Preparation of hydrogen-loaded SMF	a. Hydrogen Loader b. SMF fibers c. Excimer Laser (Lambda Physik Compex 205)
2	Setup of the CO ₂ laser system	a. Epilog Legend TT 7000 b. Grid graph paper with resolution of 1mm c. Base metal plate
3	Examination of the Corrugations on SMF	a. Microscope (CytoViva) b. SEM (JSM-5600LV)

Table 4.1: Equipment and materials list

The hydrogen loaded SMF was prepared by exposing the fiber to hydrogen with a pressure of 70psi for a period of 12 hrs. This causes the H₂ gas molecules to diffuse the into the fiber core to achieve high UV photosensitivity in the Germanium (Ge) doped fiber. The LPG was then direct-written on the hydrogen-loaded SMF core using the 248nm Excimer laser (Lambda Physik, model Compex 205) via an amplitude mask. The amplitude mask has a specification of 320 μ m period, 2.8cm length. The UV-written LPGs were annealed thereafter. The process has been described in Chapter 3, Section 3.1.2.1.

4.4.2. Setup of the CO₂ laser system

With the aid of both the grid graph paper and the alignment mark on the CO₂ laser, the fibers are aligned parallel to the CO₂ laser ablation path as shown in Figure 4.4. For this project, the CO₂ laser system used in the fabrication of the corrugation on the cladding of the LPG is the Epilog Legend TT 7000. The setup is shown in Figure 4.5. The CO₂ laser output is affected by the scanning speed of the laser and the laser output power and the parameters controlling these are %speed and %power respectively. The total length of the corrugation formed on the cladding is controlled to about 2.8cm which is the length of the LPG. The screen on the CO₂ laser system showing the parameters settings is shown in Figure 4.6.

The CO₂ laser beam has a focused beam diameter of $\sim 30\mu\text{m}$ with the size of focal spot considerably smaller than the diameter of the SMF. Through the use of a computer controlled two-dimensional optical scanner, the focused beam was scanned transversely and longitudinally as instructed by a preprogrammed routing. During the fabrication, the laser beam is firstly scanned transversely across the LPGs, with the power and speed as defined in the program. During scanning, the high-frequency CO₂ laser pulses hit repeatedly on the cladding of the LPG and induce an ablation and cause the corrugations to be formed.

Figure 4.4: Alignment of the LPGs onto the grid graph paper

Figure 4.5: Setup of the CO₂ laser system for fabrication of corrugations onto the cladding of the LPG.

Figure 4.6: Screen settings for power, speed in the Epilog Legend TT 7000 CO₂ laser system

4.3.3 Fabrication of the Corrugated Cladding LPG

The corrugation parameters, namely the period and depth of the corrugations will have an influence in the sensitivity of the LPG. At the site of the corrugations, the evanescent wave will react with a change in the effective refractive index of the cladding. Hence a change in this corrugation's period and depth will affect the effective refractive index of the cladding which will determine the amount of evanescent wave that can react with the ambient RI. The period and depth of the corrugations is shown in Figure 4.7.

Figure 4.7: (a) Corrugations period. (b) Corrugations depth

As the CO₂ laser is used to create the corrugation, the lasers' irradiation power which will impact the corrugation period and depth will be investigated. The Epilog Legend TT 7000 CO₂ Laser has a maximum power of 40W and

maximum scanning speed of 80ips (inches per sec). The ablation power of the laser is set by defining the CO₂ laser scanning speed and laser power and this is controlled by the parameters of %power and %speed in the software of the CO₂ laser system. Hence the tunable parameters are laser power and laser scanning speed. The laser output a low frequency pulsing beam of 10Hz, shown in Figure 4.8 with a spot size of 0.0762mm to 0.127mm and a maximum power of 40W.

Figure 4.8: Low frequency pulsing output from the CO₂ laser head

The scanning speed refers to the pulsating laser beam scanning the length of the surface (to be removed) in 1 sec. A change in the scanning speed changes the period of the corrugations; decreasing the scanning speed will decrease the period between the corrugations. A DOE using a matrix with a combination of different laser power and laser scanning speed was run with the following results to obtain the optimal power and speed for the corrugations with measurable and consistent dimensions. Varying the speed from 30% to 50%, (corresponding to 24ips to 40ips respectively) for power setting of 10% to 50% (corresponding to 4W to 20W respectively), it was observed that the corrugations formed with 40ips (speed) and 12W power have good and measureable corrugations dimensions, as shown in Table 4.2.

S/N	Speed (ips)	Power (W)	Observation of corrugations on SMF cladding after irradiation
1	24	4	SMF shows no periodic corrugations after exposure.
		8	
		12	SMF shows burnt marks in the laser ablation areas and the corrugations has burnt marks. This could be due to the prolonged time of exposure to the laser ablation with the low speed.
		16	
		20	
2	32	4	SMF shows no periodic corrugations after exposure.
		8	SMF shows periodic corrugations after exposure.
		12	
		16	SMF shows lesser burnt marks and the corrugations are not well defined in physical dimension. Again this is due to the low speed of the laser scanning which resulted in prolonged time of exposure to the laser ablation.
		20	
3	40	4	SMF shows no periodic corrugations after exposure.
		8	SMF shows periodic corrugations after exposure.
		12	The corrugations have the best defined corrugations dimension with period of $281.7\mu\text{m} \pm 3.4\mu\text{m}$ and depth of $20.1\mu\text{m} \pm 0.4\mu\text{m}$
		16	SMF corrugations show burnt marks.
		20	

Table 4.2: Matrix table for variation in speed and power with the Epilog Legend TT 7000 CO₂ Laser and its effect on the cladding corrugations in the SMF fibers

With the CO₂ laser irradiation on the LPG fibers' cladding, using 40ips speed and 12W power, the dimensions of the fibers corrugations were estimated using an optical microscope and tabulated in Figure 4.9 with the dimensions shown. The periodic corrugation is elliptical in shape and measured with a dimension of $201.3\mu\text{m} \pm 2.8\mu\text{m}$ by $57.1\mu\text{m} \pm 0.2\mu\text{m}$ and with depth of $20.1\mu\text{m} \pm 0.4\mu\text{m}$. The specimen collected in the corrugations is $23.91\mu\text{m}$ from the core of the fiber. The period between two adjacent corrugations is about $281.7\mu\text{m} \pm 3.4\mu\text{m}$. Figure 4.10 shows the Scanning Electron Microscope (SEM) views.

Figure 4.9(a), (b): Measurement using the microscope: Side and top view respectively

Figure 4.10(a), (b): Using the SEM: Side and top view respectively

Using an optimum setting of 40ips speed and 12W power that yields a corrugation period of $281.7\mu\text{m} \pm 3.4\mu\text{m}$ and corrugation depth of $20.1\mu\text{m} \pm 0.4\mu\text{m}$, a variation in the power and speed setting were selected to create different corrugation dimensions on the LPG cladding, shown in Table 4.3.

Speed (ips)	Power (W)	Dimension of corrugations formed
40	9	Period of $281.7\mu\text{m} \pm 3.4\mu\text{m}$, Depth of $12.3\mu\text{m} \pm 0.5\mu\text{m}$
	12	Period of $281.7\mu\text{m} \pm 3.4\mu\text{m}$, Depth of $20.1\mu\text{m} \pm 0.4\mu\text{m}$
	14	Period of $281.7\mu\text{m} \pm 3.4\mu\text{m}$, Depth of $28.2\mu\text{m} \pm 0.4\mu\text{m}$
32	12	Period of $258.5\mu\text{m} \pm 3.1\mu\text{m}$, Depth of $20.1\mu\text{m} \pm 0.7\mu\text{m}$
40		Period of $281.7\mu\text{m} \pm 3.4\mu\text{m}$, Depth of $20.1\mu\text{m} \pm 0.4\mu\text{m}$
50		Period of $306.5\mu\text{m} \pm 3.7\mu\text{m}$ Depth of $20.1\mu\text{m} \pm 0.6\mu\text{m}$

(a)

(b)

Table 4.3: (a) Variation in the corrugations dimension formed with changes in the power and speed setting of the CO₂ laser. (b) Corrugations period and depth

4.5 RI Sensitivity of the Corrugated LPG

4.5.1 Preparation of the different RI solutions

S/N	Processes	Equipment/ material
1	Preparation, testing of the sensitivity of the Corrugated LPG	a. Stabilized Broadband Source (Model 1RBL-11111-F) b. Optical Spectrum Analyzer (Andro, model AQ6317) with accuracy of $\pm 0.05\text{nm}$. c. Refractometer (RA-130), with accuracy of ± 0.0005

Table 4.4: Equipment and materials list

The mass of salt was measured and then dissolved in a bottle containing a volume of 15ml of water. The RI of the salt solution formed was measured with the Refractometer. The same processes were carried out for different mass of salt dissolved in different bottles containing 15ml of water. Using the relationship of $Concentration_{Molar} = \frac{Mass_{Molar}}{Volume_{Water}}$, the

concentration of the salt solution was computed. The concentrations of salt to achieve the different RI solutions are shown in Table 4.5.

Concentration of salt (%)	Measured RI
6.7	1.3418
11.6	1.3494
14.9	1.3544
18.4	1.3596
25.5	1.3659
28.2	1.3722
33.3	1.3787
40	1.3814

Table 4.5: Concentration of salt in water to achieve the different RI solutions

The correlation shows linearity. However the RI solutions produced with the range of concentration is only available from 1.3418 to 1.3814. The short range here is due to the saturation in the salt that can be dissolved in the water. The linear correlation is shown in Figure 4.11.

Figure 4.11: The correlation between different concentrations of salt solutions with RI

4.5.2 Testing of the sensitivity of the Corrugated LPGs to different RI solutions

The optical fiber containing the corrugated LPG was next terminated with optical connectors. The whole fiber is affixed onto a metal plate so that its position and layout is fixed throughout the test. One end of the LPG is connected to the Stabilized Broadband Source (Model 1RBL-11111-F) while the other end is connect to the Optical Spectrum Analyzer (Andro, model AQ6317), as shown in Figure 4.12. Using the different RI solutions, the wavelength responses of the sensors were measured.

Figure 4.12: Setup of the corrugated LPGs for testing with different RI solutions

It was observed that the resonance wavelength showed a shift with the same ambient RI after the corrugations had been fabricated on the LPG claddings. The corrugated LPGs were next tested with the different RI solutions and it was

also observed that the different cladding modes have different sensitivities; the shifts in different cladding modes with the same change in the ambient RI are different. [95] The highest order cladding mode or resonant wavelength has the most sensitive wavelength shift to the ambient refractive index change. The wavelength responses to different RI solutions in the 1550nm spectrum are shown in Figure 4.13 and Figure 4.14. To demonstrate consistency in the performances of the corrugated LPGs, three LPGs were fabricated with the same CO₂ process and with the corrugation period of $281.7\mu\text{m}\pm 3.4\mu\text{m}$ and depth of $20.1\mu\text{m}\pm 0.4\mu\text{m}$. They were next tested for their wavelength responses to the different RI solutions. The tested the consistency of the LPGs performances and the data were used to compute the error bars in Figure 4.14.

Figure 4.13: The spectral response curve to different RI solutions

Figure 4.14: The wavelength responses of the corrugated LPG (with period of $281.7\mu\text{m} \pm 3.4\mu\text{m}$, depth of $20.1\mu\text{m} \pm 0.4\mu\text{m}$) to different RI solutions

The corrugated LPG is then computed to have a sensitivity of approximately 611nm/ RIU for the RI range from 1.3418 to 1.3814. This improved sensitivity is attributed to the physical proximity of the salt solution to the LPG on the core of the optical fiber; with the corrugations, the salt solution are now collected and measured at $23.91\mu\text{m}$ from the core of the fiber which increases the cladding modes field interaction with the salt solution in the corrugations and this accounts for the increased in its sensitivity to the ambient RI measured. From the small error bars measured and recorded in Figure 4.14, it can be seen that the error measured from the wavelength shift due to changes in the RI is small and will not affect the computed sensitivity of the corrugated LPG sensor.

With the variation of the CO₂ lasers' speed and power, the changes to the dimension of the corrugation were observed as shown in Table 4.3 (a). Here, with a fixed 10Hz pulsating output from the laser head, the scanning speed selected will define the period of the corrugations while the variation in the power will affect the depth of the corrugations. With the corrugation period of $281.7\mu\text{m}$, LPGs with different depth of corrugation, shown in Table 4.3 (a) were tested for their response to the different RI solutions and is shown in Figure

4.15. For the different corrugations' depth, the resonance wavelengths were observed to be $\approx 1596.721\text{nm}$. From the linear fit curves estimation, it can be seen that the corrugations with depth of $12.3\mu\text{m}$, $20.1\mu\text{m}$ and $28.2\mu\text{m}$ has a sensitivity of -595nm/RIU , -611nm/RIU and -614nm/RIU respectively. With the corrugations, the salt solutions are collection nearer to the LPG that is in the core of the optical fiber. It can be observed that the sensitivity of the corrugated LPG increases with the corrugation depth. This is due to the increased in the cladding mode field distributions interaction with the salt solution in the corrugation which makes it more sensitive to the ambient RI change. As the accuracy of the Optical Spectrum Analyzer is tight ($\pm 0.05\text{nm}$; in hundredth of resolution compared to the hundreds nm measured), it will not affect the accuracy of the computed RI sensitivity of the corrugated LPG.

Figure 4.15: Graph of RI vs Wavelength Change for different corrugation depths, keeping the corrugations period constant at $281.7\mu\text{m}$

With the depth of the corrugations fixed at $20.1\mu\text{m}$, the responses of the different corrugation periods to the different RI solutions is recorded as shown in Figure 4.16. From the experiments, it can be seen that the resonance wavelength shifted to 1597.500nm , 1596.721nm , 1595.300nm for the

corrugations period of 258.5μm, 281.7μm and 306.5μm respectively. It can be seen that the changes in the corrugations periods do not have a significant change in the sensors' sensitivities.

Figure 4.16: Graph of RI vs Wavelength Change for different corrugation periods, keeping the corrugations depth constant at 20.1μm

This shift to a lower resonance wavelength for increasing corrugation periods can be explained as follows: From Equation (4.7), it can be seen that with the corrugations induced into the cladding of the LPG, the resonance wavelength will shift to a shorter wavelength as the averaged effective index of the fiber cladding increases with the corrugations introduced. Hence with a larger corrugation period, the averaged effective index will increase and a lower resonance wavelength is produced.

From Equation (4.7)

$$\lambda_{res} = \frac{\Lambda(n_{core}^{eff} - \bar{n}_{cl}^{eff})}{1 + (\bar{\kappa}_{cl} - \kappa_{core})\Lambda / 2\Pi} \quad (4.7)$$

where

Λ is the period of the corrugated LPG

n_{core}^{eff} and $\overline{n}_{cl}^{eff}$ are the effective index of the fiber core and averaged effective index of the fiber cladding respectively.

κ_{core} and $\overline{\kappa}_{cl}$ represent the self-coupling coefficient of the core mode and average self-coupling coefficient of the cladding mode respectively.

The sensitivity of the LPG with regular corrugations on its cladding is higher compared to the reduced cladding LPG that was fabricated using the chemical etch method. For this sensor, the effect of the corrugations has increased its sensitivity to ambient RI with a -611nm/ RIU for a 20.1 μ m depth corrugation. Increasing the corrugations depth will further increase the sensitivity. However the structural strength of the sensor will decrease and the sensor become brittle at the corrugated cladding area if the depth of the corrugation is large. To improve the reliability of this sensor during deployment, the sensor can be mounted permanently onto a housing prior to the CO₂ irradiation. After the corrugations are fabricated, the corrugated-cladding LPG with the housing can be deployed for onsite measurement. This will reduce possibility of the sensor from breaking during transportation.

4.6 Summary

In this chapter, an in-fiber Refractometer sensor with periodic corrugations inscribed onto the cladding of a LPG had been presented. The experimental results show that this LPG structure has good performances in terms of linearity and sensitivity and serve as a highly sensitive and cost effective sensor. The corrugations improve the sensor sensitivity to ambient RI measurement while it simultaneously also functions as a reservoir to collect and hold the specimen for testing. The corrugation parameters that are presented in this chapter are the corrugation period and corrugation depth. The study has shown that the variation of corrugation depth changes the sensitivity of the sensor while the changes in the corrugation period do not have any effect on its sensitivity. On the other hand, the variation of the corrugation depth do not change the resonance wavelength significantly while the changes in the

corrugation period will caused a shift in the resonance wavelength. In addition, this sensor can also be used to measure and identify different level of concentration in specimens. The sensor has the advantages of high sensitivity, small feature size, compact, ease of use and offers potential for use in various chemical and biological sensing applications in the future. With the in-fiber corrugations that serve also as the collection reservoir, it can also be deployed as an onsite sensor to collect specimen for measurement and disposed thereafter due to its low cost.

Chapter 5: Long Period Gratings with Controllable Refractive Index Sensitivity Using the RIE Process

5.1 Introduction

In this study, the reduced-cladding Long Period Gratings (LPG) sensor using the dry etch process from the Reactive Ion Etched (RIE) will be introduced as a sensitive ambient RI sensor. Using the dry etch process on the LPG is unique and novel as it is the semiconductor process used in the fabrication of integrated circuits but had not been used on fiber optics technology before. The reduced-cladding LPGs fabricated are then tested for its sensitivity. The work here focuses mainly on two main portions; firstly, the innovative use of the semiconductor dry etch process to develop the LPG into an ambient RI sensor with its sensitivity precisely controlled and secondly, the performances characteristics of this sensor. The experimental results show that this LPG structure has good performances in terms of linearity and sensitivity and serve as a highly sensitive and cost effective sensor. It also has the advantage of precise sensitivity that can be designed and fabricated accurately using the inherent characteristic of the dry etch process of the RIE.

There will be five sections pertaining to this chapter. The section 5.1 covers the industry practices of using wet etch process to improve the sensitivity of LPG and the motivation to use RIE process for this project. The section 5.2 covers the theory of wet etch process and the RIE process. The section 5.3 focuses on the fabrication of the RIE-treated LPG with the experimental setup of the RIE covered. The section 5.4 covers the results and discussion from the test of the RIE reduced-cladding LPG as an ambient RI sensor. The section 5.5 will give the conclusion of this study.

The theory behind the use of the LPG as an ambient RI sensor had been covered in Chapter 2. As an ambient RI sensor, the LPG has a higher inherent sensitivity due to the cladding modes extending into the cladding-air interface; with the interaction of the forward propagating core mode with the co-propagating cladding modes and the co-propagating cladding modes dissipated along the cladding of the fiber, the transmission spectrum of the LPG consists

of a series of cladding modes centered at resonant wavelengths that depends on the effective refractive index of the coupled modes and the grating pitch. As the core mode is tightly confined in the fiber core while the cladding modes extend into the cladding-air interface, this allows the cladding modes to interact with the ambient medium which leads to the improved LPG's sensitivity to ambient RI change. The sensitivity of an LPG to refractive index changes can be either improved or decreased by manipulating the fiber parameters to obtain the appropriate order of the coupled cladding modes [46]. With variations in the LPG cladding's radius, this will affect the cladding mode field distributions in the cladding-air interface; decreasing the radius allows more cladding mode field distributions to interact in the cladding-air interface which will cause the optical power related to cladding modes evanescent wave to significantly increase in the external medium as shown in Figure 5.1.

Figure 5.1: A schematic of the ion-etched LPG which shows the greater portion of the cladding modes field distribution extends into the cladding-air interface.

The LPG's high sensitivity, coupled with the small feature size and low cost makes it an attractive sensor choice for miniaturization in technology trend [100]. As an ambient RI sensor, the low cost also offers LPG as a good sensing platform that can be utilized in many chemical and biological sensing applications [102]. Many papers on LPGs based on different configurations, fabrication methods or material had been published. Two works on LPG which utilized Mach-Zehnder interferometers were reported with a RI sensitivity of

33.3nm/RIU and 58nm/RIU each; the former one was fabricated with an offset in the adjoining fiber cores while the latter was fabricated with two cascaded special long period gratings [103, 104]. Other LPG fabricated using femtosecond laser based on concatenating two micro air-cavities and the CO₂ laser fabricated symmetrical two-hole single-mode fiber were also reported with a sensitivity of 172.4nm/RIU and 141.67nm/RIU respectively [105, 106]. The LPG fabricated using thermocurable polymers was reported with a sensitivity of 165.3nm/RIU [107].

Recently, there are also several papers published using the wet/ chemical etch to further improve the LPG's RI sensitivity. The chemical etch had been used to reduced the LPG cladding and has an improved sensitivity of 262.38nm/RIU [108]. Et al A. Iadicicco had also reported using chemical etch to improve RI sensitivity; exposing the LPG cladding to a diluted 12% hydrofluoric acid to achieve an etching rate of 20µm/min [109]. Another LPG with a sensitivity of 506.9nm/RIU was reported for the 81° tilted Bragg Gratings, with a chemical etch performed on the cladding for 6 hours after the UV irradiation [110]. In all these chemical etch processes, the etch rate is high and when used to remove the cladding on a single mode fiber (SMF with diameter of about 125µm), it is not easy to control the amount to be etched accurately. As a result, using the chemical etch method, the sensitivity of the LPG which depends on the amount of cladding removed cannot be fabricated to any designed value accurately. This may be a challenge to applications (such as the biomedical) where an accurate sensitivity of the sensor had to be designed and fabricated in order to be integrated to other functionality in the chip. In addition, with the chemical etch process the chemical and its disposal cost are high.

The objective of this study is to interface the LPG as a sensitive sensor into the Lab-On-a-Chip (LOC) where several different laboratory functions can be integrated onto the silicon platform with the advantage of faster analysis and response times, compactness and lower fabrication costs; thus allowing cost-effective disposable chips [110]. The LOC finds applications as a safe platform for chemical, radioactive or biological detection with its integration of functionality and smaller stored energies with smaller fluid volumes collected.

With the LOC utilizing semiconductor processes (deposition, photolithography, ion-etch) which are well developed technologies to fabricate different functionality, the use of ion-etching with the RIE to improve the LPG sensitivity will allows the LPG to be integrated into the LOC as a sensor, fabrication of the LPG can then be carried out simultaneously with other functional devices fabrication on the silicon substrate. This will reduce the fabrication cost.

5.2 Theory

5.2.1 Effect of Cladding thickness on Resonance Wavelength and Sensitivity

From Equation (2.44) and Equation (2.48), with the external index change from n_{ext} to $\overline{n_{ext}}$ a decrease in cladding radius of the LPG will results in the sensitivity increases as shown in Equation (2.51)

$$\delta\lambda_0 \approx \frac{u_\infty^2 \lambda_0^3 \Lambda}{8\pi^2 n_{cl} \rho^3} \left[\frac{1}{(n_{cl}^2 - n_{ext}^2)^{1/2}} - \frac{1}{(n_{cl}^2 - \overline{n_{ext}}^2)^{1/2}} \right] \quad (5.1)$$

A. Ladiccio et. al, [108] published and showed experiments that reduced cladding thickness in LPG affects transmission band spectrum and the RI sensitivity. Shown in Figure 5.2, the LP_{05}^{UV} and LP_{06}^{UV} are the attenuation bands relating to the LP_{05} and LP_{06} cladding modes for the UV-irradiated LPG. The decrease in the fiber diameter results in a lowering of the effective refractive index of the cladding modes and hence in a shift of all the attenuation bands towards a higher wavelength. The reduced cladding thickness also increases its RI sensitivity, as shown in Figure 5.3. This increased in the RI sensitivity is attributed to the increase in the evanescent wave of the cladding modes coupled into ambient medium.

Figure 5.2: Transmission spectrum of the UV-irradiated LPG: Shift in attenuation band with a reduction in cladding diameter [108].

Figure 5.3: Sensitivity versus the fiber diameter for LPG with gratings pitch of $360\mu\text{m}$ for ambient RI of 1.33 [108]

5.2.2 Chemical Etch process

The diluted hydrofluoric acid (HF) is commonly used as the chemical etch process to remove the cladding of the LPG to improve its RI sensitivity. The HF reacts with the SiO_2 to produce the liquid byproduct as shown in Equation (1).

The process flow is shown in Figure 5.4 [112]. However the chemical etch process is difficult to control; one has to control the time where the fiber is exposed to the chemical and in most cases, this can only be controlled to an etch thickness with resolution in the tens of μm range. Hence controlling the rate for chemical etch process is challenging and at most instances, to use the wet etch process to get a designed cladding thickness for a specific sensitivity is difficult.

Figure 5.4: Chemical etch process for removing cladding in a LPG

5.2.3 Reactive Ion Etch process

The RIE process is an established dry etch process used in semiconductor industry. It can be used to etch silicon and silicon dioxide with fluorocarbon plasmas in the integrated circuit manufacturing. Unlike the wet-etch process, the substrate removal is done via a plasma with no chemical/ liquid involved. The etch profile is also anisotropic in nature. As shown in Figure 5.5, the plasma is formed by the application of 13.56MHz radio frequency (RF) signal which ionizes the gas molecules with the stripping of the electrons. The freed electrons are accelerated by the oscillating electric field which creates a large potential difference between the electrodes 1 and 4. This causes the radicals to be accelerated toward the electrode 4 which results in its reaction with the fiber surface and causes etching to occur on the fiber cladding. With the RIE process, a much smaller and precise etching rate can be realized as compared to chemical etch process.

Figure 5.5: A schematic of the RIE system

In the dry-etch process, the formation of the plasma by the RF and gases creates ions and free radicals. The plasma is a partially ionized gas with equal numbers of positive and negative particles. However in its true state, a wide variety of particles also exist in the discharge, such as ions, electrons, radicals, excited species, and various fractured gas molecules created by collisions between electronics and gas molecules or atoms. The ions and electrons are accelerated by the electric field (created by the electrode 1 and electrode 4 in Figure 5.5), and collide with other gas particles and bombard all surfaces. The free radicals react with the SiO_2 to form the byproduct as shown in Figure 5.6.

Figure 5.6: Reaction of the free radicals with the SiO_2 substrate

These radicals are responsible for the dry etching process. As dry etch is anisotropic in nature, to ensure uniform etch over the whole LPG cladding, CF_4

was selected as the reacting gas to achieve isotropic etching [113]. The formation of the radicals from the CF_4 gas is shown in Equation (5.3)

High F/C ratio favors etching which in this case CF_4 was used. As shown in Figure 5.7, this high F/C ratio also increases the isotropic etching in the RIE process which is important to ensure a reduced but uniform cladding on the LPG.

Figure 5.7: The selection of the F/C ratio for dry etch in the RIE [112]

Adding O_2 can increase etch rate and increases selectivity over oxide [112]. They are chemically active and react with the surfaces to produce volatile products.

The chemical reaction in the removal of the cladding of the LPG is shown below where the CO_2 and SiF_2 are the byproducts after the fiber cladding is etched.

During dry etch process of SiO_2 in CF_4 plasma, fluorination reactions take place. Assume that concentration of F atoms in CF_4 plasma increases linearly

over pressure range from 0 to 200 Pa. F atoms from the plasma adsorbed on the surface and react with SiO₂ molecules [112]:

The reaction process is characterized by frequency probability of adsorption:

$$\alpha = s\phi / C \quad (5.6)$$

where C is the concentration of surface molecules ($C = 7.86 \times 10^{18} \text{ m}^{-2}$). This reaction is dependent on the parameters of ion bombardment. Let us assume that O atoms formed are immediately desorbed, SiF₂ molecules are formed according to the following reaction [113]:

The reaction process is characterized by the Frequency Probability of Removal, ω shown in Equation (5.8). The Frequency Probability of Removal, ω is dependent on the frequency probability of desorption (ω_d) and frequency probability of sputtering (ω_s). SiF₂ molecules are desorbed and are sputtered by the incident ions. The Frequency Probability of Removal is described as follows [114]:

$$\omega = \omega_d + \omega_s = V_o \exp\left(-\frac{E_d}{kT}\right) + \left(\frac{YI_o}{C}\right) \quad (5.8)$$

where V_o is the frequency of oscillation of atoms in the solid, E_d is the desorption activation energy of SiOF molecules, Y is the sputtering yield of SiOF molecules, and I_o is the ion flux.

5.3 Fabrication of the RIE-treated LPG Sensor

5.3.1 Preparation of the hydrogen-loaded SMF

S/N	Processes	Equipment/ material
1	Preparation of hydrogen-loaded LPG	a. Hydrogen Loader b. SMF fibers c. Excimer Laser (Lambda Physik Compex 205)
2	Setup of the RIE system	a. Reactive Ion Etcher (RIE) b. Quartz plate
3	Examination and measurement of the reduced cladding thickness of the LPG	c. Microscope (CytoViva) d. SEM (JSM-5600LV)

Table 5.1: Equipment and materials list

The hydrogen loaded SMF was prepared by subjecting the fiber to hydrogen with a pressure of 70psi for a period of 12 hrs in a pressure chamber. This is used to diffuse the H₂ gas molecules into the fiber core at high pressure to achieve high UV photosensitivity in the Germanium (Ge) doped fiber. The LPG was then direct-written on the hydrogen-loaded SMF core using the 248nm Excimer laser (Lambda Physik, model Compex 205) via an amplitude mask. The amplitude mask has a specification of 320µm period, 2.8cm length. The UV-written LPGs were annealed thereafter. The process has been described in Chapter 3, Section 3.1.2.1.

5.3.2 Setup of the RIE system

The RIE chamber is first cycle-purge to clear of any debris that may be present. The LPG fibers are next placed into the RIE as shown in Figure 5.8. The following parameters were set up on the RIE. This parameters had been pre-run before and tested with an average etch rate of 46nm/ min for a SiO₂ fiber.

Chamber Parameters	Ratings
carbon tetrafluoride (CF ₄)	70sccm
Oxygen (O ₂)	5sccm
Radio Frequency (RF)	100W
Chamber Pressure	50mTorr

Table 5.2: Parameters for the dry etch of SiO₂ fiber

Fig. 5.8: Setup of the LPG on the RIE chamber

With the average etch-rate of 46nm/ min, four LPGs were RIE-etched with 0.92 μ m, 2.76 μ m, 4.60 μ m and 6.44 μ m removed from their claddings using a time of 20mins, 60mins, 100mins and 140mins respectively. This would give LPGs with a reduced diameter of 123.28 μ m, 120.74 μ m, 117.90 μ m and 114.06 μ m.

The LPG and RIE-etched LPGs were observed using the Scanning Electron Microscope (SEM). As shown in Figure 5.9a and Figure 5.9b, the RIE-etched LPGs' surface has rough texture after the ion-etch process but it is still good and functional.

Fig. 5.9(a), (b): SEM view of the LPG and RIE-etched LPG respectively

5.4 RI Sensitivity of the RIE-treated LPG

5.4.1 Preparation of the different RI solutions

S/N	Processes	Equipment/ material
1	Preparation, testing of the sensitivity of the RIE-etched LPGs	a. Stabilized Broadband Source (Model 1RBL-11111-F) b. Optical Spectrum Analyzer (Andro, model AQ6317) with accuracy of $\pm 0.05\text{nm}$. c. Refractometer (RA-130), with accuracy of ± 0.0005

Table 5.3: Equipment and materials list

Different volumes of salt were dissolved in a fixed volume of water to achieve different RI solutions, measured with a Refractometer. The concentration of salt to achieve the different the RI solutions has been shown in Table 4.5 of Chapter 4.

The correlation shows linearity. However the RI solutions produced with the range of concentration is only available from 1.3418 to 1.3814. The short range here is due to the saturation in the salt that can be dissolved in the water. The linear correlation is has been shown in Figure 4.11 of Chapter 4.

5.4.2 Testing of the sensitivity of the dry-etched LPGs to different RI solutions

The dry etched LPG has its fiber ends terminated onto optical connectors. The whole fiber is held onto a metal board so that its position and layout is fixed throughout the test. One end of the LPG is connected to the Stabilized Broadband Source (Model 1RBL-11111-F) while the other end is connect to the Optical Spectrum Analyzer (Andro, model AQ6317), as shown in Figure 5.11. Using the different RI solutions, the wavelength responses of the sensors were measured.

Figure 5.10: Setup of the Ion-etched LPGs for testing with different RI solutions

The LPG with outer diameter of $125.50\mu\text{m}$ and RIE-etched LPGs with outer diameters of $123.28\mu\text{m}$, $120.74\mu\text{m}$, $117.90\mu\text{m}$ and $114.06\mu\text{m}$ respectively, were measured. It was observed that the resonant wavelength shifted to a longer wavelength for LPGs with a larger amount of cladding removed during the RIE process. This can be attributed to the decrease in the normalized frequency of the cladding waveguide with the removal of the cladding layer. This causes the propagation constant of the cladding modes to decrease which increases the differences between the core and cladding modes' propagation constants. This explains the resonant wavelength shifting to a longer wavelength for LPGs with larger amount of cladding removed. The resonant wavelength for the LPG and RIE-etched LPGs, with final diameter of $125.50\mu\text{m}$, $123.28\mu\text{m}$, $120.74\mu\text{m}$, $117.90\mu\text{m}$ and $114.06\mu\text{m}$ were recorded to be 1550.11nm , 1553.32nm , 1556.50nm , 1557.90nm and 1560.15nm respectively for RI of 1.3418.

After testing the LPG and RIE-etched LPGs with different RI solutions as shown in Figure 5.12 and using the data with the best fitted curve, the sensitivity of 149.95nm/RIU , 165.88nm/RIU , 175.91nm/RIU , 192.54nm/RIU

and 204.50nm/ RIU were obtained for the 125.50 μm , 123.28 μm , 120.74 μm , 117.90 μm and 114.06 μm respectively. In Figure 5.13, it can be seen that using the RIE, the sensitivity varies linearly with the thickness of etched-cladding using the RIE process. The correlation between the RIE exposure time, the average final fiber thickness and the LPG sensitivity are tabulated in Table 5.5. The insertion loss of the RIE etched-cladding is estimated to be about 2 dB.

Figure 5.11: Wavelength Change (nm) for different cladding thickness of LPGs

Figure 5.12: Linear trend for the different cladding thickness etched with the RIE process against sensitivity

Fiber Type	Etch Rate (nm/min)	Total Time in RIE (mins)	Amount of Cladding etched (μm)	Average Final Diameter of Fiber (μm)	Resonant Wavelength (nm)	Sensitivity (nm/RIU)
LPG	0	0	0	125.50	1550.11	149.95
RIE-etched LPG	46	20	0.92	123.28	1553.32	165.88
RIE-etched LPG	46	60	2.76	120.74	1556.50	175.91
RIE-etched LPG	46	100	4.60	117.90	1557.90	192.54
RIE-etched LPG	46	140	6.44	114.06	1560.15	204.50

Table 5.4: The correlation between the RIE exposure times, the resultant cladding thickness, average fiber thickness and the LPG sensitivity

In comparison of this LPG sensor, the effect of the RIE etched-cladding resulted in an increased in the sensor sensitivity that varies proportionally to the amount of cladding removed. The sensitivity achieved using the RIE etched-cladding is comparable to that obtained with a chemical etched-cladding. As the RIE etch time can be controlled precisely, the amount of cladding removed can be obtained accurately; the result is the structural strength of RIE etched-cladding

sensor is better compared to the sensor that undergoes chemical etch process.

5.5 Summary

In this chapter we have presented the LPG and RIE-etched LPGs with linearity and sensitivity to ambient RI changes that allows the sensor to be designed and fabricated accurately to suit the different applications requirements. This is possible with the smaller etch rate of 46nm/min in the RIE process as compared to the chemical etch process. The RIE process reduced the cladding thickness effectively but do not damage the functionality of the LPG. With the LPG integrated into the Lab-On-A-Chip (LOC), the ion-etching using the semiconductor RIE process can be performed on the LPG to obtain the required sensitivity while simultaneously, fabricating other functional devices onboard the chip. However this will require an additional deposition process of a metal layer to protect other devices that do not requires RIE etching. In addition, the LPG can also be used as a concentration measurement sensor. This sensor offers the advantages of controllable sensitivity using the RIE process, small feature size, ease of use and offers potential for use in various chemical, biological and medical sensing applications in the future.

Chapter 6: Conclusions

6.1 Conclusions

The sensor forms a critical part of the intelligent system where it acts as a measurand for the external stimuli and allows it to be measured and analyzed so that the system can perform specific tasks in relation to the stimuli based on the algorithm programmed. For fiber-based sensors, the Fiber Bragg Gratings (FBG) and the Long Period Gratings (LPG) had been a focus of research lately as it provides several advantages over the electrical based sensor, when used in many applications; the advantages of immunity to EMI, flexibility to allow multiplexing for several sensors to be read simultaneously, low cost and high sensitivity. Comparing between the FBG and the LPG, the latter is the fiber-based sensors that is more suitable for ambient RI sensing as the evanescent field in the LPG penetrates out of the cladding-air interface which makes it more sensitive to ambient RI changes. The LPG sensor works on the principle of changes in the attenuation bands with the variation in the ambient RI. From published works, the highest sensitivity for the LPG, without any deposition is reported with a RI sensitivity of 506.9nm/RIU using the 81° tilted LPG, followed by wet-etch to a reduced cladding of 80µm [21]. Hence the LPG is the subject of this study where it has been developed into two innovative, highly sensitive, low cost ambient RI sensors; namely the LPG with the corrugated cladding and the RIE-treated LPG. The development of these sensors allows it to be deployed in chemical or biological applications where the LPG can be used as a disposable ambient RI sensor.

The first study reviewed the sensitivity of the various developments in LPG as the sensitive sensor and used it as a baseline to develop a higher sensitive LPG-based sensor that is also low cost. The novel LPG sensor, known as the **LPG with Regular Corrugations on its Cladding** had been developed and characterized as follows:

- As the CO₂ laser is used to create the corrugations on the LPG cladding, the lasers' beam power which will impact the corrugation period and depth was studied. Using the CO₂ laser with a variation in its beam power

and speed, different corrugation periods and depths were created and the LPG sensitivity tested with the different RI solution.

- The periodic corrugations are elliptical in shape and measured with a dimension of $201.3\mu\text{m} \pm 2.8\mu\text{m}$ by $57.1\mu\text{m} \pm 0.2\mu\text{m}$, with a period between two adjacent corrugations of about $281.7\mu\text{m} \pm 3.4\mu\text{m}$ and with depth of $20.1\mu\text{m} \pm 0.4\mu\text{m}$.
- The specimen collected in the corrugations is $23.91\mu\text{m}$ from the core of the fiber.
- The corrugated LPG sensor has improved sensitivity of $611\text{nm}/\text{RIU}$, compared to the other LPG sensor when tested with different RI solution ranging from 1.3418 to 1.3814.
- For the LPG with different corrugation depth (but with the corrugation period held constant at $281.7\mu\text{m}$), the corrugations with depth of $12.3\mu\text{m}$, $20.1\mu\text{m}$ and $28.2\mu\text{m}$ induced an ambient RI sensitivity of $595\text{nm}/\text{RIU}$, $611\text{nm}/\text{RIU}$ and $614\text{nm}/\text{RIU}$ respectively. This confirms that for the corrugated LPG sensor, the decrease in the LPG cladding radius will increase the cladding modes field distribution in the cladding-air interface which will increase the optical power related to cladding modes evanescent wave that interacts with the external medium. This accounts for the sensor increased sensitivity with higher corrugation depth.
- With a corrugation period ranges from $258.5\mu\text{m}$, $281.7\mu\text{m}$ to $306.5\mu\text{m}$ (but with the depth of the corrugations fixed at $20.1\mu\text{m}$), it was seen that the resonance wavelength shifted to 1597.500nm , 1596.721nm , 1595.300nm respectively. However, it can be seen that the changes in the corrugation periods do not have a significant effect in the sensor's sensitivities.

The sensor has the advantages of high sensitivity, small feature size, compact, ease of use and offers potential for use in various chemical and biological sensing applications in the future. The corrugations serve also as a reservoir to collect the specimen which allows this sensor to be used in many chemical and biological applications as a low cost sensor that can be deploy as part of the

disposable ambient RI measurement kit. This will allow onsite collection of specimens onto the LPG sensor and thereafter the LPG sensor can transport back to the laboratory for measurement and analysis. Because the improvement in sensitivity is due to the corrugations fabricated on the cladding of the LPG, there is no additional material implanted onto the LPG sensor and hence will not increase the insertion loss of this sensor.

The second study, the **LPG with Controllable Refractive Index Sensitivity** focuses on developing an innovative method to produce a LPG with high ambient RI sensitivity accurately. To improve the sensitivity of the LPG, the common technique is to reduce the cladding thickness using the chemical etch process. However the uses of the chemical etch process will post a challenge with the process control; for a SMF of 125 μm , it will require the etching of the cladding to within a few microns from the core to significantly increase the evanescent field in the cladding modes. Using diluted Hydrofluoric acid (HF) and with an high etch rate that is inherent in the wet etch process, the cladding removal can normally be controlled only to tens of μm resolution. As a result, using the chemical etch method, the sensitivity of the LPG which depends on the amount of cladding thickness removed cannot be fabricated accurately to any designed value to suit the specific application. The use of RIE to etch the claddings of LPG to achieve accurate sensitivity was conducted with the following findings:

- In the RIE process, the formation of the plasma by the RF and gases creates ions and free radicals. These radicals are responsible for the dry etching process. As dry etch is anisotropic in nature, to ensure uniform etch over the whole LPG cladding, CF_4 was selected as the reacting gas to achieve isotropic etching.
- Using the RIE process with an etch rate of 46nm/ min, RIE-etched LPGs with final diameters of 123.28 μm , 120.74 μm , 117.90 μm and 114.06 μm were fabricated successfully with the resonant wavelength recorded to be 1553.32nm, 1556.50nm, 1557.90nm and 1560.15nm respectively.
- Tested with different RI solution, ranging from 1.3418 to 1.3814, the sensitivity of 149.95nm/ RIU, 165.88nm/ RIU, 175.91nm/ RIU, 192.54nm/

RIU and 204.50nm/ RIU were obtained for the 125.50 μ m (un-etched LPG), 123.28 μ m, 120.74 μ m, 117.90 μ m and 114.06 μ m respectively. It can be seen that using the RIE, the sensitivity varies linearly with the thickness of etched-cladding using the RIE process.

The RIE can be used to develop the LPG as a sensitive sensor to be interfaced into the Lab-On-a-Chip (LOC). Together with the RIE, deposition, photolithography and etch, several different laboratory diagnostic subsystem can be fabricated simultaneously and integrated onto the silicon platform with the advantage of faster analysis and response times, compactness and lower fabrication costs; thus allowing cost-effective disposable chips. The LOC finds applications as a safe platform for chemical, radioactive or biological detection with its integration of functionality and smaller stored energies with smaller fluid volumes collected. From literature searches, there had been no reports in the used of semiconductor technology on fiber optics as they belong to different technology areas. However with this developmental work, it will be possible to design, fabricate the LPG sensor accurately with the use of the RIE process. The same RIE process can simultaneously be used to fabrication other functional subsystem for the LOC applications.

The two LPG developed are unique and suit different applications. The LPG with Corrugation on it Claddings function as a sensitive and mobile sensor that can be deployed for biological or chemical applications. Its high sensitivity of 611nm/ RIU makes it a suitable choice for detecting ambient RI. The RIE-treated LPG, on the other hand will be a suitable choice for deployment as the sensor on the LOC. With the successful use of the RIE process to etch the cladding of the LPG to achieve improved sensitivity, the different semiconductor processes (such as photolithography, RIE and deposition) can be used to fabricate functional subsystem on the LOC while the RIE process can simultaneously be used to fabricate the sensitivity of the LPG to the required designed value.

6.2 Recommendation for Future Research

6.2.1 LPG sensors

To further improve the LPG sensors developed, the following suggestions are recommended:

6.2.1.1 Theoretical work on the corrugated long period gratings as an ambient RI sensor.

Although the theoretical modeling of the corrugated long period gratings had been studied, the relations (in Chapter 4, Equation 4) only estimate with an average effective index of the fiber cladding which will not give an accurate resonance wavelength shift. In the corrugated LPG, the corrugations are periodic. Hence to achieve a better understanding and to allow more accurate prediction of the effect with the periodic corrugation on the resonance wavelength and the sensor sensitivity, a theoretical study is therefore suggested that will take into account the corrugation shape, its period and depth.

6.2.1.2 Compensation for temperature effect on the corrugated long period gratings.

The change in the ambient temperature where the two LPG sensors developed are used will affect the peak loss of the resonance wavelength change in the LPG spectrum and the sensor sensitivity. Hence the results presented in the Chapter 4 and Chapter 5 will contain the temperature change effect. The ambient temperature will affect the corrugation parameters, such as the depth and period; these changes in the corrugation depth and period will have an impact on the performance of the LPG, as shown in Chapter 4. It is therefore recommended to design and fabricate the LPGs with temperature compensation in the material and configuration (i.e. corrugations) used. With this, a temperature independent LPG can be achieved that solely measures the ambient RI only.

6.2.1.3 Development of the LOC with the LPG integrated into the chip.

With the goal to integrate the LPG onto a LOC as the sensitive sensor, other laboratory functions, such as the laser source, optical waveguide, optical spectrum analyzer can be fabricated and integrated onto the silicon platform using the semiconductor processes such as the photolithography, deposition, RIE. Hence it is recommended that the design, fabrication and integration of the various devices on the silicon platform to be studied and developed next so that cost-effective and disposable LOC which has the advantage of faster analysis and response times, compactness and lower fabrication costs can be realized and commercialized.

6.2.2 Microfiber sensor

There has also been development of the optical microfiber as an ultrahigh sensitive RI sensor. Wen Bi and team reported a maximum sensitivity of 18681.82nm/RIU through the use of a nonadiabatic microfiber sensor that has a tapered diameter of only 4.61 μ m [116]. The microfiber sensor will be studied and developed as an alternative fiber optic sensor.

References

- [1] Eric Udd, "Fiber Optic Sensors – An Introduction for Engineers and Scientists", John Wiley & Sons Inc, 1991
- [2] Optical Sensor and their application, Gaurav Puri, Electrical Engineering, Buffalo, gapuri@buffalo.edu
- [3] Bates, Regis J, Optical Switching and Networking Handbook, New York: McGraw-Hill, 2001
- [4] Wolfgang Ecke, Ines Latka, Reinhardt Willsch, Arnd Reutlinger and Roland Graue, "Fiber Optics Sensor Network for Spacecraft Health Monitoring," Meas. Sci Technology, 12, pp974-980, 2001
- [5] Kin-tak Lau, Chi-Chiu Chan, Li-Min Zhou, Wei Jin, "Strain monitoring in composite-strengthened concrete structures using optical fiber sensors," Composites: Part B, 32, pp33-45, 2001
- [6] C Fernandez-Valdivielso, IR Matias, FJ Arregui, "Simultaneous measurement of strain and temperature using a Fiber Bragg Gratings and a thermachromic material," Sensors and Actuators A, 101, pp 107-116, 2002
- [7] P. Chojnowski and KP Jedrzejewski, "Strain sensor applications of Fiber Bragg Gratings," Proceedings of SPIE Vol 5948, 2005
- [8] W. Liang, Y Huang, Y Xu, RK Lee and A Yariv, "Highly sensitive Fiber Bragg Gratings refractive index sensors," Applied Physics Letters, 86, pp 1-3, 2005
- [9] Jun Cong, Xianmin Zhang, Kangsheng Chen, Jian Xu, "Fiber Optics Bragg Gratings sensor based on hydrogels for measuring salinity," Sensors and Actuator B, 87, pp 487-490, 2002
- [10] A Trouillet, E Marin, C Veillas and H Gagnaire, "Optical fiber sensors for hydrogen leak detection," presented at 2005 AIChE Spring National Meeting, Conference Proceeding, 2005.
- [11] Wei Liang, Yanyi Huang, Yong Xu, Reginald K Lee and Amnon Yariv, "Highly sensitive Fiber Bragg Gratings refractive index sensors" Appl. Phys. Letter, 86, 151122 (3 pages), 2005
- [12] A Iadicicco, A Cusano, A Cutolo, R Bernini and M Giordano, "Thinned Fiber Bragg Gratings as highly sensitive refractive index sensor," IEEE Photonics Technology Letters, 16, pp1149-1151, 2004
- [13] Alexis Méndez, MCH Engineering, LLC, "The 14th Optoelectronics and Communication Conference", Jul 2009.
- [14] A. Ping Zhang, Yang Shao, Jin-Fei Ding, and Sailing He, "Sandwiched Long-Period Gratings for Simultaneous Measurement of Refractive Index and Temperature", IEEE Photonics Technology Letters, 17 (11), 2005

- [15] Zhaobing Tian, Scott S.-H. Yam, Jack Barnes, Wojtek Bock, Patricia Greig, James M. Fraser, Hans-Peter Loock, and Richard D. Oleschuk, "Refractive Index Sensing With Mach–Zehnder Interferometer Based on Concatenating Two Single-Mode Fiber Tapers", *IEEE Photonics Technology Letters*, 20 (8), 2008.
- [16] Zhaobing Tian, Scott S.-H. Yam and Hans-Peter Loock, "Single-Mode Fiber Refractive Index Sensor Based on Core-Offset Attenuators," *IEEE Photonics Technology Letters*, 20 (16), 2008
- [17] Tao Zhu, Yanen Fan, Ming Deng, and Yunjiang Rao, "Highly Sensitive Refractive Index Sensor Based on Two Cascaded Long Period Gratings with Rotary Refractive Index Modulation", *21st International Conference on Optical Fiber Sensors*, *Proc. of SPIE Vol. 7753*, 77539M, 2011
- [18] Juan Kang, Xinyong Dong, Mengchao Li, Zaixuan Zhang, Shangzhong Jin. "Refraction Index Measurement Using Long Period Grating Fabricated by Symmetrical-Hole Fiber", *Passive Components and Fiber-based Devices VII*, *Proc. of SPIE-OSA-IEEE Asia Communications and Photonics*, *SPIE Vol. 7986*, 79860X.
- [19] Yang, Jinpeng, Jiang, Lan; Wang, Sumei; Chen, Qianghua; Li, Benye; Xiao, Hai, "Highly sensitive refractive index optical fiber sensors fabricated by a femtosecond laser", *Photonics Journal*, *IEEE*, 3(6), 2011.
- [20] Jinhua Yan, A. Ping Zhang, Li-Yang Shao, Jin-Fei Ding, Sailing He, "Simultaneous Measurement of Refractive Index and Temperature by Using Dual Long-Period Gratings With an Etching Process", *IEEE Photonics Technology Letters*, 17 (11), 2005.
- [21] Edward Davies, Pounesh Saffari, Chengbo Mou, Kaiming Zou and Lin Zhang "Refractive Index Sensitivity Enhancement of 81° tilted Bragg Gratings by Cladding etching", *20th International Conference on Optical Fibre Sensors*, *Proc. of SPIE Vol. 7503*, 75036X.
- [22] Chien-Hsing Chen, Shan-Chien Chen, Yi-Chun Chen, Hao-Teng Hu, Tai-Huei Wei, Wei-Te Wu, Jian-Neng Wang and Jaw-Luen Tang, "Laser-induced long-period fiber grating sensor modified with gold nano-rods", *CLEO/ Pacific Rim* 2009
- [23] Jian Yang, Li Yang, Chang-Qing Xu, and Yingfu Li, "Optimization of Cladding-Structure-Modified Long-Period-Grating Refractive-Index Sensors", *Journal of Lightwave Technology* 25(1), 2007.
- [24] Edward Davies, Reeta Viitala, Mikko Salomäki, Sami Areva, Lin Zhang and Ian Bennion, "Sol–gel derived coating applied to long-period gratings for enhanced refractive index sensing properties", *J. Opt. A: Pure Appl. Opt.* 11, 2009.

- [25] Sergiy Korposha, Seung-Woo Leea, Stephen W Jamesb and Ralph P Tatamb, "Refractive index sensitivity of fibre optic long period gratings with SiO₂ nanoparticle based mesoporous coatings", 21st International Conference on Optical Fiber Sensors, Proc. of SPIE Vol. 7753, 77539A.
- [26] X. Yu, P. Shum, G. B. Ren, "Highly Sensitive Photonic Crystal Fiber-Based Refractive Index Sensing Using Mechanical Long-Period Grating", IEEE Photonics Technology Letters, Vol. 20, No. 20, 2008
- [27] A. Iadicicco, S. Campopiano, A. Cutolo, M.L. Korwin-Pawlowski, W.J. Bock, A. Cusano, "Refractive index sensitivity in thinned UV and arc induced Long-Period Gratings: A Comparative study", International Journal on Smart Sensing and Intelligent Systems, 1 (2), 2008.
- [28] "Sensor Trends 2014 – Trends in Future-Oriented Sensor Technologies", Association for Sensor Technology, Europe, 2010. http://ftp.ama-sensorik.de/Trendanalyse/AMA_Study_Sensor_Trends.pdf
- [29] Eric Udd, "Fiber Optic Sensors – An Introduction for Engineers and Scientists", John Wiley & Sons Inc, 1991
- [30] Gerd Keiser, "Optical Fiber Communications", McGraw Hill, 2000, sec 2.3.2
- [31] A.M. Vengsarkar, P.J. Lemaire, J.B. Judkins, V. Bhatia, J.E. Spie and T.E. Ergodan, "Long-period fiber gratings as band rejection filters", J. of Lightwave Technology, 14, pp 58, 1996
- [32] M. Born, E. Wolf, "Principle of Optics", New York, Pergamon, 1987, sec 8.6.1 eq (8)
- [33] H.F. Taylor, A. Yariv, "Guide Wave Optics", Proceedings of IEEE, 62, pp 1044, 1974
- [34] F. Bilodean, K.O. Hill, B. Malo, D. Johnson, I. Skinner, "Efficient narrowband LP₀₁ ↔ LP₀₂ mode converters fabricated in photosensitive fiber: Spectral response", Elect. Letter, 27, pp 682-684, 1991
- [35] Andreas Othonos, Kyriacos Kalli, "Fiber Bragg Gratings: Fundamentals and Applications in Telecommunications and Sensing", Artech House, 1999.
- [36] T.W. MacDougall, S. Pilevar, C.W. Haggans, M.A. Jackson, "Generalized Expression for the Growth of Long Period Gratings", IEEE Photonics Technology Letters, 10, pp 1449-1451, 1998
- [37] A.M. Vengsarkar, P.J. Lemaire, J.B. Judkins, V. Bhatia, J.E. Spie and T.E. Ergodan, "Long-period fiber gratings as band rejection filters", J. of Lightwave Technology, 14, pp 58, 1996
- [38] S.A. Vasiliev et al, "Properties of cladding modes of an optical fiber excited by refractive index gratings", Russian J. of Quantum Elect., 29, pp 65-68, 1999.

- [39] A. Cherin," An Introduction to Optical Fibers", McGraw Hill, Singapore, 1989.
- [40] V. Bhatia," Properties and sensing applications of long-period gratings," PhD Thesis, Virginia Tech, Blacksburg, VA, 1996.
- [41] J.W. Fleming," Material dispersion in lightguide glass", Electron. Letters, 14, pp 36-327, `1978.
- [42] N.P. Bansal, R.H. Doremus," Handbook of Glass Properties", Academic Press, Florida, 1986.
- [43] M.J. Adams," An Introduction to Optical Waveguide", Wiley-Interscience, New York, 1981.
- [43] Xuewen Shu, Lin Zhang, Ian Bennion," Sensitivity Characteristic of Long-Period Fiber Gratings", IEEE Journal of Lightwave Technology, 20, pp 255-266, 2002.
- [44] Bai-Ou Guan, Hwa-Yaw Tam, Helen L.W. Chan, Chung-Loong Choy, Muhtesem Suleyman Demokan," Growth Characteristics of Long-Period Gratings in Hydrogen-loaded fiber during and after 193nm UV inscription", Meas. Sci. Technology, 12, pp 818-823, 2001.
- [45] Mei Nar Ng, Kin Seng Chiang," Thermal Effects on the Transmission Spectra of Long-Period Fiber Gratings", Opt. Comms., 208, pp 321-327, 2002.
- [46] Heather J. Patrick, Alan D. Kersey, Frank Bucholtz," Analysis of the Response of Long Period Fiber Gratings to External Index of Refraction", IEEE J. of Lightwave Technology, 16, pp 1606-1612, 1998.
- [47] A.W. Snyder, J.D. Love," Optical Waveguide Theory", Chapman and Hall, London, 1984.
- [48] T. Erdogan," Fiber Gratings Spectra", IEEE J. Lightwave Technology, 15, pg 1277-1294, 1997.
- [49] Vikram Bhatia," Applications of Long Period Gratings to Single and Multi-parameter sensing", Opt. Express, 4, pp457-466, 1999.
- [50] Stephen W. James, Ralph P. Tatam," Optical Fiber Long Period Gratings Sensors: Characteristics and Application", Meas. Sci. Technology, 14, pp 49-61, 2003.
- [51] Shu X., Allsop T., Gwandu B, Zhang L, Bennion I., " High Temperature Sensitivity of Long Period Gratings in B-Ge codoped Fiber", IEEE Photon. Technology Letters, 13, pp818-820, 2001.
- [52] Heather J. Patrick, Alan D. Kersy, Frank Bucholtz," Analysis of the Response of Long Period Fiber Gratings to External Index of Refraction", IEEE J. of Lightwave Technology, 16, pp1606-1612, 1998.

- [53] R. Hou, Z. Ghassemlooy, A. Hassan, C.Lu, K.P. Dowker," Modelling of Long Period Fiber Gratings Response to Refractive index higher than that of cladding", Meas. Sci. Technology, 12, pp 1709-1713, 2001.
- [54] Oliver Duhem, Jean-Francois Henninot, Marc Warenghem, Marc Douay," Demonstration of Long Period Gratings Efficient Couplings with an External medium of a refractive index higher than that of silica", Appl. Opt, 37, pp 72223-7228, 1998.
- [55] H.J. Patrick, C.G. Askins, R.W. McElhanon, E.J. Friebele," Amplitude mask patterned on an Excimer Laser mirror for high intensity writing of Long Period fiber gratings", Electronics Letters 33, pp 1167-1168, 1997.
- [56] Malo B. Albert J, Hill KO, Bilodeau F and Johnson DC,"Effective index drift from molecular hydrogen diffusion in hydrogen-loaded optical fibers and its effect on Bragg Gratings fabrication", Electronics Letter, 30, pp442-443, 1994.
- [57] D.D Davis, T.K. Gaglord, E.N. Glytsis, S.Kosinski, S.C. Mettler and A.M. Vengsarkar,"Long-period fiber fabrication with focused CO₂ laser pulse", Electronics Lett, 34, pp 302-303, 1998
- [58] Yuki Kondo, Kentaro Nouchi, Tsuneo Mitsuyu, Masaru Wantanabe, Peter G. Kazansky, Kazuyuki Hirao,"Fabrication of long period fiber gratings by focused irradiation of infrared femtosecond laser pulses", Optic Lett, 24, pp 646-648, 1999
- [59] G Rego, O. Okhotnikov, E. Dianov and V. Sulimov,"High Temperature Stability of Long Period Fiber Gratings produced using an electric arc", Journal of Lightwave Technol.,19, pp 1574-1579, 2001
- [60] B.E.A. Saleh and M.C. Teich,"Fundamental of Photonics", John Wily, New York, 1991.
- [61] Hill K.O., Fuji Y. Johnson D.C. and Kawasaki B.S.,"Photosensitivity in optical waveguide: Application to reflection filter fabrication", Applied Physics Lett., **32**, pp 647-649, 1978
- [62] P.J. Lemaire, R.M. Atkins, V.Mizrahi and W.A. Reed,"High pressure H₂ loading as a technique for achieving ultrahigh UV photosensitivity and thermal sensitivity in GeO₂ doped optical fibers", Electronics Lett., 29, pp 1191-1193, 1993
- [63] William D.L., Ainslie B.J., Armitage J.R., Kashyap R. and Campbell R.J.,"Enhanced UV photosensitivity in boron codoped germanosilicate optical fibers", Electronics Lett., 29, pp 45-47, 1993.
- [64] F. Bilodeau, R. Malo, J.Albert, D.C, Johnson, K.O. Hill, Y. Hibino, M. Abe. And M.Kawachi,"Photosensitization of optical fiber and silica-on-silicon/ silica waveguides", Optics Lett., 18, pp 953-955, 1993

- [65] E. Salik, D.S. Starodubov, V. Grubsky and J. Feinberg, "Increase of photosensitivity in Ge-doped fibers under strain", Optical Fiber Communication Conference (OFC2000), paper TuH5, 2000.
- [66] Hyung Suk Ryu, Yongwoo Park, Seong Tae Oh, Youngjoo Chung and Dug Young Kim, "Effect of asymmetric stress relaxation on the polarization-dependent transmission characteristics of a CO₂ laser written long period fiber gratings", Optics Lett., 28, pp 155-157, 2003.
- [67] Y.J. Rao, T. Zhu, Z.L. Ran, Y.P. Wang, J. Jiang, A.Z. Hu, "Novel long period fiber gratings written by high frequency CO₂ laser pulses and application in optical fiber communication", Optical Comms., 229, pp209-221, 2004.
- [68] Yun-Jiang Rao, Yi-Ping Wang, Zeng-Ling Ran and Tao Zhu, "Novel Fiber Optic Sensors based on Long Period Gratings written by high frequency CO₂ laser pulses", Journal of Lightwave Technol., 21, pp 1320-1327, 2003
- [69] C. Caucheteur, A. Fotiadi, P. Megret, S.A. Slattery and D.N. Nikogosyan, "Polarization properties of long period gratings prepared by high intensity Femtosecond 352nm pulses", IEEE Photonics Technol. Lett., 17, pp 2346-2348, 2005
- [70] Bai-Ou Guan, Hwa-Yaw Tam, Helen LW Chan, Chung-Loong Choy, Muhtesem Suleyman Demokan, "Growth Characteristics of Long-Period Gratings in Hydrogen Loaded fiber during and after 193nm UV inscription", Measurement Science and Technology, 12, pp818-823, 2001
- [71] Hirouuki Kawano, Holger Muentz, Yukio Sato, Junichi Nishimae and Atsushi Sugitatsu, "Reduction of Transmission Spectrum Shift of long period fiber gratings by a UV preexposure method", Journal of Lightwave Technol., 19, pp 1221-1228, 2001.
- [72] B. Rout, C. Chera, K. J. Grant, A. Roberts and D. N. Jamieson, "Ion Beam Implantation For Fabrication Of Periodic Structures In Optical Fibres", Smart Structures, Devices, and Systems, Proceedings of SPIE Vol. 4935 (2002), v 4935, p 172-7, 2002
- [73] T Ergodan, V Mizrahi, PJ Lemaire, D Monroe, "Decay of ultra-violet induced fiber Bragg Gratings", Journal of Applied Physics, 76, pp73-80, 1994
- [74] Alexis Méndez, MCH Engineering, LLC, The 14th Optoelectronics and Communication Conference, Jul 2009.
- [75] Zhaobing Tian, Scott S.-H. Yam, Jack Barnes, Wojtek Bock, Patricia Greig, James M. Fraser, Hans-Peter Loock, and Richard D. Oleschuk, "Refractive Index Sensing With Mach-Zehnder Interferometer Based on Concatenating Two Single-Mode Fiber Tapers", IEEE Photonics Technology Letters, 20 (8), 2008.

- [76] Tao Zhu, Yanen Fan, Ming Deng, and Yunjiang Rao, "Highly Sensitive Refractive Index Sensor Based on Two Cascaded Long Period Gratings with Rotary Refractive Index Modulation", 21st International Conference on Optical Fiber Sensors, Proc. of SPIE Vol. 7753, 77539M
- [77] Jiang, Lan; Wang, Sumei; Chen, Qianghua; Li, Benye; Xiao, Hai, "Highly sensitive refractive index optical fiber sensors fabricated by a femtosecond laser", Photonics Journal, IEEE, 3(6), 2011.
- [78] Juan Kanga, b, Xinyong Dong a, Mengchao Lib, Zaixuan Zhang a, Shangzhong Jina. "Refraction Index Measurement Using Long Period Grating Fabricated by Symmetrical-Hole Fiber", Passive Components and Fiber-based Devices VII, Proc. of SPIE-OSA-IEEE Asia Communications and Photonics, SPIE Vol. 7986, 79860X.
- [79] Min-Suk Kwon and Sang-Yung Shin, "Refractive Index Sensitivity Measurement of a Long-Period Waveguide Grating", Photonics Technology Letters, IEEE, 17 (9), 2005.
- [80] Jinhua Yan, A. Ping Zhang, Li-Yang Shao, Jin-Fei Ding, Sailing He, "Simultaneous Measurement of Refractive Index and Temperature by Using Dual Long-Period Gratings With an Etching Process", IEEE Photonics Technology Letters, 17 (11), 2005.
- [81] A. Iadicicco, S. Campopiano, A. Cutolo, M.L. Korwin-Pawlowski, W.J. Bock, A. Cusano, "Refractive index sensitivity in thinned UV and arc induced Long-Period Gratings: A Comparative study", International Journal on Smart Sensing and Intelligent Systems, 1 (2), 2008.
- [82] Edward Davies, Pounesh Saffari, Chengbo Mou, Kaiming Zou and Lin Zhang "Refractive Index Sensitivity Enhancement of 81° tilted Bragg Gratings by Cladding etching", 20th International Conference on Optical Fibre Sensors, Proc. of SPIE Vol. 7503, 75036X.
- [83] Jian Yang, Li Yang, Chang-Qing Xu, and Yingfu Li, "Optimization of Cladding-Structure-Modified Long-Period-Grating Refractive-Index Sensors", Journal of Lightwave Technology 25(1), 2007.
- [84] Edward Davies, Reeta Viitala, Mikko Salomäki, Sami Areva, Lin Zhang and Ian Bennion, "Sol-gel derived coating applied to long-period gratings for enhanced refractive index sensing properties", J. Opt. A: Pure Appl. Opt. 11, 2009.

- [85] Sergiy Korposha, Seung-Woo Leea, Stephen W Jamesb and Ralph P Tatamb, "Refractive index sensitivity of fibre optic long period gratings with SiO₂ nanoparticle based mesoporous coatings", 21st International Conference on Optical Fiber Sensors, Proc. of SPIE Vol. 7753, 77539A.
- [86] Yi-Ping Wang, Wei Jin, D. N. Wang, "Strain Characteristics of CO₂-Laser-Carved Long Period Fiber Gratings", IEEE Journal of Quantum Electronics, v 43(2), p 101-8, 2007
- [87] Chia-Chin Chiang, Liren Tsai, Hao-Jhen Chang, Chih-Lang Lin, Jin-Shun Kuo, "Fabrication of Corrugated Long-period Fiber Gratings by wet bulk micromachining", Proc of SPIE - The International Society for Optical Engineering, v 7591, 2010, Advanced Fabrication Technologies for Micro/Nano Optics and Photonics III
- [88] Chunn-Yenn Lin, Lon A. Wang, Gia-Wei Chern, "Corrugated Long-Period Fiber Gratings as Strain, Torsion, and Bending Sensors", Journal of Lightwave Technology, v 19(8), p 1159-68, 2001
- [89] Hsiao-Yuh Wang, Shih-Min Chuo, and Lon A. Wang, "Fabrication and Simulation of Corrugated Long Period Microfiber Gratings", Proc of the SPIE - The International Society for Optical Engineering, v 7753, p 775397 (4 pp.), 2011
- [90] H Chih-Yu Huang, Wen-Lin Chan, Shih-Min Chuo, Jer-Haur Chang, Li-Lun Chen, Lon A Wang, "Packaged Symmetric/ Asymmetric Corrugated Long Period Fiber Gratings for Refractive Index Sensing Applications", Proceedings of SPIE - The International Society for Optical Engineering, v 7503, 2009, 20th International Conference on Optical Fibre Sensors
- [91] X. Shu, L. Zhang, I. Bennion, "Sensitivity characteristics of long period fiber gratings", Journal of Lightwave Technology, 20(2), 2002.
- [92] V. Bhatia, "Applications of long-period gratings to single and multi-parameter sensing", Opt. Exp. 4 (11), 1999.
- [93] H.J. Patrick, A.D. Kersey, F. Bucholtz, K.J. Judkins, A.M. Vengsarkar, "Chemical sensor based on long period grating response to index of refraction", Proceedings of Conference on Lasers Electro-Optics, Baltimore, p. 420, 1997.
- [94] H.J. Patrick, A.D. Kersey, F. Bucholtz, "Analysis of the response of long period fiber gratings to external index of refraction", J. Lightwave Technol. 15 (8), 1606, 1997.

- [95] L. Zhang and I. Bennion T. Allsop, "Detection of Organic aromatic Compounds in Paraffin by a Long-period fiber grating sensor with optimized sensitivity," Optics Communications, 191(181-190), 2001.
- [96] Chih-Yu Huang, Wen-Lin Chan, Shih-Min Chuo, Jer-Haur Chang, Li-Lun Chen and Lon A. Wang, "Packaged Symmetric/ Asymmetric Corrugated Long Period Fiber Gratings for Refractive Index Sensing Applications", Vol 7503, 75031C-1, 20th International Conference on Optical Fiber Sensors, 2009
- [97] Chunn-Yenn Lin, Lon A. Wang, Gia-Wei Chern, "Corrugated Long-Period Fiber Gratings as Strain, Torsion, and Bending Sensors", JOURNAL OF LIGHTWAVE TECHNOLOGY, VOL. 19, NO. 8, AUGUST 2001 1159
- [98] Turan Erdogan, "Cladding-mode resonances in short- and long period fiber grating filters", J. Opt. Soc. Am. A/ Vol. 14, No. 8/August 1997
- [99] Z. K. Wang and H. Y. Zheng, "Investigation on CO₂ laser irradiation inducing glass strip peeling for microchannel formation", American Institute of Physics, BIOMICROFLUIDICS 6, 012820 (2012)
- [100] Sensor Trends 2014, "Trends in Future-Oriented Sensor Technologies", Association for Sensor Technology, Europe.
http://ftp.ama-sensorik.de/Trendanalyse/AMA_Study_Sensor_Trends.pdf
- [101] Alexis Méndez, MCH Engineering, LLC, "The 14th Optoelectronics and Communication Conference", Jul 2009.
<http://www.ndt.net/article/panndt2007/papers/96.pdf>
- [102] Zhaobing Tian, Scott S.-H. Yam, Jack Barnes, Wojtek Bock, Patricia Greig, James M. Fraser, Hans-Peter Loock, and Richard D. Oleschuk, "Refractive Index Sensing With Mach–Zehnder Interferometer Based on Concatenating Two Single-Mode Fiber Tapers", IEEE Photonics Technology Letters, 20 (8), 2008.
- [103] Tao Zhu, Yanen Fan , Ming Deng, and Yunjiang Rao, "Highly Sensitive Refractive Index Sensor Based on Two Cascaded Long Period Gratings with Rotary Refractive Index Modulation", 21st International Conference on Optical Fiber Sensors , Proc. of SPIE Vol. 7753, 77539M
- [104] Jiang, Lan; Wang, Sumei; Chen, Qianghua; Li, Benye; Xiao, Hai, "Highly sensitive refractive index optical fiber sensors fabricated by a femtosecond laser", Photonics Journal, IEEE, 3(6), 2011.

- [105] Juan Kanga, b ,Xinyong Dong a, Mengchao Lib,Zaixuan Zhang a,Shangzhong Jina. "Refraction Index Measurement Using Long Period Grating Fabricated by Symmetrical-Hole Fiber", Passive Components and Fiber-based Devices VII, Proc. of SPIE-OSA-IEEE Asia Communications and Photonics, SPIE Vol. 7986, 79860X.
- [106] Min-Suk Kwon and Sang-Yung Shin, "Refractive Index Sensitivity Measurement of a Long-Period Waveguide Grating", Photonics Technology Letters, IEEE, 17 (9), 2005.
- [107] Jinhua Yan, A. Ping Zhang, Li-Yang Shao, Jin-Fei Ding, Sailing He, "Simultaneous Measurement of Refractive Index and Temperature by Using Dual Long-Period Gratings With an Etching Process", IEEE Photonics Technology Letters, 17 (11), 2005.
- [108] A. Iadicicco, S. Campopiano, A. Cutolo, M.L. Korwin-Pawlowski, W.J. Bock, A. Cusano, "Refractive index sensitivity in thinned UV and arc induced Long-Period Gratings: A Comparative study", International Journal on Smart Sensing and Intelligent Systems, 1 (2), 2008.
- [109] Edward Davies, Pouneh Saffari, Chengbo Mou, Kaiming Zou and Lin Zhang "Refractive Index Sensitivity Enhancement of 81° tilted Bragg Gratings by Cladding etching", 20th International Conference on Optical Fibre Sensors, Proc. of SPIE Vol. 7503, 75036X.
- [110] International Technology Roadmap for Semiconductor 2010 edition, Micro-Electro-Mechanical Systems (MEMS).
http://www.itrs.net/Links/2010ITRS/2010Update/ToPost/2010_Update_Overview.pdf
- [111] Kin Seng Chiang, Yunqi Liu, Mei Nar Ng, Xiaoyi Dong , " Analysis of etched long-period fibre grating and its response to external refractive index", Electronics Letters, Vol. 36 No. 17, May 2000
- [112] Prof. Milo Koretsky,"Wet etch vs Dry etch", Chemical Engineering dept, Oregon State University
- [113] Kay et al., "Topics in Current Chemistry", Springer-Verlag, NY (1980). Lynn Fuller, Richard Lane," Plasma Etching", Microelectronic Engineering, Rochester Institute of Technology.
- [114] R. Knizikevic~ius," Simulation of Si and SiO₂ etching in CF₄ plasma", Vacuum 82 (2008) 1191–1193

[115] GD VanWiggeren, TK Gaylord, DD Davis, E. Anemogiannis, BD Garrett, MI Braiwish, EN Glytsis, "Axial rotation dependence of resonances in curved CO₂ laser induced long period gratings", Electronics Letter, 36, pp 1354-1355, 2000

[116] Wen Bin Ji, Huan Huan Liu, Swee Chuan Tjin, Kin Kee Chow, and Anthony Lim, "Ultrahigh Sensitivity Refractive Index Sensor Based on Optical Microfiber", IEEE Photonics Technology Letters, Vol 24, No 20, Oct 15, 2012.