Carbon nanomaterials incorporated specialty optical fibers for environmental sensing applications

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**NANYANG
TECHNOLOGICAL
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**CARBON NANOMATERIALS INCORPORATED
SPECIALTY OPTICAL FIBERS FOR ENVIRONMENTAL
SENSING APPLICATIONS**

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**SCHOOL OF ELECTRICAL AND ELECTRONIC
ENGINEERING**

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Abstract

The role of optical fibers as a medium for information transfer has found many applications in the world today. One of these applications is in environmental sensing, where slight perturbations in the ambient environment can be remotely detected through modulation of light at selected points along the optical fiber cable. These perturbations exist in the form of variations in refractive index (RI), temperature or strain.

This thesis presents several optical fiber-based RI sensors, consisting of long period fiber gratings (LPFGs), Mach-Zehnder Interferometers (MZIs) and multimode interferometers (MMIs), which are individually fabricated from photonic crystal fibers (PCFs) or multimode fibers (MMFs), integrated with carbon nanomaterials. The integration process involved the deposition of a carbon nanotube (CNT) overlay on LPFGs, PCF-MZIs and MMF-MMIs, and multilayer graphene (MG) overlay on PCF-MZIs. These carbon nanomaterials with their unique properties modified the sensing scheme of the conventional sensors, allowing them to gain immunity from certain free spectral range limitations, extend their operation range and also present the potential for dual parameter sensing. For the CNT-deposited LPFGs and PCF-MZIs, the maximum achievable sensitivity within the RI range of 1.33 to 1.42 was approximately 47 dB/RIU and 24.2 dB/RIU, respectively. The maximum achievable sensitivity for the MG-deposited PCF-MZIs was approximately 17.5 dB/RIU within the RI range of 1.33-1.43. For the MMF-MMIs, the maximum achievable sensitivity was approximately 98.8 dB/RIU within the RI range of 1.33 to 1.46. These modified sensors were not only able to continuously measure variations in RI of their ambient environment while only acquiring errors mainly from source power fluctuations, but also exhibited comparable performances in terms of their stability, repeatability and reproducibility.

Table of Contents

Acknowledgements	i
Abstract	iii
Table of Contents	iv
List of Figures.....	viii
List of Tables	xii
List of Abbreviations	xiii
Chapter 1 -An Overview of this Thesis	1
1.1 Optical Fibers and their Role as Environmental Sensors	1
1.2 Motivation and Objectives.....	6
1.3 Major Contribution of the Thesis	7
1.4 Format and Organization of the Thesis	8
1.5 Concluding remarks.....	10
Chapter 2 - An Overview of Optical Fiber Sensing Probes	11
2.1 Optical Fiber Sensing Probes: Characteristics and Classification	11
2.2 Optical Fiber Gratings.....	15
2.2.1 Long Period Fiber Gratings (LPFGs).....	16
2.2.2 Fiber Bragg Gratings (FBGs)	19
2.2.3 Grating variants.....	21
2.3 Tapered Fiber	23
2.4 Optical Fiber Interferometers.....	26
2.4.1 Fabry-Perot Interferometer (FPI)	26
2.4.2 Mach-Zehnder Interferometer (MZI)	27
2.4.3 Michelson Interferometer (MI)	27
2.4.4 Multimode Interferometer (MMI)	28
2.4.5 Specialty Fiber Interferometers	28
2.5 Un-cladded Fiber Sensors and Surface Plasmon Resonance	34
2.6 Sensing Probes with Material Overlays	37
2.7 Other Environmental Sensing Applications	38
2.8 Fundamental Noise Limits.....	39
2.9 Concluding remarks.....	41
Chapter 3 - Experimental Procedures and the RI Sensing Characteristics of the Tapered Fiber, PCF-MZI and MMF-MMI.....	42
3.1 Experimental Procedures	42

3.1.1 Optical Equipment and Components	42
3.1.2 Test Solution Preparation	43
3.1.3 Behavior Characterization Procedure	44
3.1.4 Data Processing and Analysis	45
3.2 Tapered Fiber Sensing Probe	47
3.2.1 Fabrication	47
3.2.2 Results	49
3.3 PCF-MZI Sensing Probe	52
3.3.1 Fabrication	52
3.3.2 Results	54
3.4 MMF-MMI Sensing Probe	59
3.4.1 Fabrication	59
3.4.2 Results	61
3.5 Limitations	65
3.5.1 Free Spectral Range	65
3.5.2 Noise Floor Limit	68
3.5.3 Temperature Dependence	69
3.6 Concluding Remarks	70
Chapter 4 - Carbon Nanomaterial Overlays	71
4.1 Carbon Nanomaterials as Thin Film Overlays	71
4.1.1 Graphene	72
4.1.2 Carbon nanotubes (CNTs)	74
4.2 Modelling of Carbon Nanomaterial Overlays	76
4.3 Preparation of solutions	82
4.4 Raman Characterization of the Carbon Nanomaterial Solutions	82
4.5 Deposition Methods	85
4.5.1 Spray Coating	86
4.5.2 Optical deposition	88
4.6 Concluding Remarks	91
Chapter 5 - CNT-Deposited LPFG	92
5.1 Sensing mechanism of the CNT-Deposited LPFG	92
5.2 Fabrication of the CNT-Deposited LPFG	93
5.3 Sensing Behavior of the CNT-Deposited LPFG	96
5.3.1 RI Characterization	96
5.3.2 Wavelength Behavior	97

5.3.3 Repeatability and Sensitivity	98
5.4 Discussion of Results	100
5.5 Concluding Remarks.....	101
Chapter 6 - Carbon Nanomaterial-Deposited PCF-MZI	102
6.1 Sensing Mechanism of Carbon Nanomaterial-deposited PCF-MZIs.....	102
6.2 Fabrication of the Carbon Nanomaterial-Deposited PCF-MZIs.....	103
6.3 Sensing Behavior of CNT-deposited PCF-MZI.....	107
6.3.1 RI Characterization of CNT-deposited PCF-MZI	107
6.3.2 Wavelength Behavior of CNT-deposited PCF-MZI.....	108
6.3.3 Repeatability, Sensitivity and Reproducibility of CNT-deposited PCF-MZI	109
6.4 Sensing Behavior of MG-deposited PCF-MZI	111
6.4.1 RI Characterization of MG-deposited PCF-MZI.....	111
6.4.2 Wavelength Behavior of MG-deposited PCF-MZI	112
6.4.3 Repeatability, Reliability and Sensitivity of MG-deposited PCF-MZI	113
6.5 Discussion of Results	114
6.6 Concluding Remarks.....	116
Chapter 7 - CNT-Deposited MMF-MMI.....	117
7.1 Sensing mechanism of the CNT-deposited MMF-MMI	117
7.2 Fabrication of CNT-deposited MMF-MMI	118
7.3 Sensing behavior of CNT-deposited MMF-MMI.....	120
7.3.1 RI characterization of the CNT-deposited MMF-MMI.....	120
7.3.2 Wavelength Behavior of the CNT-deposited MMF-MMI	121
7.3.3 Sensitivity, Repeatability and Stability of the CNT-deposited MMF-MMI	122
7.4 Discussion of results	124
7.5 Concluding Remarks.....	125
Chapter 8 - Discussion Conclusion and Future Work.....	126
8.1 Discussion and Comparison of all Sensing Probes	126
8.2 Conclusion.....	133
8.3 Recommendations for Future work.....	135
List of Publications	138
References	139

Appendix A - MATLAB code of the Variation in Reflectance to a variation in RI of the Ambient Environment for Normal Incidence of Light at the Carbon Nanomaterial Overlay	175
Appendix B - MATLAB code of the Variation in Reflectance to a variation in RI of the Ambient Environment for Light at an Incident Angle of 30° at the Carbon Nanomaterial Overlay.....	176

List of Figures

Fig. 1-1	Schematic of an optical fiber cable indicating the cross section of the fiber core with a radius a and cladding with a radius b (left), and the cross section of an optical fiber cable with the polymer coating (right).....	1
Fig. 1-2	Geometric optics representation of the light propagation mechanism in an optical fiber. The incident, refracted and reflected rays are indicated by the dashed, dotted and dot-dash arrows, respectively.	2
Fig. 1-3	Geometric optics representation of the light propagation mechanism in a single mode fiber. The light ray is indicated by the solid arrow.....	3
Fig. 2-1	Flow diagram of various optical fiber sensing probes. Abbreviations of the various sensing probes are as follows: LPFG-long period fiber grating. FBG-fiber Bragg grating. FPI-Fabry-Perot Interferometer. MZI-Mach-Zehnder Interferometer. MI-Michelson Interferometer. SI-Sagnac Interferometer. MMI-Multimode Interferometer).	12
Fig. 2-2	Sensing setup for (a) the transmissive configuration and (b) the reflective configurations. (SMF: single mode fiber).	14
Fig. 2-3	Schematic of a grating of period Λ inscribed within the core of an optical fiber.....	15
Fig. 2-4	Schematic of the behavior of the fundamental mode (solid arrow) on interaction with a LPFG and the observable transmitted spectrum. The dashed arrows indicate the cladding modes.	17
Fig. 2-5	Schematic of the behavior of the fundamental mode (solid arrow) on interaction with a FBG and the observable transmitted and reflected spectrums. The dotted arrow indicates the back reflected mode.....	20
Fig. 2-6	Schematic of the cascaded LPFGs, tilted FBG and superstructure grating and the behavior of the fundamental mode on interaction with these sensing probes.	22
Fig. 2-7	Schematic of a tapered fiber with the down-taper, tapered and up-taper regions indicated.....	24
Fig. 2-8	Schematic of (a) a solid core and (b) a hollow core photonic crystal fiber (PCF). The uncolored circles indicate the positions of the air holes.....	29
Fig. 2-9	Schematic of a solid core photonic crystal fiber (PCF) fusion spliced between two SMFs to form a sensing probe. The core mode, excited higher order cladding modes and the evanescent waves of the excited higher order cladding modes are indicated by the solid, dashed and dotted arrows, respectively.	30
Fig. 2-10	Schematic of one type of multimode fiber multimode interferometer (MMF-MMI). The fundamental mode and the excited higher order modes are indicated by the solid and dashed arrows, respectively.....	32
Fig. 2-11	Schematic of (a) an optical fiber cable with its cladding removed through etching and (b) a side polished fiber.....	35
Fig. 2-12	Schematic of (a) Kretschmann's configuration for surface plasmon resonance (SPR) and (b) an optical fiber SPR sensing probe with a metallic layer deposited over the exposed fiber core.	36
Fig. 3-1	Plot of the variation in RI of a particular test solution due to variations in temperature of the ambient environment.....	45
Fig. 3-2	Schematic and photograph of the setup used in the fabrication of a tapered fiber.....	48

Fig. 3-3	(a) Schematic of the fabricated tapered fiber sensor with the taper dimensions and (b) the output spectra when the tapered region is immersed in a test solution with a RI of 1.3332.	49
Fig. 3-4	(a) Behavior of the interference dip at a wavelength of 1550.4 nm to variations in RI of the ambient environment. (b) Amount of red shift in the wavelength of the interference dip within the RI range of 1.3332-1.33445.	51
Fig. 3-5	Microscopy image of the region where a SMF is fusion spliced to the PCF segment.	53
Fig. 3-6	Output spectra of the two PCF-MZIs in air for a PCF length of (a) 2.7 cm and (b) 3.0 cm, respectively.	54
Fig. 3-7	Behavior of the interference dip at a wavelength of 1531.84 nm to variations in RI of the ambient environment for a PCF-MZI with a 2.7 cm PCF segment. (b) Amount of red shift in the wavelength of the interference dip within the RI range of 1.33-1.42.	55
Fig. 3-8	Behavior of the interference dip at a wavelength of 1579.00 nm to variations in RI of the ambient environment for a PCF-MZI with a 3.0 cm PCF segment. (b) Amount of red shift in the wavelength of the interference dip within the RI range of 1.33-1.42.	57
Fig. 3-9	Output spectrum of the two MMF-MMIs in air for a MMF length of (a) 1.2 cm and (b) 1.5 cm.	60
Fig. 3-10	Behavior of the interference peak at a wavelength of 1557.04 nm to variations in RI of the ambient environment for a MMF-MMI with a 1.2 cm MMF segment. (b) Amount of variation in the intensity of the interference peak within the RI range of 1.33-1.46.	62
Fig. 3-11	Behavior of the interference peak at a wavelength of 1555.96 nm to variations in RI of the ambient environment for a MMF-MMI with a 1.5 cm MMF segment. (b) Amount of variation in the intensity of the interference peak within the RI range of 1.33-1.46.	63
Fig. 3-12	Wavelength behavior of the interference peak to variations in RI of the ambient environment for the MMF-MMIs with a (a) 1.2 cm MMF segment and (b) 1.5 cm MMF segment.	64
Fig. 3-13	Behavior of all interference dips within the wavelength range of 1510-1630 nm to variations in RI of the ambient environment for the 10 μ m tapered fiber.	65
Fig. 3-14	Behavior of all interference dips within the wavelength range of 1510-1630 nm to variations in RI of the ambient environment for the (a) 2.7 cm and the (b) 3.0 cm PCF-MZIs.	67
Fig. 4-1	Schematic of the two dimensional honeycomb lattice structure of graphene. Carbon atoms are indicated by the colored circles.	73
Fig. 4-2	Schematic of the honeycomb lattice structure of graphene to be folded along the chiral vector Ch. Carbon atoms are indicated by the colored circles. The chiral vector of a (4, 1) CNT is shown in the figure.	74
Fig. 4-3	Schematic of the deposition of carbon nanomaterials onto the surfaces and end-faces of the LPFGs, PCFIs and MMFIs.	76
Fig. 4-4	Modelling of the fiber, carbon nanomaterials and ambient environment as individual layers with different RIs based on (a) based on the normal incidence of light at the interface between the fiber and carbon nanomaterial layers and (b) light interacting with the carbon nanomaterial layer at an incident angle.	77

Fig. 4-5	Simulation based on equation (4.21) of the variation in reflectance as the RI of the ambient environment varied for the case of light interacting with the carbon nanomaterial overlay at normal incidence and at an incident angle of 30°	81
Fig. 4-6	(a) Raman spectra of the CNT solution with the D, G and G' bands marked. (b) Absorbance spectra of the CNT in DMF.	83
Fig. 4-7	(a) Raman spectra of the MG solution with the D, G and G' bands marked. (b) Scanning electron microscope (SEM) image of the MG flakes on a silicon wafer.	84
Fig. 4-8	Schematic of the setup for spray coating of a carbon nanomaterial overlay onto an optical fiber sensing probe.....	86
Fig. 4-9	Schematic of the setup for optical deposition of a CNT overlay onto the end-face of a MMF-MMI.	88
Fig. 4-10	Variation in intensity with time for the optical deposition process.	89
Fig. 4-11	Schematic of the optical deposition mechanism showing the four forces acting on the CNTs.....	90
Fig. 5-1	Schematic of the light propagation mechanism of a CNT-deposited LPFG. ...	92
Fig. 5-2	Schematic of the setup for the inscription of a long period grating into a SMF.	94
Fig. 5-3	Optical microscope image of the LPFG (a) before and (b) after the deposition of the CNT overlay.	95
Fig. 5-4	Behavior of the attenuation band at a wavelength of 1463.2 nm to variations in RI of the ambient environment.....	96
Fig. 5-5	Behavior of the attenuation band at a wavelength of 1463.2 nm to variations in RI of the ambient environment.....	97
Fig. 5-6	Variation in wavelength of the attenuation band at a wavelength of 1463.2 nm to variations in RI of the ambient environment.....	98
Fig. 5-7	Variation in intensity of the attenuation band at a wavelength of 1463.2 nm to variations in RI of the ambient environment for two separate trials.	99
Fig. 6-1	Schematic of the light propagation mechanism of a carbon nanomaterial-deposited PCF-MZI.	103
Fig. 6-2	Output spectrum of the PCF-MZIs before (dotted line) and after (solid line) deposition of (a) a CNT overlay and (b) a multilayer graphene (MG) overlay.	104
Fig. 6-3	Microscopy images of a small region of the PCF-MZI (a) before and (b) after the deposition of a CNT overlay onto the sensing probe.	105
Fig. 6-4	Microscopy images of a small region of the PCF-MZI (a) before and (b) after the deposition of a MG overlay onto the sensing probe.....	106
Fig. 6-5	Behavior of the interference dip at a wavelength of 1580.20 nm to variations in RI of the ambient environment.....	108
Fig. 6-6	Variation in wavelength of the interference dip at a wavelength of 1580.20 nm to variations in RI of the ambient environment.....	109
Fig. 6-7	Variation in intensity of the interference dips of two PCF-MZIs to variations in RI of the ambient environment for two CNT-deposited PCF-MZIs. Data points of the first CNT-deposited PCF-MZI are indicated by the diamonds and hexagons and fitted with a solid line indicate while that of the second CNT-deposited PCF-MZI are indicated by the hollow circles and fitted with a dotted line.	110

Fig. 6-8	Behavior of the interference dip at a wavelength of 1577.68 nm to variations in RI of the ambient environment.....	112
Fig. 6-9	Variation in wavelength of the interference dip at 1580.20 nm to variations in RI of the ambient environment.....	113
Fig. 6-10	Variation in intensity of the attenuation band at 1463.2 nm to variations in RI of the ambient environment for two separate trials.....	114
Fig. 7-1	Schematic of the light propagation mechanism of a CNT-deposited MMF-MMI.....	117
Fig. 7-2	Output spectrum of the MMF-MMI before (dotted line) and after (solid line) deposition of a CNT overlay.....	118
Fig. 7-3	Microscopy images of the MMF-MMI (a) before and (b)after deposition of CNTs.....	119
Fig. 7-4	Behavior of the interference peak at a wavelength of 1558.84 nm to variations in RI of the ambient environment.....	121
Fig. 7-5	Variation in wavelength of the interference peak at a wavelength of 1558.84 nm as the RI of the ambient environment varied.	121
Fig. 7-6	Variation in intensity over two trials of the interference peak at 1558.84 nm as the RI of the ambient environment varied.	122
Fig. 7-7	Variation in intensity of the interference peak at 1558.84 nm over 60 mins for the CNT-deposited MMF-MMI immersed in a test solution with a RI of 1.3334.....	123

List of Tables

Table 1	Table showing a list of commonly used equipment with their model and their abbreviation for use in this thesis.	43
Table 2	Table comparing the sensitivities and the minimum achievable resolution of all optical fiber RI sensors studied in this thesis.....	130
Table 3	Table comparing the sensitivities and the maximum achievable resolution of all optical fiber RI sensors studied in this thesis.....	131

List of Abbreviations

Abbreviation	Expression
ASE	Amplified Spontaneous Emission
CNT	Carbon Nanotube
CVD	Chemical Vapor Deposition
DMF	Dimethylformamide
EDFA	Erbium-Doped Fiber Amplifier
ESAM	Electrostatic Self-Assembly
FBG	Fiber Bragg Grating
FPI	Fabry-Perot Interferometer
LB	Langmuir-Blodgett
LPFG	Long Period Fiber Grating
MMI	Multimode Interferometer
MG	Multilayer Graphene
MI	Michelson Interferometer
MIR	Mid-Infrared
MMF	Multimode Fiber
MWCNT	Multi-Wall Carbon Nanotube
MZI	Mach-Zehnder Interferometer
OSA	Optical Spectrum Analyzer
PCF	Photonic Crystal Fiber
RBW	Resolution Bandwidth
RI	Refractive Index
RIN	Relative Intensity Noise
SEM	Scanning Electron Microscope
SMF	Single Mode Fiber
SNR	Signal-to-Noise Ratio
SPR	Surface Plasmon Resonance
SWCNT	Single-Wall Carbon Nanotube
TIR	Total Internal Reflection
TLS	Tunable Laser Source

Chapter 1 -An Overview of this Thesis

This chapter will briefly introduce optical fibers and provide a basic idea of their information transfer mechanism based on the geometric optics approach. It will then go on to explain the need for environmental sensors and how optical fibers can be applied as environmental sensors. The motivations, objectives and the major contributions would be discussed next. This would then be followed by a brief outline of the thesis.

1.1 Optical Fibers and their Role as Environmental Sensors

An optical fiber is a long strand of dielectric material that is capable of trapping electromagnetic energy in the form of optical radiation at one end, and guiding this optical radiation in a direction parallel to its axis to the other end [1, 2]. The schematic of one such optical fiber cable is shown in Fig. 1-1 [1]. The conventional optical fiber consists of a cylindrical dielectric core with radius a and surrounded by a solid dielectric

Fig. 1-1 Schematic of an optical fiber cable indicating the cross section of the fiber core with a radius a and cladding with a radius b (left), and the cross section of an optical fiber cable with the polymer coating (right).

Fig. 1-2 Geometric optics representation of the light propagation mechanism in an optical fiber. The incident, refracted and reflected rays are indicated by the dashed, dotted and dot-dash arrows, respectively.

cladding with radius b [3]. The refractive indices (RIs) of the fiber core and fiber cladding are given by n_{core} and n_{clad} , where n_{core} is greater than n_{clad} [2]. An elastic polymer material, which not only enhances the mechanical strength of the fiber but also acts as a protective buffer for the fiber against any abrasive or rough surfaces, encapsulates the optical fiber [1].

Light (optical radiation) propagates through the core of the optical fiber via the total internal reflection (TIR) mechanism. Fig. 1-2 shows the geometric optics representation of light propagation through an optical fiber [1]. This approach simplifies the propagation mechanism of light in an optical fiber by treating the propagating light as discrete rays instead of waves as in the wave propagation representation. One such ray is shown propagating through the optical fiber from left to right. The incident ray (dashed arrow) is skewed at an angle to the central axis of the fiber cable and encounters the interface between the optical fiber core and cladding at an incident angle θ_i . As

mentioned above, since n_{core} is designed to be larger in value than n_{clad} , part of the ray (dotted arrow) will be partially refracted at an angle of refraction θ_r away from the normal (vertical dashed line), and part of it will be reflected (dot-dashed arrow) [4]. The relationship between θ_i and θ_r can be obtained from the modified form of Snell's law [2]:

$$n_{core} \sin \theta_i = n_{clad} \sin \theta_r \quad (1.1)$$

From here, it can be seen that θ_r increases as θ_i increases. At some value of θ_i , θ_r would become 90° and that value of θ_i would be known as the critical angle θ_c [2, 4]. Any light ray incident on the core-cladding interface at an angle greater or equal to the critical angle θ_c would experience TIR. With this mode of light confinement within the optical fiber and the appropriate equipment, large amounts of light and thus information, can be transferred through a single optical fiber.

Fig. 1-3 Geometric optics representation of the light propagation mechanism in a single mode fiber. The light ray is indicated by the solid arrow.

The above description is for that of a multimode fiber (MMF) where multiple rays (modes) with different θ_i can propagate through the fiber at once. For a single mode fiber (SMF), only one mode (solid arrow) propagates through the fiber as shown in Fig 1-3. These fibers are fabricated with core diameters on the order of a few wavelengths (approximately 8-10 μm) with small differences in RI of the fiber core and cladding [1]. SMFs are preferred for telecommunications as they have lower pulse delay distortion, lower power attenuation and no modal noise due to the propagation of only one mode through them [5].

This mechanism for information transfer has allowed optical fibers to be used as tools for high-speed transfer of large amounts of information. This would also allow it to be applied in other areas. One of these applications is in environmental sensing, specifically in the fields relating to water purity. Water is one of basic necessities for life, and clean water is crucial for the survival of many of the Earth's living organisms. However, with the advent of industrialization in the world today, water can knowingly or unknowingly be contaminated [6]. Apart from that, it can also serve as a transport medium for certain viruses [7], which have the potential to create outbreaks or even epidemics. As such, efficient treatment and detection of contaminants in water is required [8]. This is especially crucial for a country like Singapore where naturally available water is insufficient and various planning and management policies have to be put in place to sustain its demand [9-11]. These policies culminate in what is commonly referred to as the "four national taps" which consists of NEWater that supplies 30% of the daily demand, desalinated water that supplies 25% of the daily demand, and imported water and water from local catchment areas supplementing the remainder of the daily demand [12, 13]. These sources of water are either recycled, imported or from

natural sources and have to be checked for their purity and any contaminants before they can be piped to the masses for use.

One good measure of the purity of water is its RI. Changes in the RI of water can provide information on the status of certain environmental events. A variation in RI of a large body of water would imply a variation in the salinity level which could aid in the study of tidal dynamics along coastal areas [14]. In the chemical, pharmaceutical and biological industries, the presence of various compounds in an aqueous medium can be correlated to the RI of the medium. The presence and concentrations of various organic compounds [15], and the molecular bindings and interactions of proteins [16, 17] can be monitored through observing the variations in RI of the aqueous medium consisting of these compounds.

Conventional refractometers can be calibrated to measure the RI and thus the purity of water. Some of these are variants of the Abbe refractometer and are able to measure the RI of a solution in the visible and near-infrared regions of the electromagnetic spectrum. These refractometers have demonstrated sensitivities on the order of 10^{-3} [18] or 10^{-4} [19] within a wavelength range of 1.30 to 1.70.

Optical fibers can be modified at certain areas along their length or at the ends to form sensing probes. Light guided through an optical fiber can respond to various physical, chemical or biological perturbations in the immediate environment of these modified regions (ambient environment), to produce an output in relation to these perturbations [20]. Apart from that, in what is commonly referred to as multiplexing, several of these sensing probes can be located along a single or several optical fiber cables to form a large array of sensors which can be simultaneously monitored from a remote location [21]. This coupled with certain inherent advantages over their conventional and electronic counterparts allow optical fibers to have the potential to be

applied as real-time and continuous detectors in water treatment plants as well as industrial processes with RI specific requirements.

1.2 Motivation and Objectives

The need for real-time and continuous online monitoring of the RI of water is crucial and the optical fiber sensor has the potential to fulfil this need. Sensors in general are required to demonstrate behavior that fulfil the following criteria: (1) high sensitivity, (2) acceptable sensing resolution, (3) large measurement range, (4) good mechanical strength, (5) simple and cost effective fabrication processes and (6) low design complexity. Many optical fiber sensing probes have been demonstrated over the years, each with their own advantages and limitations. For these sensing probes, there is usually a trade-off between the above mentioned criteria. For example, sensing probes which are able to achieve a high sensitivity and acceptable sensing resolution tend to have a small measurement range or are extremely fragile. Sensing probes that are able to fulfil the first four criteria above would however have a high fabrication cost and high design complexity.

The objective of this thesis is thus to demonstrate a new type of optical fiber sensing probe which would be able to fulfil the six general criteria stated above. This will be done through the integration of three novel ideas: (i) optical fiber sensing probes with a deposited material overlay for enhancement of their sensing capabilities, (ii) carbon nanomaterials as overlay materials and (iii) relatively low cost and low complexity deposition methods. Various sensing probes will be fabricated based on these three ideas to study their applicability as RI sensing probes based on the stated criteria above.

1.3 Major Contribution of the Thesis

This thesis studies various established optical fiber sensing probes and their fabrication processes and behavior to variations in RI of the ambient environment. The various sensing probes studied are the Tapered fiber, the photonic crystal fiber in a Mach-Zehnder Interferometer configuration (PCF-MZI) and the multimode interferometer (MMI) consisting of a short segment of a multimode fiber (MMF) fusion spliced to a single mode fiber (SMF). The advantages and limitations of these sensing probes based on the six general criteria would be highlighted and discussed.

The thesis also proposes and describes, theoretically, a new and novel type of optical fiber sensing probe which would enhance the sensing capabilities and performance of some of these previously demonstrated sensing probes. This new type of sensing probe involves the deposition of a carbon nanomaterial overlay onto various optical fiber sensing probes. The unique optical properties of these carbon nanomaterials were also studied and shown to be compatible with the sensing probes for RI sensing.

It also presents two low cost, low complexity, simple and reliable deposition methods for depositing an overlay of carbon nanomaterials onto the surfaces of several sensing probes. These are the spray coating and optical deposition methods. The morphology of the deposited carbon nanomaterials was also studied and were shown to form a thin overlay over the sensing probes upon deposition, which demonstrates the suitability of these deposition methods.

The long period fiber grating (LPFG), PCF-MZI and MMF-MMI were chosen as the sensing probes to be deposited with the carbon nanomaterials. Two commonly found carbon nanomaterials, carbon nanotubes (CNTs) and multilayered graphene (MG), were explored as overlay materials. The carbon nanomaterial-deposited sensing

probes were found to experience an enhancement in RI sensing performance and capabilities due to the modification of their sensing mechanisms by the carbon nanomaterials. The achievable resolutions were generally improved by as much as two orders of magnitude for some sensing probes, while the continuous RI measurement range was found to be much larger as well.

1.4 Format and Organization of the Thesis

This thesis is organized as follows. Chapter one will give an introduction to optical fibers and their light guiding mechanism based on the geometric optics representation. It will then go on to discuss the need for optical fiber sensing probes in environmental sensing, particularly in the fields of RI sensing, and how optical fibers can potentially fill this need. The motivation and objectives behind working on optical fiber sensors as well as the major contributions of the thesis would be provided. The chapter will conclude with a description of the format of the thesis.

In chapter 2, the various optical fiber sensing probes will be discussed. The chapter will first introduce the various sensing probes based on a simple classification scheme and go on to describe the various sensing configurations and their advantages and limitations. It will then go on to discuss several sensing probes focusing particularly on the optical fiber gratings, tapered fibers, interferometers and the cladding removed optical fibers. The sensing mechanisms and sensing capabilities of each of these sensing probes will be discussed in detail. The chapter will conclude with the brief introduction of a novel sensing probe involving the deposition of a material overlay onto some of the sensing probes discussed earlier in the chapter.

Chapter 3 will provide details on the experimental processes including the equipment used, the preparation of the test solutions, characterization procedures and

the data processing techniques used. This would be followed by a discussion of the tapered fiber, PCF-MZI and MMF-MMI sensing probes. A description of the fabrication processes as well as the response of these sensing probes to variations in RI of the ambient environment would be studied and presented. The chapter will conclude with a discussion of the various limitations in sensing capabilities that these sensing probes would experience due to the nature of their sensing schemes.

Chapter 4 will introduce the carbon nanomaterials to be deposited onto the various sensing probes. The properties of graphene and CNTs will be described followed by a modelling of their behavior upon deposition onto the various sensing probes. The process of preparing the carbon nanomaterial solutions for deposition and the characterization techniques will also be described. The chapter will conclude with a description of the spray coating and optical deposition methods used to deposit an overlay of both carbon nanomaterials onto the various sensing probes. The deposition mechanisms as well as their applicability to the various sensing probes would also be discussed.

Chapter 5 will look at the first type of carbon nanomaterial-deposited sensing probe. The sensing mechanism and fabrication process of the CNT-deposited LPFG will first be described. This will be followed by a discussion of the response of the sensing probe to variations in RI of the ambient environment.

Chapter 6 will look at the carbon nanomaterial-deposited PCF-MZIs. The sensing mechanism and fabrication process of the CNT- and MG-deposited PCF-MZIs will be described. The response of both carbon nanomaterial-deposited PCF-MZIs to variations in RI of the ambient environment would be discussed and compared.

Chapter 7 will focus on the last sensing probe to be introduced. The sensing mechanism and fabrication process of the CNT-deposited MMF-MMI would first be described. This would be followed by a discussion of its response to variations in RI of the ambient environment. A final overall discussion of all sensing probes discussed in this thesis would be provided at the end of the chapter. A comparison of the sensing behavior of the sensing probes would be carried out and the enhancement in sensing capabilities afforded through the deposition of the two carbon nanomaterials would also be discussed.

The last chapter will give a conclusion of the thesis and propose several directions for future work.

1.5 Concluding remarks

Optical fibers which were once only used as communication tools have recently found applications in environmental sensing applications. This has led to an increase in research and development of many new and novel optical fiber sensing probes. The next chapter will describe several of these sensing probes and their applications in environmental sensing.

Chapter 2 - An Overview of Optical Fiber Sensing

Probes

Optical fiber sensing probes come in many forms with the most basic type making use of Fresnel reflection at the cleaved end-face of the optical fiber [22]. Though the focus of this thesis is on RI sensing, it would be more useful to discuss the environmental sensing capabilities of optical fibers in general before dealing specifically with RI sensing.

In this chapter, a brief survey of the currently available optical fiber sensing probes including how they would be classified in this thesis will be conducted. The sensing configuration and mechanism of various sensing probes as well as their behavior towards various environmental perturbations will be discussed. A novel concept involving the deposition of a material overlay over an optical fiber sensing probe to enhance its sensing capabilities would be introduced. Lastly discussions on the environmental sensing applications and the fundamental noise limits which would affect these optical fiber sensors would be presented.

2.1 Optical Fiber Sensing Probes: Characteristics and

Classification

Since their first application as environmental sensors in 1977 [23], optical fibers have received a lot of attention from the research community. Optical fibers are generally more advantageous for sensing applications as compared to their electrical counterparts due to their electrical passivity which allows them to operate in an environment with varying electromagnetic fields, chemically and biologically inert properties, long distance information transfer capabilities for remote operation, and

Fig. 2-1 Flow diagram of various optical fiber sensing probes. Abbreviations of the various sensing probes are as follows: LPFG-long period fiber grating. FBG-fiber Bragg grating. FPI-Fabry-Perot Interferometer. MZI-Mach-Zehnder Interferometer. MI-Michelson Interferometer. SI-Sagnac Interferometer. MMI-Multimode Interferometer).

corrosion resistance [20]. Apart from that, large numbers of optical fiber sensing probes can be multiplexed together to form a sensing array which can be further interconnected to form a sensing network covering a large area [20, 21]. The behavior of the sensing probes in the array to any perturbations in the ambient environment can then be monitored from a single remote location.

A general classification of optical fiber Sensors in the form of a flow diagram is shown in Fig. 2-1. Optical fiber sensors can be classified into point sensors which measure various perturbations in the ambient environment of a particular location, and distributed sensors which measure perturbations throughout the length of an optical fiber cable [21]. The focus of this thesis will be on point sensors which are generally short segments of optical fiber cables with their properties modified to form a sensing probe.

Point sensors can be further divided into various classes including gratings, tapers, interferometers and cladding removed optical fibers. Fiber gratings consist of the long period fiber gratings (LPFGs) and fiber Bragg gratings (FBGs). Variations of these two types of gratings have also been proposed through the cascading of two or more LPFGs, writing multiple FBGs onto a single optical fiber cable to form a superstructure grating or angling the fiber during the FBG writing process to form a tilted-FBG. The class of interferometers can be sub-divided into the Fabry-Perot Interferometers (FPIs), Mach-Zehnder Interferometers (MZIs), Michelson Interferometers (MIs), Sagnac Interferometers (SI) and for this thesis only, multimode interferometers (MMIs). Cladding removed optical fibers, are optical fiber cables which have a short segment of their cladding removed through chemical etching or mechanical polishing to form the sensing probe. These sensing probes can be further coated with a metallic layer to create an effect known as surface plasmon resonance which would greatly enhance their sensitivity. Right at the bottom of the flow diagram is a bracket marked out to show that all these point sensors can be further integrated with various materials to form a new category of sensing probes known as thin film sensors.

The sensing configurations for these point sensors can be described as either transmissive or reflective [21]. Fig. 2-2 shows the two configurations. The transmissive configuration in Fig. 2-2(a) is usually for optical fibers which have had a short segment along their lengths modified to form the sensing probe. In this configuration, the sensing probe is fusion spliced between two SMF cables, SMF 1 and SMF 2. SMF 1 is connected to a light source while SMF 2 is connected to a detector, usually a power meter or an optical spectrum analyzer (OSA). Light is coupled into SMF 1 and enters the sensing probe. The light in the sensing probe will then be modulated by any perturbations in the ambient environment and propagate via SMF 2 into the detector. The advantage of using

Fig. 2-2 Sensing setup for (a) the transmissive configuration and (b) the reflective configurations. (SMF: single mode fiber).

such a configuration is that most of the light from the source would enter the OSA which allows for the use of detectors with a higher noise floor or smaller detection range. However, the drawbacks of such a configuration come in the form of its compactibility and applicability to specific detection environments. For example, unless a bent sensing probe can be fabricated, this sensing configuration would make it very difficult to compact the source and detector into a single unit. It would also be very difficult for these sensing probes to access small and hard-to-reach areas without bending the sensing probe.

The reflective configuration shown in Fig. 2-2(b) is usually for optical fibers that have their ends modified to form the sensing probe. The sensing probe is fusion spliced at one end to SMF 2 and is connected to a circulator (or coupler). Light is coupled into the circulator via SMF 1 and into the sensing probe. Perturbations to the sensing probe would modulate the light in the sensing probe. The modulated light would then be reflected along SMF 2 into the circulator and coupled into SMF 3 and then the detector. Such a sensing configuration would allow the sensing probe to access hard to reach areas without having to physically modify the sensing probe, allowing it to access most environments. However, a large input intensity from the source or a detector with a low noise floor would be required as the intensity of the light coupled into the detector is usually very low due to losses at the sensing probe. For both configurations, any perturbations in the ambient environment would result in instantaneous real-time modulation of the propagating light which would be registered on the detector.

2.2 Optical Fiber Gratings

Diffraction gratings are defined as gratings in any arrangement which would result in a variation in amplitude or phase of any incident wave [24]. Similarly, an optical

Fig. 2-3 Schematic of a grating of period Λ inscribed within the core of an optical fiber.

waveguide with a periodic variation in RI along its length would impose a variation in phase on an electromagnetic wave propagating through it and hence behave like a grating [25]. Hence optical fibers with periodic variations in RI of the fiber core can behave as gratings. A schematic of such a grating is shown in Fig. 2-3. Optical fiber gratings can exist in many forms and the difference in grating types would arise from the period Λ of these RI variations within the fiber core.

2.2.1 Long Period Fiber Gratings (LPFGs)

The LPFG, first reported in 1996, is a single mode fiber (SMF) with a periodic modulation in refractive index (RI) in a short segment of the fiber core [26]. Unlike the Bragg grating, the period of modulation is usually in the order of 100 μm to 1 mm [27]. The grating can be fabricated by four processes: single-beam internal technique, dual-beam holographic technique, amplitude mask technique and point-by-point fabrication technique [25].

The sensitivity of the LPFG to perturbations in its ambient environment is largely dependent on the difference in propagation constants between its core mode (LP_{01}) and its forward propagating cladding modes (LP_{0m}) in the cladding [27] as shown in Fig. 2-4. Assuming a symmetric fiber with a symmetric grating, as the propagating light in the core of the SMF is perturbed by the grating, energy from the LP_{01} mode (solid arrow) would be coupled to the LP_{0m} modes (dashed arrows). This coupling is specific and would only occur with the satisfaction of the following phase matching condition [26]:

$$\beta_{01} - \beta_{clad}^m = \frac{2\pi}{\Lambda}, m = 1, 2, 3... \quad (2.1)$$

Fig. 2-4 Schematic of the behavior of the fundamental mode (solid arrow) on interaction with a LPFG and the observable transmitted spectrum. The dashed arrows indicate the cladding modes.

where β_{01} and β_{clad}^m are the propagation constants of the fundamental mode and the forward propagating cladding modes, respectively. The forward propagating modes would decay rapidly leaving several attenuation bands at certain discrete wavelengths, and these attenuation bands can be observed as dips in the output spectrum of the transmitted light as shown in Fig. 2-4. The wavelengths at which these attenuation bands can be observed are referred to as the resonant wavelengths λ_{res} and are given by [28]:

$$\lambda_{res} = [n_{eff,core}^{01}(\lambda_{res}, n_{core}, n_{clad}) - n_{eff,clad}^{0m}(\lambda_{res}, n_{clad}, n_{env})] \Lambda \quad (2.2)$$

where $n_{eff,core}^{01}$ and $n_{eff,clad}^{0m}$ are the effective RIs of both the fundamental core mode and the forward propagating cladding modes, respectively. n_{core} , n_{clad} and n_{env} are the RIs of the core, cladding and ambient environment of the LPFG, respectively.

Over the last two decades, the response of the LPFG to variations in RI, strain and temperature of the ambient environment have been theoretically [29-34] and experimentally [27, 35-39] studied. The response of the LPFG to variations in RI of the

ambient environment would depend on whether the RI of the ambient environment is at a value lower than that of the fiber cladding, approximately equal to the fiber cladding, or above that of the fiber cladding.

For the range of RIs below that of the fiber cladding, the cladding modes would experience reflection and refraction at the fiber cladding-ambient environment interface [30] similar to that shown in Fig. 1-2. In this case, equation (2.2) would hold and it can be seen that the resonant wavelengths would vary if the effective indices of the core mode and the forward propagating cladding modes varied. Since the core mode is confined within the core of the fiber, it is unsuceptible to any variations in RI of the ambient environment. Hence only the effective indices of the forward propagating cladding modes would be affected by any variations in RI of the ambient environment, directly contributing to the variation in the resonant wavelengths [29]. As the RI of the ambient environment increases, a corresponding red shift in the resonant wavelength would be observed [27, 29].

For RIs at approximately that of the fiber cladding, the amplitude of the attenuation band at the resonant wavelengths would decrease at an exponential rate as the value of the RI approaches that of the fiber cladding [27]. This occurs as more and more of the energy is coupled to radiation modes [30].

For RIs above that of the fiber cladding, the equation (2.2) is no longer valid as there would be no reflection of energy at the fiber cladding-ambient environment interface. All the energy is coupled from the confined core mode to the radiation modes. In this case, the attenuation band would be observed to reappear at a slightly higher resonant wavelength and increase in magnitude as the RI of the ambient environment increases [29, 30].

As can be seen from equation (2.2), the resonant wavelengths would be affected both by the RI of the ambient environment as well as by the grating period. Hence, simultaneous variations in RI, temperature and strain of the ambient environment would result in a variation in the wavelengths of the attenuation bands, and it would be impossible without further improvements in sensing setup to de-couple relevant information for the individual variations.

Since light in the LPFG is coupled only from the core mode to the forward propagating cladding modes and no backward propagation of light occurs, the sensing configuration would generally be that of the transmissive configuration. However, it should be noted that the LPFG can also work in the reflective configuration if a highly reflective mirror is coated on the reflective end of the sensor [40-42]. A high reflectance mirror deposited at the fiber tip would greatly enhance the amount of reflectance which would lead to a higher intensity reading in the reflectance spectrum. This however would incur additional fabrication and procedural costs and complexity.

2.2.2 Fiber Bragg Gratings (FBGs)

FBGs are commonly used in optical fiber communication systems as wavelength filtering devices [43]. In the fields of sensing, they have been applied as effective thermocouples [44] and strain gauges [45, 46]. Like LPFGs, FBGs also consist of periodic RI perturbations formed through the exposure of the fiber core to a high intensity optical interference pattern along a short segment of the optical fiber [47]. The conventional FBG works based on the satisfaction of the phase matching condition between the forward and backward propagating LP_{01} modes [48] resulting in a reflection spectrum [49] as shown in Fig. 2-5. The strongest coupling between these two modes would occur at the Bragg wavelength which can be expressed as [47]:

Fig. 2-5 Schematic of the behavior of the fundamental mode (solid arrow) on interaction with a FBG and the observable transmitted and reflected spectra. The dotted arrow indicates the back reflected mode.

$$\lambda_{Bragg} = 2n_{eff}\Lambda \quad (2.3)$$

where n_{eff} is the effective index of the LP_{01} mode and Λ is the period of the grating. As can be seen from the figure, the forward propagating light (solid arrow) in the optical fiber core is reflected (dotted arrow) by the RI variations forming the grating, resulting in a detectable peak at the Bragg wavelength in the reflected spectrum [47]. On the other hand, the transmitted spectrum will display an attenuation band at the Bragg wavelength itself. Perturbations like strain and temperature would increase or decrease the grating period and thus cause a shift in the wavelength position of the reflected peak or transmitted attenuation band. Conventionally, the reflected peak is monitored for these perturbations due to the impracticality of detecting an attenuation band which might be obscured by the noise floor in a detector. Hence most FBG sensors work in the reflection configuration.

As can be seen from Fig. 2-5, the coupling of the energy of the forward propagating core mode (solid arrow) to the backward propagating core mode (dotted arrow) occurs only within the core of the optical fiber. Hence no light is interacting with the ambient environment, which is one of the reasons that FBGs are seldom used for RI sensing. This can however be resolved by reducing the diameter of the cladding around the region of the grating to allow the evanescent field of the core mode to interact with the ambient environment [50]. This is usually done by etching the fiber cladding with a hydrofluoric (HF) acid solution [51-54]. Other methods for accessing the evanescent field would be to use specialized sensor setups [55] or inscribe the grating in the cladding [56]. The drawbacks of utilizing FBGs in RI sensing lie in the need for cladding removal which usually involves polishing or the handling of the very toxic and corrosive HF solutions.

2.2.3 Grating variants

The fabrication processes of optical fiber gratings can be modified in certain ways to give rise to several grating-based sensing probes as shown in Fig. 2-6. One of these new variations involve the cascading of two or more LPFGs on a single SMF. For a two LPFG cascade, the fundamental mode (solid arrow) propagating through the SMF would be diffracted by the first grating and excite the higher order cladding modes (dashed arrows) [57]. At the second LPFG, the cladding modes are recombined with the core mode [58]. The result would be an interference fringe pattern in the transmission spectrum similar to that of a LPFG, but with interference fringes within each attenuation band [57]. Cascaded LPFG sensors have been observed to be as versatile as LPFGs in terms of their ability to detect perturbations in the ambient environment like curvature [57], temperature [58-60], strain [57, 60], and RI [61]. The advantages of cascaded

Fig. 2-6 Schematic of the cascaded LPFGs, tilted FBG and superstructure grating and the behavior of the fundamental mode on interaction with these sensing probes.

LPFGs lie in their ability to offer application-based design flexibility depending on whether a high sensing resolution or a high sensitivity is required by tuning the distance between the LPFGs [59, 60].

Another of these grating variants is that of a superstructure fiber grating (SFG). SFGs, are like LPFGs, only that the length of the RI modulations is much longer than that of the distance between the RI modulations [62, 63]. These gratings possess both the properties of LPFGs and FBGs. Like the LPFG, a SFG would couple the forward propagating core mode (solid arrow) to the forward propagating cladding modes (dashed arrows) which would decay in the cladding, resulting in the formation of several attenuation bands in the transmitted spectrum [64-66]. Like FBGs, the forward propagating core mode would also couple with the backward propagating core mode (dotted arrow) at various resonant wavelengths [61]. This would result in the formation of several equally spaced peaks in the reflected spectrum [63]. SFGs have been shown to be able to be sensitive to perturbations like strain [64, 65], pressure [66] and temperature [64, 66] in the ambient environment.

Tilted fiber Bragg gratings (TFBGs) are another of these grating variants where a Bragg grating is inscribed in a SMF but blazed at an angle from the fiber axis [67]. This blazed grating would cause reflections of the incoming light like a FBG. Unlike the FBG, these reflections would be off the fiber axis [68]. This results in an enhancement in the coupling of the energy of the forward propagating core mode (solid arrow) to the backward propagating cladding modes (dashed arrow) but reduces the amount of coupling to the backward propagating core mode (dotted arrow) at certain phase matched wavelengths [69, 70]. The backward propagating cladding modes will attenuate quickly and will not be observable in the reflection spectrum, but can be observed as numerous attenuation bands at wavelengths below that of the Bragg wavelength in the transmission spectrum [67, 71]. TFBGs are much more versatile than their FBG counterparts in their ability to detect more environmental perturbations. Apart from strain and temperature [72], TFBGs can also detect variations in RI of their ambient environment [70-67].

2.3 Tapered Fiber

As shown in Fig. 2-7, a tapered fiber is a fiber where the diameter of a short segment of an optical fiber is reduced in size. The fabrication of a tapered fiber usually involves heating a segment of the fiber to be tapered and slowly pulling the ends of the fiber apart to get a thin elongated region. The resultant taper profile would depend on the heating as well as the speed at which the ends were pulled apart. Tapered fibers can be classified as adiabatic and non-adiabatic tapers [75].

Fig. 2-7 Schematic of a tapered fiber with the down-taper, tapered and up-taper regions indicated.

Adiabatically tapered fibers have long and gradual down-taper and up-taper transitions. The gradual tapers result in little or no loss in energy of the propagating light to the surroundings and as a result, the output spectrum of such a taper would be similar to that of the input spectrum. Such a taper has limited sensing applications. Henceforth, all mention of tapered fiber sensors in this thesis would be referring to non-adiabatic tapers unless otherwise stated.

Non-adiabatic tapers have more abrupt down-taper and up-taper transitions as compared to adiabatically tapered fibers. This abrupt diametric variation of the fiber profile would result in the formation of a modal interference spectrum in the output [76]. As the fundamental mode propagating through the SMF enters the down-taper region, higher order modes will be excited [77]. Most of the energy from the fundamental mode will be coupled to the HE_{11} and HE_{12} modes in the tapered region [76] with the coupling efficiency dependent on the relative phase of the modes [75]. The excited modes will propagate together with the fundamental mode through the tapered region, which can be assumed to be a coreless multimode region [78]. The propagating modes will interfere and recombine at the up-taper region resulting in an interference spectrum at

the output. The output spectra would be sinusoidal in nature and can be simplified to that of the interference of two dominant modes [76]:

$$I = I_{11} + I_{12} + 2\sqrt{I_{11}I_{12}} \cos(\Delta\beta L) \quad (2.4)$$

$$\Delta\beta = \frac{\lambda(U_2^{\infty 2} - U_1^{\infty 2})}{4\pi n_{clad} a^2} e^{-\frac{2}{V}} \quad (2.5)$$

$$V = \frac{2\pi a}{\lambda} \sqrt{n_{clad}^2 - n_{env}^2} \quad (2.6)$$

where I_{11} , I_{12} and are the intensities of the power coupling between the HE_{11} and HE_{12} modes, respectively. L is the length of the tapered region while U_1^{∞} and U_2^{∞} are the asymptotic values of the U parameters. a , λ , n_{clad} and n_{env} are the radius of the tapered region, wavelength of light propagating through the tapered region, RI of the fiber cladding and RI of the ambient environment, respectively.

From the equation (2.4) to (2.6), it can be seen that a variation in RI of the ambient environment would result in the variation of the effective index of the propagating modes in the tapered region [79] resulting in a variation in the output spectra. In general, a wavelength shift of the interference spectrum would be observed as the RI of the ambient environment varied [75, 76]. Apart from their common application as highly sensitive RI sensing probes [75-77, 79-82], there has also been ongoing research into their applicability as strain gauges [75] and thermocouples [75, 81]. Although they have been shown to have a very high sensitivity, the main drawback of tapered fibers as sensing probes is the low mechanical strength of the tapered region.

Tapered fiber sensors can work in both the transmissive and reflective configurations. The choice of sensing configuration would be application specific and

the only difference is that the extinction ratio in the output spectrum would be larger for a tapered fiber in the reflective configuration [76].

2.4 Optical Fiber Interferometers

Optical fibers can be modified in certain ways to produce sensing probes that mimic the behavior of various free space interferometers. Various specialty fibers can be spliced to SMFs to form optical versions of Fabry-Perot interferometers (FPIs) [83-91], Mach-Zehnder interferometers (MZIs) [4, 92-100], Michelson interferometers (MIs) [40-42,101-103] and the Sagnac interferometers (SIs). The last type of interferometer, exclusive in nomenclature only to this thesis, is that of the multimode interferometers (MMIs) [104-111]. In the following subsections, the optical fiber FPIs, MZIs, MIs and MMIs will be described in further detail. This would be followed by an introduction of two optical fiber interferometers fabricated from specialty fibers consisting of the photonic crystal fiber (PCF) and the multimode fiber (MMF).

2.4.1 Fabry-Perot Interferometer (FPI)

The FPI, sometimes referred to as the Fabry-Perot etalon [83], is an interferometer that works based on the interference of multiple reflected and transmitted light beams from two parallel reflecting surfaces within a cavity [112]. Optical fiber FPIs can be classified as intrinsic FPIs (IFPIs) [83-86] and extrinsic FPIs (EFPIs) [87-91]. IFPIs are interferometers where the cavity is also a fiber [112]. The cavity can be fabricated by fusion splicing a specialty fiber between two SMFs [83-85] or fusion splicing a short fiber segment with reflective coatings at both ends of the fiber segment to a SMF [86]. EFPIs on the other hand have cavities that are external to the system and not made up of the fiber material. These EFPIs can be fabricated by micro machining an air cavity [87-89] or a groove [87] within the fiber itself, or by aligning two fibers

with a short space between them to get an air cavity [91]. Many fringes would be present in the output of these interferometers as a result of the multiple reflections occurring within the cavity and any perturbations in the environment of the specialty fiber or cavity would lead to a variation in the spectral pattern of these fringes.

2.4.2 Mach-Zehnder Interferometer (MZI)

A MZI is an interferometer which works on the principle of interfering two light beams which pass through the interferometer only once. One beam of light would serve as the reference arm while the other would be the sensing arm. Both beams of light will interfere to form an interference pattern which would vary when the sensing arm is exposed to perturbations from the ambient environment [4]. The optical fiber MZI works in the same way as well where light propagating through a SMF is divided by various mechanisms into the reference arm and sensing arm and recombined to give an interference spectrum at the output [92]. This is usually done by splicing a specialty fiber (like an all-silica fiber, for example) which would initiate multi modal excitation resulting in light propagating in the core and the cladding and result in an interference spectrum in the output [92-100]. The light propagating in the specialty fiber would be ‘split’ into the isolated core mode (reference arm) and higher order modes (sensing arm) which are susceptible to perturbations from the ambient environment [92].

2.4.3 Michelson Interferometer (MI)

A MI is similar to that of a MZI except that the beams in each arm are reflected and thus pass through the interferometer twice [4]. Similarly, the optical fiber MI works based on the excitation of modes in a specialty fiber [101, 102], grating [40-42] or tapered fiber [103] which are then reflected at the fiber end-face, usually coated with a high reflectance material. The sensing operation is similar to that of the MZI. The

effective sensing area is however doubled due to the use of reflection, which is beneficial for certain sensing probes which perform better for a longer light interaction path length.

2.4.4 Multimode Interferometer (MMI)

The last type of optical fiber interferometer that does not belong to any of the previously mentioned groups is the multimode interferometer (MMI). These interferometers usually fabricated by fusion splicing a specialty fiber to a SMF [104-109] or modifying the existing fiber structure [110, 111]. The propagating light from the SMF enters the sensing region and excites the higher order modes [104-109, 111]. The interference of these modes will result in the formation of an interference spectra consisting of several peaks and dips [106]. The difference between these type of interferometers and that of the MZI and MI is that both the core and excited modes would be modulated by perturbations in the ambient environment.

2.4.5 Specialty Fiber Interferometers

Specialty fibers are fibers that differ in structure and dimensions to that of the standard SMF fiber. These fibers have special light confinement and propagating properties and can thus be applied as interferometers. In recent years, many of these specialty fibers like the thin core fiber [96, 101, 109, 113], coreless fiber [114], multicore fiber [115, 116], photonic crystal fiber (PCF) [92, 97, 116-119] and multimode fiber (MMF) [104-106, 120, 121] have been shown to exhibit good sensing characteristics. In the following subsections, two specialties fibers, namely the PCF and the MMF will be discussed in further detail.

2.4.5.1 Photonic Crystal Fiber (PCF)

First reported in 1996, a photonic crystal fiber (PCF) is generally a length of fused silica fiber consisting of a periodic array of air holes arranged in a hexagonal pattern running along its length [122]. There are in general, two different types of PCFs as shown in Fig. 2-8. The first type PCF consists of a solid silica core surrounded by an air silica cladding, and the light guiding mechanism through the fiber is via modified total internal reflection (TIR) [123]. The other type of PCF consists of a hollow core with an air-silica cladding, and the light guiding mechanism is through the photonic bandgap effect [123]. To fabricate a sensing probe, the most common way is to fusion splice a short segment of a PCF between two single mode fibers (SMF) [117, 118] as shown in Fig. 2-9. The fusion splicing process will result in a tapering and collapse of the air holes at the splice point, leading to the formation of a solid silica region between the SMFs and PCF at both ends [123]. Due to the properties of the PCF, such a sensing probe would be sensitive to variations in RI and strain in its ambient environment [117-119].

Fig. 2-8 Schematic of (a) a solid core and (b) a hollow core photonic crystal fiber (PCF). The uncolored circles indicate the positions of the air holes.

Fig. 2-9 Schematic of a solid core photonic crystal fiber (PCF) fusion spliced between two SMFs to form a sensing probe. The core mode, excited higher order cladding modes and the evanescent waves of the excited higher order cladding modes are indicated by the solid, dashed and dotted arrows, respectively.

The PCF sensing probe usually works in the transmissive configuration [118, 119] but can also work in the reflection configuration if one of the SMF ends is perfectly cleaved [125] or made to reflect [108, 126]. Light propagating through the SMF (solid arrow in Fig. 2-9) is diffracted as it enters the collapsed region [92, 116, 119]. In modal theory, this is the equivalent to the diffraction or broadening of the mode, resulting in the excitation of higher order cladding modes in the PCF [92]. Both the core mode and the excited higher order cladding modes (dashed arrow in Fig. 2-9) will propagate through the PCF segment, but with different propagation constants and hence different optical path lengths [92]. At the end of the PCF segment, the propagating modes will once again be diffracted and will interfere and recombine with each other and this can be observed from the formation of several interference peaks and dips in the resulting transmitted spectrum. The resulting transmitted light can be expressed as [97]:

$$T = E_{core}^2 + \sum_{i=1}^m E_i^2 + 2 \sum_{i=1}^m [E_{core} E_i \cos \frac{2\pi(n_{core} - n_i)L}{\lambda}] + 2 \sum_{i=1}^{m-1} \sum_{j=i+1}^m [E_i E_j \cos \frac{2\pi(n_i - n_j)L}{\lambda}] \quad (2.7)$$

where E_{core} , E_i and E_j are the energies of the core mode, the excited i^{th} order cladding modes and the excited j^{th} order cladding modes, respectively. n_{core} , n_i and n_j are the effective indices of the core mode, excited i^{th} order cladding modes and excited j^{th} order cladding modes, respectively. m , L and λ are the number of excited higher order cladding modes, length of the PCF fiber segment and the operating wavelength of the source, respectively.

It can be observed that such a sensing probe is similar to that of the MZI. The core mode and the excited higher order cladding modes can be treated as the two arms of a MZI. The difference lies in the working principle where the output of the MZI would rely on the difference in physical path lengths of the two arms while that of the PCF sensing probe would rely on the difference in optical path lengths [92]. Hence in this thesis, all further reference to a sensing probe with the same structure as that shown in Fig. 2-9 would be referred to as a PCF-MZI.

The sensing mechanism of the PCF-MZI relies on the interaction of the evanescent waves (dotted arrows in Fig. 2-9) at the cladding-ambient environment interface of the excited higher order cladding modes with the ambient environment. As the RI of the ambient environment varies, this would result in a variation in the effective indices of the excited higher order modes. The effective index of the core mode would remain unchanged as it is isolated from the ambient environment [119]. From equation (2.7), it can be seen that any perturbation in the ambient environment would lead to a variation in the effective indices of the cladding modes and cause a variation in the

cosine terms, resulting in a shift in wavelength of any spectral features in the output spectrum.

2.4.5.2 Multimode Fiber (MMF)

The MMF is an optical fiber that allows more than one mode to propagate through it at any time. The number of allowed modes would depend on the V parameter, given by [1]:

$$V = \frac{2\pi a}{\lambda} \sqrt{n_{core}^2 - n_{clad}^2} \quad (2.8)$$

where a and λ are the optical fiber core radius and wavelength of the propagating light, respectively. n_{core} and n_{clad} are the RIs of the core and cladding, respectively. In a communications system, the large core radius of a MMF allows for the easier coupling of light using a more cost effective light emitting diode (LED) as compared to a SMF which requires a laser source [1]. The disadvantage of using a MMF arises from the

Fig. 2-10 Schematic of one type of multimode fiber multimode interferometer (MMF-MMI). The fundamental mode and the excited higher order modes are indicated by the solid and dashed arrows, respectively.

propagation of many modes within the fiber which results in intermodal dispersion [1]. Although the propagation of many modes through the fiber may be detrimental to a communications system, it can be beneficial for sensing applications.

For sensing applications, a short segment of a MMF is usually fusion spliced to a SMF at one [104, 106, 120] or two SMFs at each end [105, 121] to form a MMI. The sensing configuration would generally depend on whether the MMF is fusion spliced at one end (reflective) or at both ends (transmissive). Fig. 2-10 shows the schematic of a MMF-MMI fabricated through the fusion splicing of a MMF to a SMF and cleaving the MMF to a particular length to form the sensing probe. Light propagating from the SMF (solid arrow) would excite the higher order modes (dashed arrow) in the MMF [104]. The excited higher order modes would co-propagate with the fundamental mode along the MMF segment. Like the PCF, the excited higher order modes would propagate with different propagation constants from that of the fundamental mode and this difference would lead to the formation of an interference spectra consisting of several interference peaks and dips in the reflected output. For the MMF-MMI shown in Fig. 2-10, the excited higher order modes and the fundamental mode would be modulated by perturbations in RI, temperature or strain in the ambient environment and the effects of these perturbations can be observed in the behavior of the interference peaks and dips in the output spectrum. Variations in RI of the ambient environment would modulate the intensity of all light at the end-face of the MMF leading to a variation in the intensity of the back-reflected fundamental and excited modes. The amount of this reflectance R is governed by the well-known Fresnel reflection equation:

$$R = \left(\frac{n_{MMF} - n_{env}}{n_{MMF} + n_{env}} \right)^2 \quad (2.9)$$

where n_{MMF} and n_{env} are the RIs of the MMF core and ambient environment, respectively. From the equation, it can be seen that for $n_{env} < n_{MMF}$, the value of R would decrease as the value of n_{env} increased until $n_{env} = n_{MMF}$, where R would become zero. From there, R would increase as n_{env} increased further.

Such MMF-MMIs have been applied as strain [105, 121], RI [106] and temperature [104, 121] sensing probes with good sensitivity and resolution. One of the advantages of using MMF-MMIs as the sensing probe is that of their simple fabrication. Unlike the fusion splicing of a PCF to a SMF, as long as their diameters match, MMFs can be easily fusion spliced to a SMF using the same splicing parameters as that of fusion splicing two SMFs together. Another advantage of using a MMF is that they are already commercially available and no special fiber drawing techniques are required to fabricate them.

2.5 Un-cladded Fiber Sensors and Surface Plasmon

Resonance

As most of the light in an optical fiber propagates through the core of the fiber, little or no light gets to penetrate the cladding and interact with the ambient environment. Hence, various novel point sensing probes involving the partial or complete removal of the cladding region of various optical fibers have been proposed. This can be done by immersing a short segment of the fiber in a HF acid solution to etch away the cladding or by polishing one side of the fiber until part of the core of the fiber is exposed to the ambient environment as shown in Fig. 2-11. In the absence of a cladding, the evanescent waves of the light propagating in the exposed core will interact with any perturbations in the ambient environment which would lead to a modulation of the intensity or

Fig. 2-11 Schematic of (a) an optical fiber cable with its cladding removed through etching and (b) a side polished fiber.

wavelength of the evanescent wave [127, 128]. The amount of interaction would depend on the penetration depth of the evanescent wave and also on the length of the exposed core [129].

Such sensing probes are highly sensitive and have been used to measure variations in RI [129-133] and temperature [134] of the ambient environment. The drawback of using these sensing probes lie in their fabrication. The etching of the cladding requires the use of HF acid which is a very toxic and corrosive acid. Also, like the tapered fiber, the removal of the cladding will result in a decrease in the mechanical strength of the sensing probe, making it more fragile.

To further enhance the sensitivity of these sensing probes, metallic materials like gold and silver, have been deposited over the exposed fiber core. This is most commonly referred to as Kretschmann's configuration [135] as shown in Fig. 2-12. Fig. 2-12(a) shows the original Kretschmann's configuration where laser light entering a prism is refracted at the air-prism interface. The refracted light would propagate through the prism and interact with a glass slide coated with the metallic material. No refraction occurs at the prism-glass slide interface as the glass slide and prism are made up of the

Fig. 2-12 Schematic of (a) Kretschmann's configuration for surface plasmon resonance (SPR) and (b) an optical fiber SPR sensing probe with a metallic layer deposited over the exposed fiber core.

same material and thus have the same RI. The light would interact with the metallic material at an angle θ . Surface plasmon resonance (SPR) would occur when the value of θ is the same as that of the resonance angle, leading to the excitation of surface plasmons in the metallic material [136].

The same effect can be observed in an optical fiber with a metallic material deposited onto its exposed core as shown in Fig. 2-12(b). Evanescent waves propagating in the fiber core would interact with the metal at the metal-fiber interface and upon satisfaction of the resonance angle condition, excite surface plasmons in the metal [137] resulting in SPR. These excited surface plasmons would generate a strong localized

electromagnetic field which would scatter light, resulting in the formation of plasmon absorption bands at certain resonant wavelengths [138]. Variations in RI of the ambient environment would affect the wavelength at which this plasmon absorption occurs resulting in a variation in resonant wavelength of the absorption bands [139]. Such SPR sensing probes have been mainly used as RI sensors and have been demonstrated using etched fibers [138, 140], side polished fibers [139, 141, 142] and also through the deposition of gold nanoparticles onto the end-face of an optical fiber [143].

2.6 Sensing Probes with Material Overlays

Apart from gold and silver, the last two decades have seen an increase in the amount of research into the integration of other metallic and nonmetallic materials with the currently available optical fiber sensing probes to enhance their sensitivity [144]. These sensing probes were generally used to detect any variations in the relative humidity (RH) and RI of the ambient environment. The materials can be deposited over LPFGs [145-155] and other grating variants [156-159], onto the cleaved end-face of a fiber to form a FPI [160-163], over a PCF-MZI [164-168] or as a sensitive overlay which varies in reflectance upon exposure to various perturbations in the ambient environment [169-173].

The sensing mechanism of these sensing probes can work in several ways. One involves the absorption of the analyte which results in an increase in thickness or effective RI of the overlay material. Another way is through the direct altering of the properties of the overlay material in response to the RI of its ambient environment. These variations in material properties would result in a variation in the behavior of the light propagating in the fiber and lead to a variation in its wavelength or intensity.

These materials can be deposited in a variety of ways such as electrostatic self-assembly (ESAM), various physical and chemical vapor deposition (CVD) methods, the Langmuir-Blodgett (LB) method, dip coating or drop casting, spray coating or optical deposition. Each of these methods have their own advantages and disadvantages and are catered to the type of material as well as the morphological requirements of the deposited overlays.

2.7 Other Environmental Sensing Applications

This chapter has provided a description of the more commonly demonstrated optical fiber sensing probes, focusing mainly on their RI sensing capabilities. As mentioned in the previous sections, several of these sensing probes can also be used to detect various perturbations like temperature, strain and pressure in the ambient environment. Various grating-based sensing probes [174, 175], tapered fibers [176-178], fiber interferometers [98, 99, 106, 179, 180], SPR-based sensing probes [181] and sensing probes with material overlays [173] have demonstrated temperature sensing behaviors with sensitivities between 0.98 pm/°C [173] and 2.3 nm/°C [99] for temperatures below 250 °C. Strain and bend sensing probes have also been demonstrated with fiber interferometers using specialty fibers like MMFs and coreless fibers [100] and PCFs [97, 182-184]. Fiber interferometers fabricated with cavities [89] have also exhibited pressure sensing behavior up to a few mBar.

Apart from this, optical fiber sensors have also been used in chemical and bio-sensing applications. Sensing probes using PCFs [165, 166, 185] and fiber interferometers [186] have demonstrated humidity detection capabilities for humidity levels in the range of 20-95 %RH (where RH is the relative humidity). Various chemical compounds like ammonia [187-189], chlorophyll [190], chloride ions [41], and volatile

organic compounds [191-195], have been detected at concentrations as low as 0.1 ppm (where ppm is the parts-per million). Other applications in the fields of chemical and bio-sensing also involve the use of optical fiber sensing probes as “noses” for odour detection [196] and pH sensors [197].

In recent years, specialty optical fibers fabricated from soft glass materials with the capability to operate in the mid-infrared (MIR) region of the electromagnetic spectrum have also demonstrated chemical sensing capabilities. Chemical compounds leave unique “fingerprints” in the form of absorption bands at specific wavelengths in the MIR region when illuminated with light [198]. These chemical compounds would lead to the partial absorption of the evanescent wave at the fiber-environment interface when they are absorbed onto the fiber, resulting in a reduction in the amount of light transmitted at a particular wavelength in the transmitted spectrum [199]. Various chemical compounds such as chlorinated hydrocarbons [200], pathogens that can be found in food [201], carbon dioxide [202] and various alcohols [201] have been detected at concentrations as low as 5 ppb (parts-per billion) using these soft glass fibers.

2.8 Fundamental Noise Limits

The fundamental noise limits for an optical fiber sensor are similar to those that exist in optical fiber communication systems. For sensing systems, noise would be present in the light source, optical fiber and detector (see Fig. 2-2). Fluctuations in intensity of the optical source would lead to relative intensity noise (RIN) [1]. These fluctuations arise due to spontaneous emission in the output of the laser or variations in temperature [203]. RIN would limit the sensitivity and resolution of sensing probes that encode perturbations in the ambient environment in the intensity of their output. To

negate the effects of this RIN, a reference arm is usually included in the sensing configuration to measure the relative intensity change.

Transmission of light from a coherent source through a MMF may also lead to the generation of modal noise [2]. The coherent light propagating through the MMF would initially excite a few modes. A time delay less than the coherence time of the source between the modes would result in the back and forth coupling of power between the propagating modes and lead to a fluctuation in intensity of the output. This form of noise can be eliminated through the use of incoherent sources like light emitting diodes which can uniformly couple power into the various excited modes due to their wider spectral output range.

Detectors typically consist of photodiodes and these photodiodes would experience shot noise, noise due to the dark current and thermal noise [203]. Shot noise is a fundamental limit of the photodiode and arises from the statistical nature of the collection of photons on the photodiode and the subsequent generation of electrons by the photodiode. The dark current is a form of residual current which arises from the generation of a current by the photodiode even when no photons are incident on it. Thermal noise arises from the thermal agitation and random motion of electrons at any particular temperature. These types of noise would increase the amount of noise in the output, leading to a decrease in the signal-to-noise ratio (SNR). For sensors which make use of low power light sources or work in the reflection configuration, this would result in a decrease in resolution of the output. Sensing probes that encode perturbations in the ambient environment through variations in intensity of their output would experience a limitation in the measurable range of the perturbation. On the other hand, sensing probes that encode these perturbations through variations in wavelength of their output would

experience poor wavelength resolution of the various spectral features which would limit their sensitivity.

2.9 Concluding remarks

This chapter has given a broad overview of optical fiber sensing probes and their applications in environmental sensing. The fabrication processes and sensing mechanisms of these sensing probes have been extensively discussed. The chapter has also introduced a novel concept involving the deposition of various materials onto these sensing probes which would serve as the basic idea behind this thesis. Apart from that, brief discussions on the other environmental sensing applications of the various sensing probes and certain fundamental noise limits which would affect these optical fiber sensors have also been presented.

Chapter 3 - Experimental Procedures and the RI Sensing Characteristics of the Tapered Fiber, PCF-MZI and MMF-MMI

As discussed in chapter 2, many optical fiber sensing probes have been demonstrated over the years. Though high in sensitivity, some of these sensing probes do face certain limitations in their sensing capabilities which can be looked into.

This chapter will first give an introduction to the experimental procedures including the equipment used, preparation of test solution, RI characterization procedure and the data collection methods used throughout the thesis. It will go on to study the behavior of the tapered fiber, PCF-MZI and MMF-MMI sensing probes to variations in RI of the ambient environment and discuss their advantages and limitations.

3.1 Experimental Procedures

The experimental procedures can be divided into several parts. The following subsections will describe the equipment used, the test solution preparation process, the characterization procedures and the methods for data analysis.

3.1.1 Optical Equipment and Components

Several light sources, detectors, specialty fibers and optical equipment are used in this thesis. A list of the more commonly used equipment including their model and abbreviation for use in this thesis is shown in Table 1. All abbreviations for equipment used henceforth, unless otherwise stated, would be referring to those stated in Table 1.

Equipment	Model	Abbreviation
Amplified Spontaneous Emission (Light Source)	Optical Link Limited CL 15-16 ASE	ASE
White Light Source	Yokogawa AQ4305	-
Tunable Laser Source	Ando AQ8203	TLS
Optical Spectrum Analyzer	Ando AQ6317B	OSA
Power Meter	Anritsu MT9810A	-
Continuous Wave Erbium Doped Fiber Amplifier	KEOPSYS	EDFA
Multimode Fiber	Thor Labs AFS105/125Y	MMF
Photonic Crystal Fiber	NKT Photonics LMA-10	PCF

Table 1 Table showing a list of commonly used equipment with their model and their abbreviation for use in this thesis.

For the optical spectrum analyzer (OSA), the wavelength resolution was set at 0.1 nm for all experiments. The intensity resolution was a fixed value of 0.01 dB.

3.1.2 Test Solution Preparation

The test solutions were prepared by first dissolving sugar in deionized (DI) water until a saturated solution was obtained. The saturated solution was then divided into equal portions and each individual portion was diluted with a different volume of DI water to obtain test solutions with distinct RIs. The RI of each test solution was determined using a commercially available digital refractometer (Kruss DR201-95) with a working resolution of 0.0001 refractive index units (RIUs).

3.1.3 Behavior Characterization Procedure

To characterize the response of the sensing probes to variations in RI of the ambient environment, the sensing probes were secured to a fiber holder and held over a rectangular glass slide. For a sensing probe working in the transmissive configuration, there is the additional requirement of ensuring that the sensing probe is held straight without any additional strain on it. Sensing probes working in the reflective configuration only need to be secured to the holder to ensure that they do not move around when immersed in the test solutions. These sensing probes were then completely immersed in approximately 1 ml of the individual test solutions which were dropped over the glass slide. After each RI measurement was carried out, the glass slide and the sensing probe were completely rinsed with DI water and allowed to dry. It was ascertained that the spectral pattern for the sensing element in air was recovered before the next measurement was carried out. All trials were carried out at an ambient temperature of approximately 23.0 ± 0.1 °C to ensure that there was no temperature induced perturbations in the external environment which would affect the RI measurements in any way. For repeatability testing, a second trial is usually carried out approximately a week from the first trial.

The effects of the slight variation in temperature on the RI of the test solution can be modelled using the Cauchy equation [204, 205]:

$$n(\lambda, T) = A(T) + \frac{B(T)}{\lambda^2} + \frac{C(T)}{\lambda^4} + \frac{D(T)}{\lambda^6} \quad (3.1)$$

where

$$A(T) = 1.3208 - 1.2325 \times 10^{-5} T - 1.8674 \times 10^{-6} T^2 + 5.0233 \times 10^{-9} T^3 \quad (3.2)$$

$$B(T) = 5208.2413 - 0.5179 T - 2.284 \times 10^{-2} T^2 + 6.9608 \times 10^{-5} T^3 \quad (3.3)$$

$$C(T) = -2.5551 \times 10^8 - 18341.336T - 917.2319T^2 + 2.7729T^3 \quad (3.4)$$

$$D(T) = 9.3495 + 1.7855 \times 10^{-3}T + 3.6766 \times 10^{-5}T^2 - 1.2932 \times 10^{-7}T^3 \quad (3.5)$$

From here the variation in RI due to an increase in temperature for a given test solution can be plotted as shown in Fig 3-1.

From Fig. 3-1, it can be seen that a 0.1 °C change in temperature would result in a 1×10^{-5} change in RI. The time taken for such a change in temperature, based on the measured intensity of the ASE source at 1550 nm, to occur was calculated to be approximately 9 hours. Since the individual sensing probes are immersed in each test solution for no more than 5 minutes at a time, laser irradiation from the individual sensing probes would have a negligible effect on the temperature of the individual test solutions.

Fig. 3-1 Plot of the variation in RI of a particular test solution due to variations in temperature of the ambient environment.

3.1.4 Data Processing and Analysis

The output spectrum of all sensing probes are presented in an intensity versus wavelength plot. Due to the nature of the OSA, the intensity axis would be in the units

of decibels per division (dB/div). For all sensing probes, the spectral feature with the highest extinction ratio and nearest to that of the telecommunications wavelength of 1550 nm was chosen for observation of its response to variations in RI of the ambient environment. This is due to the fact that the high extinction ratio would enable the easier resolution of any changes to the spectral feature and also that light propagating through silica near this wavelength would experience the least power loss. To calculate the sensitivity, the interference peak or dip corresponding to the lowest RI value is taken as the reference point and the amount of variation in intensity or wavelength of the peak at higher RI values are normalized relative to this lowest point. A fitting line is then plotted based on the relevant data and the sensitivity is calculated from there. For nonlinear fits, the sensitivity would be obtained by clustering the data which show a linear behavior within a certain RI range. The sensitivity would then be individually calculated for each cluster.

Depending on the parameter used, the sensitivity S of the individual sensing probes can be calculated using:

$$S = \frac{\Delta x}{\Delta n_{env}} \quad (3.6)$$

where x is the intensity or wavelength parameter. The units of S would be either in nanometers per refractive index unit (nm/RIU) or in decibels per refractive index unit (dB/RIU). Due to the difference in units of the sensitivity for various sensors, it is usually much more convenient to compare sensor performance based on their achievable resolution. In this thesis, the resolution will be defined as the smallest change in RI that can be measured by a particular optical fiber sensor for a given RI range. Using equation (3.6), the achievable resolution r_{sensor} can be calculated using:

$$r_{sensor} = \frac{r_{x,det}}{S} \quad (3.7)$$

where $r_{x,det}$ is the maximum detector resolution of $\mathcal{X}$. For sensors that exhibit nonlinear variations in their output to variations in RI of the ambient environment, S and r_{sensor} would be calculated based on the selected RI range.

3.2 Tapered Fiber Sensing Probe

The subsequent sub-sections would provide a description of the fabrication process of the tapered fiber sensing probe and its response to variations in RI of the ambient environment. The results would then be discussed and the achieved sensitivity and resolution would be calculated.

3.2.1 Fabrication

As mentioned in section 2.3, a tapered fiber can be fabricated by heating a short section of a short length of optical fiber and stretching the fiber to the required taper length and diameter. A schematic of the tapering setup of a commercially available optical fiber tapering machine (GPX-3000, Vytran, Morganville, NJ, USA) is shown in Fig. 3-2.

The polymer coating of a short segment of a SMF was first removed and the exposed fiber was cleaned with a methanol solution. The SMF was then held straight and secured at each end to the translation stages, with the exposed portion of the fiber aligned with the graphite filament furnace. The SMF was then held straight and secured at each end to the translation stages, with the exposed portion of the fiber aligned with the nozzle of the electric arc. The stages and the nozzle were linked to a PC control unit which was specially installed with a software to control the tapering parameters and the

Fig. 3-2 Schematic and photograph of the setup used in the fabrication of a tapered fiber.

optical fiber tapering machine. During the tapering process, the electric arc would soften the exposed segment of the SMF through heating. At the same time, the translation stages would move in opposite directions, ‘stretching’ the fiber. In order to fabricate a uniform taper, both stages would move at different speeds during the tapering process. Using this fabrication method, a tapered fiber sensor with a taper waist of approximately

10 μm was fabricated. Each end of the tapered fiber was then fusion spliced to a SMF which were then respectively connected to an ASE source and an OSA in the transmissive configuration.

3.2.2 Results

The tapered profile and the initial spectrum in a test solution with a RI of 1.3332 is shown in Fig. 3-3. The resolution bandwidth (RBW) is calculated to be approximately 3.9 nm. As shown in Fig. 3-3(a), the lengths of the down-taper region, tapered region

Fig. 3-3 (a) Schematic of the fabricated tapered fiber sensor with the taper dimensions and (b) the output spectra when the tapered region is immersed in a test solution with a RI of 1.3332.

and up-taper region are 1 mm, 6 mm and 3 mm, respectively. The reason for the difference in lengths of the down-taper and up-taper regions is due to the fact that in order to achieve a taper waist and length with the stated dimensions, the translation stages have to move apart at different speeds resulting in a difference in dimensions of the transition regions. The tapered region was then immersed in a test solution with a RI of 1.3332 and the output spectrum can be seen in Fig. 3-3(b). As can be seen from the figure, the tapering of the SMF resulted in the formation of several interference peaks and dips in the output spectrum of the tapered fiber.

Fig. 3-4(a) shows the behavior of the interference dip at a wavelength 1550.4 nm as the RI of the ambient environment varied. As discussed in section 3.1.4, the interference dip at this wavelength was chosen for refractive index (RI) sensing due to its proximity to the telecommunications wavelength of 1550 nm, which is wavelength at which the standard SMF experiences the least power loss. From the figure, it can be seen that the interference dip experienced a red shift as the RI of the ambient environment increased. This is in accordance with equations (2.4) – (2.6) and has also been experimentally verified by the authors of [75] and [76]. The magnitude of the intensity of the interference dip showed a general decrease as well. However, this was not used as a measurement parameter as the magnitude of the intensity fluctuated at certain RI values and did not show any observable trend. The variation in wavelength of the interference dip to variations in RI of the ambient environment is plotted in Fig. 3-4(b) for a RI range of 1.3332 to 1.3345. The sensitivity of the tapered fiber within this RI range was calculated to be approximately 3373.4 nm/RIU. The simulation of the behavior of the fabricated tapered fiber to variations in RI of the ambient environment for the same RI range is included as well. As can be seen, the experimental results do

(a)

(b)

Fig. 3-4 (a) Behavior of the interference dip at a wavelength of 1550.4 nm to variations in RI of the ambient environment. (b) Amount of red shift in the wavelength of the interference dip within the RI range of 1.3332-1.33445.

not differ much from the simulated data. Slight deviations between the simulated and experimental data are likely due to fluctuations from the equipment and environment throughout the experimental process. From here, the minimum achievable resolution of the tapered fiber sensing probe within the RI range of 1.3332-1.3345 was calculated to

be approximately 2.96×10^{-5} . However, the maximum error was calculated to be approximately 2.00×10^{-5} RIU which is larger than the achievable resolution. Hence the minimum achievable resolution of the sensor would be 2.00×10^{-5} RIU. The resolution was also recalculated based on the sensitivity of interference dip at 1530.6 nm. From here, the deviation in the achieved sensitivity was calculated to be approximately 6% which shows that the resolution is repeatable.

3.3 PCF-MZI Sensing Probe

The fabrication process and the responses of two PCF-MZIs to variations in RI of the ambient environment would be described in the following sub-sections. This would be followed by a discussion of the results and the achieved sensitivity and resolution of both PCF-MZIs would be calculated. The PCF used was the LMA-10 model which was purchased from NKT Photonics. The LMA-10 is made up of a solid silica core with a hexagonal array of air holes surrounding a solid core. As such, light would be expected to be guided through the fiber core by modified total internal reflection. The core diameter, cladding diameter and attenuation at 1550 nm are approximately 10 μm , 125 μm and 2 dB/km, respectively. The PCF used is similar to that described in [206] and has been shown to exhibit a sensitivity of approximately 1.2 nm/degree of bending. Such a drift in wavelength due to bending would be sufficient to affect the RI measurements. Hence care was taken to ensure that the interferometer was secured and held straight throughout the measurement procedure.

3.3.1 Fabrication

The polymer coating of a short segment of the PCF was first removed using a razor. The exposed segment of the PCF was then cleaned with methanol. Each end of the PCF was then manually fusion spliced to a SMF to form the PCF-MZI. Using these

steps, two PCF-MZIs with PCF lengths of 2.7 cm and 3.0 cm were fabricated. A labelled microscopy image of one of the fusion spliced points of one of the PCF-MZIs is shown in Fig. 3-5. As discussed in section 2.4.5.1, the fusion splicing of a SMF to a PCF would result in the formation of a collapsed region between the SMF and a PCF which has been labelled in Fig. 3-5.

The output spectra of the two PCF-MZIs in air with a RBW of 4.1 nm and 6.8 nm, respectively, are shown in Fig. 3-6. As can be seen in Fig. 3-6 and also previously discussed in section 2.4.5.1, the interference of the excited higher order modes with the core mode resulted in the appearance of several interference peaks and dips in the output spectrum.

Fig. 3-5 Microscopy image of the region where a SMF is fusion spliced to the PCF segment.

(a)

(b)

Fig. 3-6 Output spectra of the two PCF-MZIs in air for a PCF length of (a) 2.7 cm and (b) 3.0 cm, respectively.

3.3.2 Results

Fig. 3-7(a) shows the variation in the interference dip at 1531.84 nm of the PCF-MZI with a PCF length of 2.7 cm. As can be seen from the figure, there is a fluctuation in intensity of the interference dip. This fluctuation is due to many reasons and one of

the possible explanations for the fluctuations in intensity can be inferred from equation (2.7). From the equation, it can be seen that the transmitted spectrum is a summation of the contributions from the core mode and the excited higher order cladding modes. This coupled with the non-flat band output of the ASE source would result in the random

Fig. 3-7 Behavior of the interference dip at a wavelength of 1531.84 nm to variations in RI of the ambient environment for a PCF-MZI with a 2.7 cm PCF segment. (b) Amount of red shift in the wavelength of the interference dip within the RI range of 1.33-1.42.

variation in intensity of the interference dip. Hence, the variation in intensity would not be suitable for characterizing the sensing probe to variations in RI of the ambient environment. From Fig. 3-7 (a), it can be seen that there was a distinct red shift of the interference dip as the RI of the ambient environment increased in value. Similar to that of the tapered fiber, the magnitude of the intensity of the interference dip was not used as a measurement parameter as it did not exhibit any observable trend to variations in RI of the ambient environment. The variation in wavelength of the interference dip to variations in RI of the ambient environment is shown in Fig. 3-7(b). The nonlinear behavior of the PCF-MZI is approximated by two linear lines corresponding to the range of RIs to be studied. From here, the sensitivity of the PCF-MZI with a 2.7 cm PCF segment can be calculated to be approximately 144.1 nm/RIU and 433.0 nm/RIU for the RI ranges of 1.33-1.38 and 1.38-1.42, respectively. The achievable resolution for this PCF-MZI for the RI range of 1.33-1.38 was calculated to be approximately 6.94×10^{-4} RIU. The maximum error was calculated to be approximately 5.33×10^{-4} RIU which is lower than the achieved resolution. Hence the achievable resolution should be taken as 6.94×10^{-4} RIU. The maximum error in accuracy was calculated to be approximately 4.65×10^{-3} RIU which shows that the maximum error is still within the claimed measurement accuracy. The deviation in sensitivity was calculated to be approximately 2.3% which implies that the accuracy is repeatable.

The interference dip at a wavelength of 1579.00 nm for the PCF-MZI with a 3.0 cm PCF segment is shown in Fig. 3-8(a). It can be seen from the figure that the PCF-MZI also exhibited a distinct red shift in wavelength of the interference dip as the RI of the ambient environment increased. The magnitude of the intensity of the interference dip did not display any observable trend as well. Fig. 3-8(b) shows the variation in wavelength of the interference dip with a variation in RI. The variation in wavelength

(a)

(b)

Fig. 3-8 Behavior of the interference dip at a wavelength of 1579.00 nm to variations in RI of the ambient environment for a PCF-MZI with a 3.0 cm PCF segment. (b) Amount of red shift in the wavelength of the interference dip within the RI range of 1.33-1.42.

exhibited a similar nonlinear trend as that of the other PCF-MZI and is similarly approximated by two linear lines corresponding to the range of RIs to be studied. From here, the achieved sensitivity was calculated to be approximately 50.8 nm/RIU and 263.7 nm/RIU for the RI ranges of 1.33-1.38 and 1.38-1.42, respectively. The minimum

achievable resolution for this PCF-MZI for the RI range of 1.33-1.38 was calculated to be approximately 1.97×10^{-4} RIU. The maximum error was calculated to be approximately 9.08×10^{-5} RIU which is lower than the achieved resolution. Hence the achievable resolution for the PCF-MZI for the RI range of 1.33-1.38 is approximately 1.97×10^{-4} RIU. The maximum error in accuracy was calculated to be approximately 3.17×10^{-3} RIU and this shows that the maximum error is still within the claimed measurement accuracy. The deviation in sensitivity was calculated to be approximately 7.4% which indicates that the accuracy is also repeatable.

As can be seen from Fig. 3-7 and Fig. 3-8, both PCF-MZIs were able to show clear and distinguishable changes in wavelength as the RI of the ambient environment varied. The simulated performance of the PCF-MZIs are also shown in Fig. 3-7(b) and Fig. 3-8(b) for the respective PCF-MZIs. As can be seen from the figures, the experimental observation follows closely to that of the theoretical prediction. Slight variations between the theoretical and experimental observations are likely due to the assumptions in the significant digits of some of the numerical values used as well as to minor fluctuations in the experimental environment during the RI measurement procedure. From here, it can be seen that the nonlinear behavior of the two PCF-MZIs discussed in this section is similar to that demonstrated in [118] and [119], thus verifying the repeatability of the system. However, the attained sensitivity and resolution for a similar range of RIs for the PCF-MZI in this thesis is higher than that presented in [119]. The difference in performance can be attributed to the type of PCF used as well as the usage of a more optimized length for the PCF segment. The similar resolution to that compared of the digital refractometer used also indicates the potential for real-time industrial applications of such a sensor.

Lastly, the referencing of the data values is based on the methodology described in section 3.1.3, where a baseline (that of the sensor in air) is established. For all experiments, it was ensured that the spectrum returned to the baseline position after each test solution was measured before measuring the next test solution. In this case, possible drifts from the baseline in the form of a difference in intensity levels would occur during the baseline re-establishment. This was however, not observed throughout the measurement process. As previously discussed, slight bending to the PCF segment would result in a shift in the wavelength. Hence, it was ensured that the interferometer was always held straight.

3.4 MMF-MMI Sensing Probe

This section would discuss the fabrication and responses to variations in RI of the ambient environment of two MMF-MMIs with different MMF segment lengths. The achieved sensitivities and resolution would be calculated and results of their wavelength stability would also be presented and discussed.

3.4.1 Fabrication

The MMF-MMIs were fabricated by first stripping one end of a SMF and a MMF of their polymer coatings. The two ends were then cleaved and fusion spliced together. The MMF was then cleaved to a length of 1.2 cm to obtain a sensing probe similar in structure to that shown in Fig. 2-10. A second MMF-MMI with a 1.5 cm MMF segment was fabricated using the same method. The length of the MMF would determine the interference length of light within the MMF segment and this interference would result in the formation of various spectral features (interference peaks or dips) at various wavelengths. Hence the lengths of MMFs are selected based on the formation of these

Fig. 3-9 Output spectrum of the two MMF-MMIs in air for a MMF length of (a) 1.2 cm and (b) 1.5 cm.

spectral features with at least one interference peak or dip occurring near the telecommunications wavelength of 1550 nm.

The MMF-MMIs were connected in the reflective configuration and their output spectrum in air, with RBW of 20.6 nm and 20.0 nm, respectively, is shown in Fig. 3-9. As can be observed from the figure, several interference peaks and dips could be

observed. The formation of these interference patterns, which has been previously discussed in section 2.4.5.2, are due to the interference of the excited higher order modes with that of the core mode. From the figure, it can be seen that the spectral feature nearest to the telecommunications wavelength of 1550 nm is that of the interference peak at 1557.04 nm and 1555.96 nm for the MMF-MMIs with MMF segments of 1.2 cm and 1.5 cm, respectively. Hence the interference peak at these wavelengths of the respective MMF-MMIs would be used as reference points for RI characterization in this case.

3.4.2 Results

The behavior of the interference peak to variations in RI for the MMF-MMIs with MMF segments of 1.2 cm and 1.5 cm are shown in Fig. 3-10(a) and Fig. 3-11(a), respectively. As can be observed, the magnitude of the intensity of the interference peak decreased as the RI of the ambient environment increased. The change in intensity for both sensors are plotted in Fig. 3-10(b) and Fig. 3-11(b), respectively. A fitting line was plotted through the data points and it can be seen from both figures that like the PCF-MZI, there is a nonlinear variation in intensity with RI of the ambient environment. This behavior can be explained by the Fresnel relation (equation (2.9)) and has also been experimentally verified in [106]. The achieved sensitivity for the MMF-MMI with a 1.2 cm MMF segment was calculated to be approximately 93.19 dB/RIU and 216.25 dB/RIU for the RI ranges of 1.33-1.40 and 1.40-1.46, respectively. For the MMF-MMI with a 1.5 cm MMF, the achieved sensitivity was calculated to be approximately 91.96 dB/RIU and 153.31 dB/RIU for the RI ranges of 1.33-1.40 and 1.40-1.46, respectively. The minimum achievable resolution for these MMF-MMIs was calculated to be 1.07×10^{-4} for the RI range of 1.33-1.40. The maximum error in the RI range of 1.33-1.40 was calculated to be approximately 6.88×10^{-4} RIU and 3.97×10^{-4} RIU for the MMF-MMIs with MMF segments of 1.2 cm and 1.5 cm, respectively. This value is higher than that

(a)

(b)

Fig. 3-10 Behavior of the interference peak at a wavelength of 1557.04 nm to variations in RI of the ambient environment for a MMF-MMI with a 1.2 cm MMF segment. (b) Amount of variation in the intensity of the interference peak within the RI range of 1.33-1.46.

of the calculated resolution and must be considered as the detection limit. The maximum error in accuracy was calculated to be approximately 2.04×10^{-3} RIU and 1.78×10^{-3} RIU for the MMF-MDIs with MMF segments of 1.2 cm and 1.5 cm, respectively. From here, it can be seen that the maximum error is still within the claimed measurement

(a)

(b)

Fig. 3-11 Behavior of the interference peak at a wavelength of 1555.96 nm to variations in RI of the ambient environment for a MMF-MMI with a 1.5 cm MMF segment. (b) Amount of variation in the intensity of the interference peak within the RI range of 1.33-1.46.

accuracy. The deviation in the achieved sensitivity was calculated to be lesser than 4.5% in both cases which shows that the accuracy is repeatable.

The variation in wavelength of the interference peak for both MMF-MMIs is shown in Fig. 3-12. In fig. 3-12(a), the wavelength of the interference peak at 1557.04

(a)

(b)

Fig. 3-12 Wavelength behavior of the interference peak to variations in RI of the ambient environment for the MMF-MMIs with a (a) 1.2 cm MMF segment and (b) 1.5 cm MMF segment.

nm of the MMF-MMI with the 1.2 cm MMF segment fluctuated within the wavelength range of 1557.28-1558.12 nm. The wavelength fluctuation of the interference peak at 1555.96 nm of the MMF-MMI with the 1.5 cm MMF segment was within the wavelength range of 1555.96-1557.88 nm. The fluctuations for both MMF-MMIs were greater than 1 nm which highlights an inherent stability limitation for the MMF-MMIs

which can be attributed to fluctuations from the light source and the cleaved end-face of the MMF segment.

3.5 Limitations

The tapered fiber, PCF-MZI and MMF-MMI sensing probes have shown high sensitivity to variations in RI of the ambient environment. These sensors do however, have their drawbacks. Apart from issues like low mechanical strength of some of the sensing probes, they also do come with other inherent limitations brought on by their detection scheme which will be discussed in the following sub-sections.

3.5.1 Free Spectral Range

It can be seen from sections 3.2 and 3.3 that the tapered fiber and the PCF-MZIs make use of wavelength as a measurement parameter to characterize the variations in RI of the ambient environment. It can also be seen in Fig. 3-3(b) and Fig. 3-6 that the output spectra of these sensing probes have many interference peaks and dips and the interval between any two adjacent peaks or dips is known as the free spectral range

Fig. 3-13 Behavior of all interference dips within the wavelength range of 1510-1630 nm to variations in RI of the ambient environment for the 10 μ m tapered fiber.

(FSR). As previously shown, each peak would shift to a particular wavelength for a particular RI value of the ambient environment. Limitations by the FSR would start to surface when the shift in wavelength exceeds the FSR. This is illustrated in Fig. 3-13 and Fig. 3-14.

Fig. 3-13 show the behavior of all the observable interference dips within the observable wavelength window to variations RI of the ambient environment for the 10 μm taper fiber sensor. To present a clearer figure, only selected data are shown in the figure. The tapered fiber sensing probe was immersed in test solutions with RIs above that of 1.3345 and the red shift in wavelength of the interference dips were recorded. The wavelength of interference dip 2 at an ambient environment RI of 1.3404 was 1569.6 nm while that of interference dip 3 at 1.3332 was 1569 nm. Hence the wavelength of interference dip 2 would have overlapped that of interference dip 3, and the sensor would not be able to distinguish between the two different ambient environment RIs. A similar occurrence can be observed between interference dips 3 and 4. The wavelength of interference dip 3 for an ambient environment RI of 1.3404 was 1588.5 nm which is the same value as that of interference dip 4 for an ambient environment RI of 1.3332. Again, the sensor would be unable to distinguish between the two different RIs of the ambient environment.

Fig 3-14 shows the behavior of all the interference dips within the observable wavelength window to variations in RI in the ambient environment of the PCF-MZIs with PCF lengths of 2.7 cm and 3.0 cm, respectively. For the PCF-MZI with a PCF length of 2.7 cm, it can be seen in Fig 3-14(a) that the wavelength of interference dip 2 at an ambient environment RI of 1.4262 was 1553.44 nm and the wavelength of interference dip 3 at an ambient environment RI of 1.3330 was 1551.52 nm. It can be seen from Fig 3-14(b) that the wavelength of interference dip 2 for the PCFI with the

(a)

(b)

Fig. 3-14 Behavior of all interference dips within the wavelength range of 1510-1630 nm to variations in RI of the ambient environment for the (a) 2.7 cm and the (b) 3.0 cm PCF-MZIs.

3.0 cm PCF segment at an ambient environment RI of 1.4213 was 1560.52 nm while the wavelength for interference dip 3 at an ambient environment RI of 1.3330 was 1558.24 nm. Hence in both cases, the wavelength position of a lower interference dip has overlapped that of the next interference dip, and the sensor would not be able to distinguish between the two different RIs of the ambient environment.

Hence the FSR limitation constrains these sensing probes to be able to continuously measure the RIs of the ambient environment only within a selected range of RIs. RI values of the ambient environment beyond this range of RIs would require the use of new reference points.

3.5.2 Noise Floor Limit

As seen in section 3.4, the interference peaks of the MMF-MMI experience a variation in intensity as the RI of the ambient environment varies. Unlike, the tapered fibers and the PCF-MZIs, these sensors would not be limited by their FSR. They are however limited by the detection range of the detector. In Fig. 3-10 (a) and Fig. 3-11(a) it can be seen that the magnitude of the intensity of the interference peak showed a distinct decrease as the RI of the ambient environment increased. For the MMF-MMI with a 1.2 cm MMF segment, it can be seen that the spectrum starts to experience distortions for ambient environment RIs above that of 1.4408. The same phenomenon is observable for ambient environment RIs above that of 1.4355 for the MMF-MMI with a 1.5 cm MMF segment.

These distortions are due to the intensity of the interference peaks decreasing to that of the detector's minimum detectable intensity level. Near this limit, the signal to noise ratio (SNR) would be very low and the output spectrum would be affected by noise and become distorted. Hence the effect of noise limits the detection of ambient environment RIs above a certain RI as well. To overcome this limitation, light sources with a higher output intensity or detectors with a lower detection limit can be used. This would however involve the use of more high cost equipment which is not in line with the objectives of a viable optical fiber sensing probe.

3.5.3 Temperature Dependence

As discussed in section 3.1.3, a 0.1 °C variation in temperature of the test solution would result in a 10^{-5} variation in the RI of the test solution. For some of the sensing probes presented in this chapter, this is an effect which would result in measurement errors. Based on the error corrected resolution presented in section 3.2.2, a temperature variation of 0.1 °C would be detectable by the tapered fiber. This would limit the performance of the tapered fiber as it would be able to detect the 10^{-5} variation in RI resulting from fluctuations in the temperature of the test solution.

PCF-MZIs have been shown to be temperature insensitive, exhibiting a linear 3 pm shift in wavelength for a 1 °C change in temperature [117]. Since the ambient environment is kept at a temperature of 23.1 ± 0.1 °C, a 0.3 pm (or 0.00003 nm) variation in wavelength would register for the amount of fluctuation. This variation in wavelength is however below the minimum resolution limit of the OSA used and would not be significantly resolvable. Apart from this, based on the maximum resolution of the PCF-MZIs presented in this chapter, a 10^{-5} flux in RI of the solution due to the fluctuation in temperature would also not be significantly resolvable by the PCF-MZIs as well.

In the case of the MMF-MMI, it has been shown that there would be a 10.8 pm variation in wavelength if the temperature of the test solution varies by 1 °C with no variation in intensity [106]. For the sensitivity and resolution of the MMF-MMI presented in this chapter, a change in temperature of approximately 15 °C of the test solution would be required before a variation in intensity would be detected. Hence the MMF-MMI would be relatively temperature insensitive under the current experimental conditions.

3.6 Concluding Remarks

This chapter has described the various experimental equipment and procedures which will be used throughout this thesis. The fabrication processes and the RI sensing characteristics of the tapered fiber, PCF-MZI and MMF-MMI sensing probes were also presented. The achieved sensitivity and minimum achievable resolution for each of these sensing probes were calculated and the limitations encountered by these sensing probes have also been highlighted.

Chapter 4 - Carbon Nanomaterial Overlays

The tapered fiber, PCF-MZI and MMF-MMI sensing probes have demonstrated good RI sensing characteristics. These sensing probes however, have been shown to experience certain limitations in their sensing performance.

As discussed in section 2.6, various sensing probes can be deposited with a material overlay to enhance their sensing performance and this chapter will explore two types of carbon nanomaterials as material overlays. The optical properties of these carbon nanomaterials, a modelling of their behavior upon deposition onto an optical fiber, their preparation procedures and deposition methods would also be discussed in this chapter.

4.1 Carbon Nanomaterials as Thin Film Overlays

Carbon nanomaterials are simply zero, one or two dimensional materials that are entirely made up of carbon [207]. In this theses, the one dimensional carbon nanotubes (CNTs) and the two dimensional graphene are explored in their applications as a material overlay to be deposited onto various optical fiber sensing probes. This would be carried out through the deposition of the various carbon nanomaterials onto the various sensing probes like the LPFGs, PCF-MZIs and MMF-MMIs. These sensing probes are selected for their mode coupling or mode excitation capabilities which would result in the formation of various spectral features that can be used as reference points during the RI measurement process. For the LPFGs, since the grating is inscribed directly into the fiber, no additional fusion splicing would affect the mechanical strength of the sensing probe. For PCFs and MMFs, their availability and low fabrication costs (especially for the MMFs), as well as their versatility in terms of their utilization in various sensing configurations (see section 2.1) would make them viable for adoption

in commercial applications. The high RIs of the deposited carbon nanomaterials would be expected to increase the continuous measurable RI range of the sensing probes and also pave the way for exploration into dual parameter sensing applications (to be discussed in section 8.1). Apart from that, these carbon nanomaterials can be further functionalized (see section 8.3) to allow certain specific analytes to bind to them, thus allowing them to be integrated into the chemical and biomedical sensing industries as well. The optical properties and applications of these carbon nanomaterials for optical fiber sensing applications will be discussed in the subsequent sub-sections.

4.1.1 Graphene

Graphene is a flat, two dimensional honeycomb lattice of sp^2 hybridized carbon atoms [208, 209] as shown in Fig. 4-1. It was first produced in 2004 through mechanical exfoliation of pyrolytic graphite [210]. Despite its one atom thickness, graphene has been shown to exhibit unique optical properties. The transmittance T_G and reflectance R_G of a monolayer of graphene can be expressed as [211]:

$$T \equiv \left(1 + \frac{2\pi G}{c}\right)^{-2} = (1 + 0.5\pi\alpha)^{-2} \quad (4.1)$$

$$G = \frac{e^2}{4\hbar} \quad (4.2)$$

$$\alpha = \frac{e^2}{\hbar c} \quad (4.3)$$

$$R \equiv 0.25\pi^2\alpha^2T \quad (4.4)$$

where G is the high-frequency conductivity for Dirac fermions and α is the fine structure constant. The universal physical constants c , e and $\hbar$ are the speed of light

in vacuum, elementary charge and the reduced Planck's constant, respectively. From here the absorption of a monolayer of graphene can be calculated to be approximately 2.3% and this absorption increases linearly with the number of layers [209, 211].

The difference between graphene and graphite lie in the number of layers of carbon atoms. Hence to reliably determine whether a particular sample is graphene or graphite, the thickness of the sample must be determined and this can be done by Raman spectroscopy. The Raman spectra for graphite and graphene would contain two bands, a G band at approximately 1580 cm^{-1} and a G' or 2D band at approximately 2700 cm^{-1} [212]. For monolayer graphene, the location of the G band would be approximately 3-5 cm^{-1} above that of graphite [208]. The difference between both spectra lies in the height of the G' band. The G' band of graphene would be approximately four times higher than its G band but that of graphite would only be half the height [212]. For graphite, there may be an additional D band at approximately 1350 cm^{-1} which indicates the presence of broken symmetry, broken edges or high density defects, which is common in the morphology of graphite samples [208].

Due to their unique optical properties, monolayer and multilayer graphene have also been incorporated with optical fiber sensors. In the last decade, many RI sensing

Fig. 4-1 Schematic of the two dimensional honeycomb lattice structure of graphene. Carbon atoms are indicated by the colored circles.

probes based on the deposition of monolayer or multilayer graphene onto cladding removed fibers [212, 214], fiber gratings [215] and SPR-based fiber sensors [216, 217] have been demonstrated. Apart from optical fiber RI sensing probes, graphene-based optical fiber temperature [218] and pressure [219, 220] sensing probes have also been demonstrated through the adherence of a monolayer or multilayers of graphene onto the end-face of an optical fiber.

4.1.2 Carbon nanotubes (CNTs)

CNTs are one dimensional tubes formed from rolled up graphene which are approximately 1 μm and 1 nm in length and diameter, respectively [207]. They can be classified as multi-walled CNTs (MWCNTs) [221] and single-walled CNTs (SWCNTs) [222]. MWCNTs consist of several rolled up graphene sheets, each with different diameters, enclosing each other. SWCNTs on the other hand are individual rolled up graphene sheets which are not enveloped by larger nanotubes. Depending on their structure, SWCNTs can be either metallic or semiconducting in nature. The difference in forms arise from how the SWCNTs are rolled up from a single graphene sheet. This is referred to as its chirality and can be expressed as a chiral vector $\vec{C}_h$ given by [223]:

Fig. 4-2 Schematic of the honeycomb lattice structure of graphene to be folded along the chiral vector C_h . Carbon atoms are indicated by the colored circles. The chiral vector of a (4, 1) CNT is shown in the figure.

$$\vec{C}_h = n\vec{a}_1 + m\vec{a}_2 \quad (4.5)$$

where the unit vectors within the hexagonal honeycomb lattice are denoted by $\vec{a}_1$ and $\vec{a}_2$ as shown in Fig. 4-2. n and m are integers. The chiral vector indicates the connection between two crystallographically equivalent sites on the graphene sheet [223]. The nature of the SWCNTs can then be determined using the following relation:

$$|n-m| \bmod 3 \begin{cases} = 0, & \text{metallic} \\ \neq 0 & \text{semiconducting} \end{cases} \quad (4.6)$$

If the magnitude of the difference between the integers n and m is divisible by 3, then the SWCNT would be metallic in nature, otherwise it would be semiconducting in nature. In the case of Fig. 4-2, a chiral vector of a (4, 1) CNT is shown. Based on equation (4.6), it can be calculated to show that a SWCNT formed from the rolling up of a graphene sheet with this chiral vector would be metallic in nature.

A mixture of metallic and semiconducting CNTs are usually used when they are deposited on optical fiber sensing probes. CNTs have been shown to be nanomaterials with a relatively high RI and unique optical properties [224] and have mainly been deposited onto fiber ends through the LB method [225-229], dip coating [230] and drop casting [231] methods for volatile organic compound detection. Apart from that, they have also been deposited onto etched FBGs for protein detection [232] and humidity sensing [233].

4.2 Modelling of Carbon Nanomaterial Overlays

Fig. 4-3 Schematic of the deposition of carbon nanomaterials onto the surfaces and end-faces of the LPFGs, PCFIs and MMFIs.

The carbon nanomaterials were deposited onto LPFGs [155], PCF-MZIs [167, 168] and onto the end-face of the MMF-MMI as shown in Fig 4-3. The amount of reflectance of the carbon nanomaterial overlay can be modelled using the Transfer Matrix Method [4, 234]. The reflectance and transmittance properties of light propagating through the fiber with a carbon nanomaterial overlay in a particular ambient environment can be simplified into the three-layer model as shown in Fig. 4-4.

The optical fiber region, carbon nanomaterial overlay and ambient environment are each modelled as a layer in the three-layer model with RIs n_F , $\tilde{n}_C$ and n_{env} , respectively. The $\sim$ above a variable indicates that the variable is a complex number. The interface between the fiber-carbon nanomaterial and carbon nanomaterial-ambient environment layers can each be represented by a transition matrix:

$$H_{F-C} = \frac{1}{\tau_{F-C}} \begin{pmatrix} 1 & r_{F-C} \\ r_{F-C} & 1 \end{pmatrix} \quad (4.7)$$

$$H_{C-env} = \frac{1}{\tau_{C-env}} \begin{pmatrix} 1 & r_{C-env} \\ r_{C-env} & 1 \end{pmatrix} \quad (4.8)$$

where τ_{n-m} and r_{n-m} are the transmission and reflection coefficients of the interface between layers n and m , respectively. For the case of normal incidence of light as shown in Fig. 4-4(a), the reflection coefficients can be expressed as:

Fig. 4-4 Modelling of the fiber, carbon nanomaterials and ambient environment as individual layers with different RIs based on (a) based on the normal incidence of light at the interface between the fiber and carbon nanomaterial layers and (b) light interacting with the carbon nanomaterial layer at an incident angle.

$$r_{F-C} = \frac{n_F - \tilde{n}_C}{n_F + \tilde{n}_C} \quad (4.9)$$

$$r_{C-env} = \frac{\tilde{n}_C - n_{env}}{\tilde{n}_C + n_{env}} \quad (4.10)$$

Electromagnetic energy propagating through the carbon nanomaterial layer would incur an additional phase which can be represented by the propagation matrix:

$$L_C = \begin{pmatrix} e^{-i\beta_C} & 0 \\ 0 & e^{+i\beta_C} \end{pmatrix} \quad (4.11)$$

$$\beta_C = \frac{2\pi}{\lambda} n_C d_C \cos \theta_C \quad (4.12)$$

where β_C , λ , d_C and θ_C are the propagation constant, wavelength of the propagating electromagnetic energy, thickness of the carbon nanomaterial and the incident angle of the light on the carbon nanomaterial layer, respectively. The positive or negative exponentials indicate the direction of propagation. From here, the three layers can be represented by the following stack matrix:

$$S = \begin{pmatrix} S_{11} & S_{12} \\ S_{21} & S_{22} \end{pmatrix} = H_{F-C} L_C H_{C-env} \quad (4.13)$$

Substituting equations (4.7), (4.8) and (4.11) into equation (4.13) and simplifying:

$$S = \frac{1}{\tau_{F-C} \tau_{C-env}} \begin{pmatrix} e^{-i\beta_C} + r_{F-C} r_{C-env} e^{+i\beta} & r_{C-env} e^{-i\beta_C} + r_{F-C} e^{+i\beta} \\ r_{F-C} e^{-i\beta_C} + r_{C-env} e^{+i\beta} & r_{F-C} r_{C-env} e^{-i\beta_C} + e^{+i\beta} \end{pmatrix} \quad (4.14)$$

The total energy propagating within the fiber and ambient environment layers at any time can be denoted by E_F and E_{env} , respectively. As shown in Fig. 4-4(a), these

in turn can be divided into the reflected ($E_{n,R}$) and transmitted ($E_{n,T}$) energy components, where n denotes the layer. Since there is no reflected energy in the ambient environment layer, the energy propagating in the layers can thus be represented using the following matrices:

$$E_F = \begin{pmatrix} E_{F,R} \\ E_{F,T} \end{pmatrix} \quad (4.15)$$

$$E_{env} = \begin{pmatrix} 0 \\ E_{env,T} \end{pmatrix} \quad (4.16)$$

Hence the relationship between the total energy propagating in the fiber layer and the total energy propagating in the ambient environment layer can be expressed as:

$$E_F = SE_{env} \quad (4.17)$$

Substituting equations (4.15) and (4.16) into (4.17):

$$\begin{pmatrix} E_{F,R} \\ E_{F,T} \end{pmatrix} = \begin{pmatrix} S_{11} & S_{12} \\ S_{21} & S_{22} \end{pmatrix} \begin{pmatrix} 0 \\ E_{env,T} \end{pmatrix} = \begin{pmatrix} S_{12}E_{env,T} \\ S_{22}E_{env,T} \end{pmatrix} \quad (4.18)$$

From equation (4.18), the reflection coefficient of the carbon nanomaterial layer can be defined as:

$$r_C = \frac{E_{F,R}}{E_{F,T}} = \frac{S_{12}}{S_{22}} \quad (4.19)$$

Substituting the values of the matrix in equation (4.14) into equation (4.19):

$$r_C = \frac{r_{C-env}e^{-i\beta} + r_{F-C}e^{+i\beta}}{r_{F-C}r_{C-env}e^{-i\beta} + e^{+i\beta}} \quad (4.20)$$

The total amount of reflectance of the carbon nanomaterial layer can then be simplified from equation (4.20) and defined as:

$$R_C = |r|^2 = \left| \frac{r_{F-C} + r_{C-env} e^{-i\tilde{k}_C}}{1 + r_{F-C} r_{C-env} e^{-i\tilde{k}_C}} \right|^2 \quad (4.21)$$

$$\tilde{k}_C = \frac{4\pi\tilde{n}_C d_C}{\lambda} \quad (4.22)$$

In the case of LPFGS and PCF-MZIs, light propagating through the fiber interacts with the carbon nanomaterial layer as shown in Fig 4-4(b). To account for this non-normal incidence, equations (4.9) and (4.10) have to be modified to include the incident and transmitted angles:

$$r_{F-C} = \frac{n_F \cos \theta_{I,F} - \tilde{n}_C \cos \theta_{T,C}}{n_F \cos \theta_{I,F} + \tilde{n}_C \cos \theta_{T,C}} \quad (4.23)$$

$$r_{C-env} = \frac{\tilde{n}_C \cos \theta_{I,C} - n_{env} \cos \theta_{T,env}}{\tilde{n}_C \cos \theta_{I,C} + n_{env} \cos \theta_{T,env}} \quad (4.24)$$

where the $\theta_{I,F}$, $\theta_{T,C}$, $\theta_{I,C}$ and $\theta_{T,env}$ are the incident angle of light in the fiber layer, transmitted angle of light in the carbon nanomaterial layer, incident angle of light in the carbon nanomaterial layer and the transmitted angle of light in the ambient environment, respectively. By substituting equations (4.9), (4.10), (4.23) and (4.24) into equation (4.21), the general behaviour of R_C as n_{env} varied was simulated using MATLAB and is shown in Fig. 4-5. The complete program code is given in Appendix A and Appendix B for the reflectance due to the interaction of light with the carbon nanomaterial overlay at normal incidence and at an incident angle of 30° , respectively. The range of n_{env} and the thickness of the carbon nanomaterial overlay was set as 1.33-4 and 40 nm,

respectively. The value of $\tilde{n}_C$ was set as $2.71 + 1.71i$ based on that measured in [235]. The complex RI of the carbon nanomaterial overlay would result in an inherent wavelength dependent optical absorption [236]. This can be observed from equation 4.21 where the expansion of $\tilde{n}_C$ would lead to a wavelength dependent exponential term due to the complex component of $\tilde{n}_C$. As can be seen from Fig. 4-5, R_C decreases as n_{env} increased from 1.33 to 2.79 for the case of normal incidence. For the case of light interacting with the cladding at an angle of 30° , n_{env} increased to 2. From here, it can be seen that the possible range of continuously measurable RIs would be up to 2.79 and 2.46 for the respective cases before reference points in the spectra have to be obtained, which is in contrast to that of the bare sensing probes as previously discussed in section 3.5.

Fig. 4-5 Simulation based on equation (4.21) of the variation in reflectance as the RI of the ambient environment varied for the case of light interacting with the carbon nanomaterial overlay at normal incidence and at an incident angle of 30° .

4.3 Preparation of solutions

The carbon nanomaterials had to be dispersed into a liquid medium before deposition onto the various sensing probes. CNT solution was obtained from collaborators from the National University of Singapore while the graphene solution was prepared in-house following the steps in [237]. The general procedure for preparing both solutions is as follows. CNT powder or Kish Graphite were first individually dispersed into dimethylformamide (DMF) solution. DMF was chosen for its ability to overcome the strong van der Waals forces between CNTs and graphene flakes [238]. The solutions were next ultrasonicated in a water bath for over 30 hours in the case of CNTs, and over 72 hours in the case of Kish graphite. For the CNT solution, it was to ensure that CNTs were evenly dispersed throughout the entire solution. In the case of Kish graphite, it was to attempt to obtain nano-sized sheets of graphene [237]. The solutions were then centrifuged to remove the larger and more massive particles and flakes and the resulting supernatant was decanted. The end result was a solution with the smallest CNTs or the smallest and thinnest graphene flakes. 1 ml of each solution was transferred onto a silicon wafer and left to dry. The weight of the silicon wafer before and after the deposition of the carbon nanomaterial solutions were measured. From here, the concentration of the CNT and MG solutions were calculated to be 0.9 g/L and 10.8 g/L, respectively.

4.4 Raman Characterization of the Carbon Nanomaterial Solutions

The Raman and absorption spectra of the CNT used is shown in Fig. 4-6. From Fig. 4-6(a), the D, G and G' bands occur at 1343 cm^{-1} , 1595 cm^{-1} and 2677 cm^{-1} , respectively. The small magnitude of the D band indicates that the CNTs used are of relatively good

quality with very little defects. Fig. 4-6(b) shows the absorption spectra of the CNT dispersed in DMF, which is similar to that of denatured CNT [239, 240]. The CNTs were prepared by the catalytic carbon monoxide (CO) disproportionation method as described in [241]. CO was introduced to a nickel magnesium oxide precursor at approximately 800 K. The products of the reaction were immersed in nitric acid to remove any particulate matter from the catalyst. The purified product was then washed with de-

Fig. 4-6 (a) Raman spectra of the CNT solution with the D, G and G' bands marked. (b) Absorbance spectra of the CNT in DMF.

(a)

(b)

Fig. 4-7 (a) Raman spectra of the MG solution with the D, G and G' bands marked. (b) Scanning electron microscope (SEM) image of the MG flakes on a silicon wafer.

ionized water and dried at approximately 400 K. the CNTs obtained in this way are approximately 10 μm multi-walled with diameters in the range of 7-35 nm [242].

To determine the nature of the graphene solution, a small portion of the solution was sprayed (see section 4.5.1) over a silicon wafer. Raman spectroscopy (WITec) was carried out and the Raman spectra is shown in Fig. 4-7(a). Three bands at 1349 cm^{-1} , 1578 cm^{-1} and 2719 cm^{-1} can be seen. These correspond to the D, G and G' bands, respectively. The heights of the G and G' bands show that the prepared solution is

similar to that of bulk graphite or multilayer graphene (MG) described in [210]. The appearance of the D band is due to the presence of defects in the MG flakes incurred during the ultra-sonication process which is to be expected.

The wafer was then observed under a scanning electron microscope (SEM, JEOL JSM-5910LV). As shown in Fig. 4-7(b), graphene flakes were randomly and evenly distributed and the morphology can be assumed to be similar to that of the MG overlay deposited onto the surface of the PCF-MZI (to be discussed in chapter 6).

4.5 Deposition Methods

Two relatively simple and cost effective methods were explored for the deposition of the carbon nanomaterials onto the various optical fiber sensing probes. The first is a spray coating method which makes use of pressurized air to coat the surface of the fiber with a carbon nanomaterial overlay. The second is an optical deposition method where high intensity light was used to deposit the carbon nanomaterials onto the cleaved end-face of the optical fiber. Both methods involved only the cold transfer of the carbon nanomaterials onto the sensing probe with no need for any additional heat or chemical treatment [243]. The carbon nanomaterials would adhere strongly onto the fiber surface through van der Waal's forces.

The choice of deposition method would depend on the sensing probe. For sensing probes like the LPFG and the PCF-MZI, a large area has to be deposited with carbon nanomaterials and coupling high intensity light into the sensing probe would increase the probability of damaging it. Hence for these sensing probes, the spray coating method would be preferred. On the other hand, due to the small surface area of the end-face of the MMF-MMI, it would not only be difficult to control the amount of carbon nanomaterials deposited but also result in solution wastage if the spray coating

method were used to deposit the carbon nanomaterials onto the end-face of the MMF-MMI. Hence for this sensing probe, the optical deposition method would be a more practical approach.

4.5.1 Spray Coating

The spray coating method is one of the simplest methods for depositing a material overlay over a substrate [243-245]. The setup for depositing the carbon nanomaterials over an optical fiber sensing probe is shown in Fig. 4-8. The sensing probe is first connected at one end to an ASE and an OSA at the other end in the transmissive configuration. The purpose of the ASE and OSA is to monitor any variations in the observable spectral features throughout the deposition process. These spectral features are that made up of the interference peaks and dips similar to that shown in Fig. 3-6. These spectral features would vary in wavelength and intensity throughout the carbon nanomaterial deposition process. Hence, the amount of deposited carbon nanomaterials

Fig. 4-8 Schematic of the setup for spray coating of a carbon nanomaterial overlay onto an optical fiber sensing probe.

would have to be deposited in a controlled manner to ensure that certain spectral features around the telecommunications wavelength of 1550 nm would remain resolvable.

The sensing probe is held straight over a silicon or glass substrate the purpose of which is to characterize the thickness of the deposited carbon nanomaterial overlay. The spray gun (Harder and Steenbeck, Evolution Two in One) is connected to a nitrogen gas supply, and the pressure of the gas flowing into the spray gun is set at 5 Psi. 5 ml of the carbon nanomaterial solutions was transferred to the solution cup of the spray gun. The solution was then spray coated onto the surface of the fiber for a total duration of 5s. This was carried out by aligning the nozzle of the spray gun directly over the sensing probe. To deposit the carbon nanomaterials, a valve is released and nitrogen gas would be pumped into the spray gun and disperse the carbon nanomaterials over the sensing probe via the nozzle. The spray gun would be moved in a horizontal direction along the length of the sensing probe to ensure that the entire sensing probe is coated with the carbon nanomaterials. At the same time, a heat source is placed at an angle to the nozzle of the spray gun to evaporate the DMF that is released with the carbon nanomaterials. The fiber holder was rotated after one spray cycle and the spray coating process was repeated for the same duration. The orientation of the fiber holder was then reversed and the afore described spray coating process was repeated. Upon completion of the deposition process, the thickness of the deposited nanomaterial overlay would be estimated by placing the silicon wafer or glass substrate in a surface profiler (Dektak³). The surface profiler would measure the thickness of the deposited carbon nanomaterials and from there, the thickness of the deposited carbon nanomaterials on the surface of the fiber can be estimated.

One important point to note about this method is that although the various parameters like gas pressure and spray duration can be controlled to give a homogeneous

deposited overlay, it is very difficult to ensure that the homogeneity extends over the entire surface. Hence the thickness of the deposited carbon nanomaterial has to be measured by simultaneously spraying a silicon or glass wafer and estimating the thickness from the scanning electron microscope image of the wafer.

4.5.2 Optical deposition

Optical deposition is another method for depositing carbon nanomaterials onto the cleaved end-face of a fiber [246, 247]. In this thesis, optical deposition was used to deposit an overlay of CNTs onto the cleaved end-face of the MMF-MMI. The setup for optical deposition is shown in Fig. 4-9. The MMF-MMI was vertically immersed into the prepared CNT solution. Light from a TLS operating at a wavelength of 1550 nm was amplified to 19 dBm by an EDFA and coupled via a circulator into the MMF-MMI. The back-reflected light was then coupled into the circulator and into the power meter. An instantaneous spike in magnitude of the intensity of the back-reflected light would indicate the deposition of CNTs onto the end-face of the MMI. The data series for the progress of deposition is shown in Fig 4-10. Data values were manually recorded at each

Fig. 4-9 Schematic of the setup for optical deposition of a CNT overlay onto the end-face of a MMF-MMI.

interval of time and recorded to two significant figures. From the figure, it can be seen that the reflected intensity (circles) increased to approximately -34 dBm when the EDFA (diamonds) was switched on to give an output intensity of 19 dBm. An instantaneous spike in magnitude of the intensity to -21 dBm of the reflected light was observed approximately 6 s after the EDFA was switched on. This would indicate that CNTs have been deposited onto the MMF-MMI. The deposition process was stopped 5 s after this change in intensity occurred, and the CNT-deposited MMF-MMI was removed from the solution. The back-reflected intensity at this point was approximately -38 dBm which indicates that CNTs have been deposited onto the end-face of the MMF-MMI.

A schematic of the deposition mechanism is shown in Fig. 4-11. As can be seen from the figure, the high intensity of the light entering the solution would result in four physical forces which would affect the movement of CNTs around the fiber region. The first force is the gradient force where CNTs would move across an intensity gradient, travelling from a region of low light intensity to a region of higher light intensity. This force acts in the direction normal to the direction of the light beam exiting the fiber. The

Fig. 4-10 Variation in intensity with time for the optical deposition process.

Fig. 4-11 Schematic of the optical deposition mechanism showing the four forces acting on the CNTs.

next force is the force due to convection. The high intensity of the light would heat up the solution, resulting in the generation of convection currents. These induced currents would result in the movement of the CNTs upwards towards the fiber. The third force is the scattering force. This is the only force that acts against the deposition. This force acts in the propagation direction of the beam and would scatter the CNTs away from the fiber end-face. The last force, which is the dominant force, is thermophoresis. This force involves the movement of CNTs across a temperature gradient, from the heated solution towards the colder fiber end-face [248].

Like the spray coating method, the thickness of the deposited CNT overlay can be controlled by varying the intensity of the input light and deposition duration. However, unlike the spray coating method, a secondary wafer cannot be deposited at

the same time. Hence the thickness of the deposited CNT overlay have to be estimated through a microscopy image of the deposited end-face.

4.6 Concluding Remarks

This chapter has introduced the two carbon nanomaterials to be deposited onto the various sensing probes. It has also modelled their optical properties using the Transfer Matrix Method. The preparation of these carbon nanomaterials and the methods used to deposit these carbon nanomaterials onto the sensing probes have also been described extensively. The material in this chapter, particularly that from section 4.2, will serve as a theoretical framework for the experimental work which will be carried out in chapters 5, 6 and 7.

Chapter 5 - CNT-Deposited LPFG

In this chapter, a LPFG would be spray coated with CNT as the material overlay. The sensing mechanism and the fabrication of this CNT-deposited LPFG would first be presented. This would be followed by a detailed discussion of the behavior of the spectral features in its output spectrum to variations in RI of the ambient environment. A discussion of the sensing performance, repeatability and stability of the sensing probe will also be provided at the end of the chapter.

5.1 Sensing mechanism of the CNT-Deposited LPFG

A schematic of the sensing probe and the light propagation mechanism is shown in Fig. 5-1. The CNT overlay can be treated as an additional dielectric overlay of finite thickness with a higher RI than that of the cladding of the LPFG and the behavior of the LPFG would be similar to that when it is in a medium of higher RI. As such, the phase matching condition of equation (2.1) is no longer satisfied. In this case, the sensing probe can be modelled as a cylindrical waveguide with four layers where the LP modes, cross-coupling coefficients and self-coupling coefficients can be calculated [249]. From

Fig. 5-1 Schematic of the light propagation mechanism of a CNT-deposited LPFG.

here, assuming that the RI profile of the fiber does not vary azimuthally, the power in each mode can be expressed as [31, 250]:

$$p_{0n} = \frac{\beta_{0n}}{2\omega\mu_0} \int_{r=0}^{2\pi} d\phi \int_{r=0}^{r_{core}} R_{0n}^2(r) dr, n=1, 2, 3... \quad (5.1)$$

$$\omega = k_0 c \quad (5.2)$$

$$k_0 = \frac{2\pi}{\lambda} \quad (5.3)$$

where β_{0n} is the propagation constant of the n^{th} LP mode, μ_0 is the permeability of free space, r_{core} is the radius of the fiber core, ϕ is the azimuthal angle and $R(r)$ is the radial variation of the modal field. C is a constant defined as the speed of light in vacuum and λ is the free space wavelength.

As can be seen in Fig. 5-1, at the interface between the fiber cladding and CNT overlay, a portion of the power in the cladding modes (dashed arrow) would be multiply reflected and transmitted (dotted arrows) [30]. Based on the three-layer model described in section 4.2, the overall reflectance would be governed by equation (4.21). From here, a variation in the magnitude of the attenuation bands in the output spectrum of the CNT-deposited LPFG would be expected as the RI of the ambient environment varied.

5.2 Fabrication of the CNT-Deposited LPFG

The amplitude mask technique using an excimer laser (Lambda Physik) was used to inscribe the long period grating into the core of a SMF fiber. To increase its photosensitivity, the SMF was hydrogen loaded for 10 days before the grating inscription was carried out. As shown in Fig. 5-2, an amplitude mask was placed at a

Fig. 5-2 Schematic of the setup for the inscription of a long period grating into a SMF.

pre-calibrated distance from the hydrogen loaded optical fiber and a Krypton Fluoride (KrF) laser with a laser output wavelength of 248 nm was scanned normally across the amplitude mask. The laser power was set at 200 mJ with a repetition rate of 10 Hz. The length and period of the grating were 2.6 cm and 320 μm , respectively. The laser beam was diffracted into several beams by the amplitude mask and the constructive and destructive interference of these beams on the fiber core resulted in the formation of a periodic RI pattern in the fiber core [25]. The spray coating of a CNT overlay onto the LPFG was achieved through the spray coating method described in section 4.5.1 where the fiber was rotated throughout the spray coating process, ensuring that the CNTs were evenly distributed over the entire surface of the LPFG. The volume and concentration of the CNT solution used were 5 ml and 0.9 g/L, respectively. From here the thickness of the CNT was estimated, using the method described in section 4.5.1, to be approximately 5 microns.

Fig. 5-3 Optical microscope image of the LPFG (a) before and (b) after the deposition of the CNT overlay.

The optical microscope images focusing on a small section of the LPFG before and after deposition of the CNT overlay are shown in Fig. 5-3. As can be observed from the figure, CNTs were uniformly deposited over the entire surface of the LPFG.

Fig. 5-4 shows the output spectrum of the CNT-deposited LPFG. The RBW was calculated to be approximately 11.2 nm. The output spectrum before and after deposition remained relatively the same with only a decrease in magnitude of the attenuation band at a resonant wavelength of 1463.2 nm after the deposition of the CNT overlay. This decrease in magnitude indicates that the deposition of CNTs onto the surface of the fiber resulted only in losses with no other spectral effects. This can be explained by treating the CNT-deposited LPFG as a LPFG surrounded by a medium with a higher RI as described in the previous section. The high RI of CNT would result in a situation similar to that described in section 2.2.1 for a LPFG exposed to an ambient environment with RI higher than that of the fiber cladding. In this case, there would be a change in distribution of the cladding modes where part of the light in the cladding is confined in

the CNT overlay. The confined light would result in the development of radiation modes [251], which would lead to the formation of attenuation bands at various resonant wavelengths [252].

Fig. 5-4 Behavior of the attenuation band at a wavelength of 1463.2 nm to variations in RI of the ambient environment.

5.3 Sensing Behavior of the CNT-Deposited LPFG

The CNT-deposited LPFG was connected to a white light source and an OSA in the transmissive configuration. In the subsequent sub-sections, the results of the sensing behavior of the CNT-deposited LPFG would be presented and a short discussion of the results would be given.

5.3.1 RI Characterization

The RI characterization procedure was carried out and the response of the CNT-deposited LPFG to variations in RI of the ambient environment is shown in Fig 5-5. The behavior to the range RIs shown in the figure was obtained continuously with no need for any reference points to be taken. From the figure, it can be seen that there were clear

Fig. 5-5 Behavior of the attenuation band at a wavelength of 1463.2 nm to variations in RI of the ambient environment.

and distinct variations in the magnitude of the intensity of the attenuation band at 1463.2 nm. The intensity of the attenuation band was measured to be approximately -74 dBm. At this intensity level, the detector would be susceptible to some of the noise limitations as discussed in section 2.8. Hence the slight ripple-like fluctuations in the attenuation band can be overcome by using higher performance detectors. As can be seen, the magnitude of the intensity of the attenuation band decreased as the RI of the ambient environment increased.

5.3.2 Wavelength Behavior

The variation in wavelength in relation to variations in RI of the ambient environment is shown in Fig. 5-6. The wavelength remained relatively constant, deviating within the wavelength range of 1460.80-1463.2 nm as the RI of the ambient environment varied. This deviation is insufficient to draw any conclusions on the encoding of RI variations of the ambient environment in the wavelength. The slight

Fig. 5-6 Variation in wavelength of the attenuation band at a wavelength of 1463.2 nm to variations in RI of the ambient environment.

fluctuations observed can be attributed to errors from the detector and also the strain on the LPFG caused by minor differences in the volume of each test solution used. From here and section 5.3.1, it can be seen that there is little wavelength dependence of the attenuation band with RI of the ambient environment implying that any variation in RI of the ambient environment can be solely encoded by the variation in intensity of the attenuation band.

5.3.3 Repeatability and Sensitivity

To test the repeatability of the sensing probe, a second RI characterization trial was carried out. The variations in intensity of the attenuation band of both trials (diamonds for the first trial, circles for the second trial) were normalized and are shown in Fig. 5-7. As can be seen, the change in intensity for both trials increased as the RI of the ambient environment increased. A single fitting line was plotted through the experimental data from both trials and it can be seen that there is a nonlinear relation between the intensity of the attenuation band and the RI of the ambient environment.

Fig. 5-7 Variation in intensity of the attenuation band at a wavelength of 1463.2 nm to variations in RI of the ambient environment for two separate trials.

From section 4.2 and equation (4.21), it can be seen that this nonlinearity is to be expected. The achieved sensitivity was calculated to be approximately 30.7 dB/RIU and 46.8 dB/RIU for the RI ranges of 1.33-1.38 and 1.38-1.42, respectively. Based on these values, the minimum achievable resolution for the CNT-deposited LPFG for the RI range of 1.33-1.38 was calculated to be approximately 3.23×10^{-4} . The maximum error due to the repeatability was calculated to be approximately 0.4 dB and this error can be attributed to fluctuations from the light source and errors from the detector. These results indicate that the sensor has good repeatability over time and is capable of demonstrating distinct variations in intensity as the RI of the ambient environment varied. The maximum error for the RI range of 1.33-1.38 was calculated to be approximately 7.14×10^{-4} , which is higher than the minimum achievable resolution. Hence the minimum achievable resolution should be taken to be 7.14×10^{-4} . The accuracy error was calculated to be approximately 3.98×10^{-3} . From here, it can be seen that the error is still within the claimed accuracy.

5.4 Discussion of Results

From the results, it can be seen that the variation in RI of the ambient environment can be solely encoded in the variation in intensity of the attenuation band in the output spectrum of the CNT-deposited LPFG. This modification in sensing scheme would allow the CNT-deposited LPFG to encode a secondary parameter through variations in wavelength of the attenuation band (see section 8.1) The CNT-deposited LPFG has also shown good repeatability tests with no conclusive degradation in sensing performance.

The sensitivity of the CNT-deposited LPFG can be further improved as well. The attenuation band that was chosen was at a wavelength position far from the telecommunications wavelength which would lead to problems related to power losses as discussed in section 3.1.3. One of the ways to overcome this issue is to use LPFGs with a shorter grating period Λ (see equation (2.2)). Gratings with shorter periods would have their higher order resonance bands, and thus attenuation bands, blue shifted to lower wavelengths to within the detectable region [29, 33]. These higher order resonance bands have been shown to exhibit greater sensitivity [29, 249] and their blue shifting to wavelengths in the detectable range of the OSA (or power meter) would enable their utility for better sensitivity performance of the sensing probe.

It should be mentioned that the sensitivity of a bare LPFG with a similar grating period has been previously shown to be approximately 0.158 nm/RIU for the RI range of 1-1.36 [33] and approximately 1300 nm/RIU for the RI range of 1.40-1.45 [27]. These values would indicate that the maximum achievable resolution for a bare LPFG would be approximately 6.33×10^{-2} and 7.69×10^{-5} [27] for the respective RI ranges. From here, it can be seen that the performance of the CNT-deposited LPFG is at least one

order of magnitude lower than that of the bare LPFG for the RI range of 1.40-1.45 but two orders of magnitude higher than that of the bare LPFG for the RI range of 1.33-1.38. The deviation in sensitivity for the RI range of 1.33-1.38 was calculated to be approximately 6.9 % which shows a good repeatability. Despite the lower performance at the higher RI ranges, it is believed that the sensitivity of the CNT-deposited LPFG can be increased by optimizing certain properties like the period of the grating. This coupled with the potential for simultaneous dual parameter sensing (which is not feasible in the bare LPFG) as well as the potential for large RI range measurement capabilities (as highlighted in section 4.2), would not decrease the viability of the CNT-deposited LPFG for sensing applications.

5.5 Concluding Remarks

The response of the CNT-deposited LPFG to variations in RI of the ambient environment has been presented in this chapter. The sensitivity and repeatability have also been discussed. Through the modification of the sensing scheme, the sensing capabilities of the CNT-deposited LPFG have been further enhanced in terms of their potential to measure a larger range of RIs and in dual parameter sensing. The fabrication complexity and costs of a LPFG however, still places it at a comparative disadvantage. As such, other sensing probes with considerably lower fabrication complexity can be explored and these will be discussed in the subsequent chapters.

Chapter 6 - Carbon Nanomaterial-Deposited PCF-MZI

The PCF-MZI and its response to variations in RI of the ambient environment have been discussed in section 3.3. Apart from exhibiting good sensitivity to variations in RI of the ambient environment, the PCF-MZI also possesses the inherent advantage of having a lower fabrication complexity as compared to that of the LPFG.

CNTs and MG were deposited onto the PCF-MZI and the sensing mechanism and fabrication of the carbon nanomaterial-deposited PCF-MZIs would first be described in this chapter. From there, the sensing behavior, repeatability and stability of the CNT-deposited and MG-deposited PCF-MZIs to variations in RI of the ambient environment would be presented and discussed in detail.

6.1 Sensing Mechanism of Carbon Nanomaterial-deposited PCF-MZIs

A schematic of the light propagation mechanism of a carbon nanomaterial-deposited PCF-MZI is shown in Fig. 6-1. As previously explained in section 2.4.5.1, light propagating as the core mode (solid arrows) in the SMF would excite the higher order cladding modes (dashed arrows) as it enters the first collapsed region. These excited modes would propagate in the cladding region of the PCF and recombine and interfere with the core mode at the second collapsed regions. As they propagate through the PCF segment, the evanescent waves (dotted arrows) of the excited higher order cladding modes would interact with the ambient environment. With the incorporation of a carbon nanomaterial overlay, these evanescent waves would experience a reflectance. Based on equation (4.21), the intensity of the evanescent waves and thus

Fig. 6-1 Schematic of the light propagation mechanism of a carbon nanomaterial-deposited PCF-MZI.

that of the excited higher order cladding modes would vary as the RI of the ambient environment varied. From equation (2.7), a variation in the intensity of the cladding modes would result in a varying of the intensity of the various spectral features generated in the output spectra of the carbon nanomaterial-deposited PCF-MZIs.

6.2 Fabrication of the Carbon Nanomaterial-Deposited PCF-MZIs

Two PCF-MZIs were fabricated using the procedure which has been described in section 3.3.1. The lengths of both PCF segments were approximately 30 mm. Both were then connected in the transmissive configuration to an ASE and an OSA and spray coated, one with the CNT solution and the other with the MG solution.

The output spectrum of both PCF-MZIs before and after the deposition of the CNT and MG overlays, respectively are shown in Fig. 6-2. For the CNT-deposited PCF-MZI, the RBW was calculated to be approximately 4.2 nm and 11.5 nm before and after the deposition, respectively. For the MG-deposited PCF-MZI, the RBW was calculated to be approximately 4.6 nm and 9.8 nm, respectively. For both carbon nanomaterial-

(a)

(b)

Fig. 6-2 Output spectrum of the PCF-MZIs before (dotted line) and after (solid line) deposition of (a) a CNT overlay and (b) a multilayer graphene (MG) overlay.

deposited PCF-MZIs, it can be seen that there is a slight variation in wavelength and a decrease in magnitude of some of the interference peaks and dips of the output spectrum after the spray coating of the carbon nanomaterials. This can be seen from equation (2.7), where the carbon nanomaterial overlay can be treated as a medium with a specific RI surrounding the bare PCF-MZI. Hence the difference in RI between the fiber

cladding and carbon nanomaterial overlay would result in a variation in the effective indices of the excited higher order cladding modes, resulting in a shift in wavelength of the various spectral features in the output spectrum. This would imply that spectral features from the shorter wavelength would be red shifted to the longer wavelength region which can be seen for both carbon nanomaterial-deposited PCF-MZIs shown in Fig. 6-2. Both diagrams in the figure show a red shift of approximately one FSR of the spectral features.

As previously discussed in section 3.1.4, the interference dips with the highest extinction ratios and nearest the telecommunications wavelength of 1550 nm were selected. For the CNT-deposited PCF-MZI shown in Fig. 6-2(a), the selected interference dip was at 1580.20 nm and its magnitude was approximately 17 dB lower than that of the bare PCF-MZI with an interference dip at 1579.60 nm. For the MG-deposited PCF-MZI in Fig. 6-2(b), the selected interference dip at 1579.24 nm had a 9

Fig. 6-3 Microscopy images of a small region of the PCF-MZI (a) before and (b) after the deposition of a CNT overlay onto the sensing probe.

Fig. 6-4 Microscopy images of a small region of the PCF-MZI (a) before and (b) after the deposition of a MG overlay onto the sensing probe.

dB attenuation in magnitude as compared to that of bare PCF-MZI with an interference dip at 1577.68 nm.

The optical microscopy images of a region of the PCF-MZI sensing probe before and after the deposition of CNTs and MG are shown in Fig. 6-3 and Fig. 6-4, respectively. As shown in Fig. 6-3(a) and Fig. 6-4(a), parts of the microstructure within the PCF segment of the PCF-MZI can be observed before the deposition of the respective carbon nanomaterials. These microstructures are no longer visible after the deposition of the carbon nanomaterials onto the PCF segments as shown in Fig. 6-3(b) and Fig. 6-4(b). Similar to that of the CNT-deposited LPFG, the carbon nanomaterials were also uniformly deposited over the entire surface of both PCF-MZIs. The thickness of the deposited CNT overlay was estimated to be in the order of a few nanometers. For the deposited MG overlay, a surface profiler (Dektak³) was used to measure the difference in height between silicon wafer and the deposited MG and the thickness of the deposited MG overlay was calculated to be approximately 10 μm . The difference in

surface morphology of both carbon nanomaterial-deposited PCF-MZIs lie in the fact that MG consists of several layers of graphene sheets. Compared to the rolled-up one-dimensional structure of CNTs, these layers are much larger in size. As the deposition process is random, the carbon nanomaterials would overlap when deposited on the fiber surface. The larger size of the MG layer would result in a greater visual unevenness of the deposited overlay as compared to that of CNTs.

6.3 Sensing Behavior of CNT-deposited PCF-MZI

The CNT-deposited PCF-MZI was connected to an ASE source and an OSA in the transmissive configuration for the RI characterization. In the subsequent subsections, the results of the sensing behavior of the CNT-deposited PCF-MZI would be presented and a short discussion of the results would be provided.

6.3.1 RI Characterization of CNT-deposited PCF-MZI

The results of the RI characterization for the CNT-deposited PCF-MZI are shown in Fig. 6-5. The interference peak at a wavelength of 1580.20 nm can be seen to demonstrate a resolvable decrease in the magnitude of its intensity as the RI of the ambient environment increased. The ripple like behavior occurring throughout the spectrum was due to the ASE source and OSA and can be resolved using a more stable light source and a higher resolution setting on the OSA. The higher resolution settings however, would decrease the SNR and give rise to the noise floor limitations described in section 3.5.2.

The decrease in magnitude of the intensity of the interference dip is to be expected. From section 4.2, it has already been shown based on equation (4.21) that the amount of reflectance would decrease as the RI of the ambient environment increases. Hence more of the evanescent wave is coupled out and the intensity of the excited higher

Fig. 6-5 Behavior of the interference dip at a wavelength of 1580.20 nm to variations in RI of the ambient environment.

order cladding modes would be expected to decrease as the RI of the ambient environment increased. Based on equation (2.7), a decrease in intensity of the excited higher order cladding modes would lead to a decrease in their interference with the core mode resulting in a decrease in the magnitude of the intensity of the attenuation bands in the output spectrum.

6.3.2 Wavelength Behavior of CNT-deposited PCF-MZI

The variation in wavelength of the CNT-deposited PCF-MZI to variations in RI of the ambient environment is shown in Fig. 6-6. As can be observed, the wavelength varied minimally with a slight fluctuation within the range of 1581.76-1583.08 nm. These fluctuations can also be attributed to equipment error. A point to note is that the deviation in wavelength of the CNT-deposited PCF-MZI is lesser than that of the CNT-deposited LPFG which implies that despite the errors from source and detector, the CNT-deposited PCF is a much more stable than the CNT-deposited LPFG.

Fig. 6-6 Variation in wavelength of the interference dip at a wavelength of 1580.20 nm to variations in RI of the ambient environment.

6.3.3 Repeatability, Sensitivity and Reproducibility of CNT-deposited PCF-MZI

A second RI characterization trial was carried out for the CNT-deposited PCF-MZI and the variation in intensity of the interference dip at a wavelength of 1580.20 nm was plotted relative to the variation in RI of the ambient environment as shown in Fig. 6-7. A fitting line (solid line) was plotted through the data values (diamonds for the initial trial, hexagons for the second trial) for both trials. As can be seen, the intensity of the interference dip varied nonlinearly with the RI of the ambient environment. The maximum error due to repeatability for both trials was calculated to be approximately 0.2 dB which is a better margin of error as compared to that of the CNT-deposited LPFG. The achieved sensitivity was calculated to be approximately 19.4 dB/RIU and 24.2 dB/RIU for the RI ranges of 1.33-1.38 and 1.38-1.42, respectively. The error corrected minimum achievable resolution for the RI range of 1.33-1.38 was calculated to be

Fig. 6-7 Variation in intensity of the interference dips of two PCF-MZIs to variations in RI of the ambient environment for two CNT-deposited PCF-MZIs. Data points of the first CNT-deposited PCF-MZI are indicated by the diamonds and hexagons and fitted with a solid line indicate while that of the second CNT-deposited PCF-MZI are indicated by the hollow circles and fitted with a dotted line.

approximately 1.08×10^{-3} RIU. In contrast with the theoretical limit of 5.15×10^{-3} RIU, this low resolution is possibly due to the high error from the fluctuations in the measurement data collected. This can be overcome through the collection of more measurement data which would better approximate the performance of the sensing probe.

To test the reproducibility of the sensing probe, another CNT-deposited PCF-MZI was fabricated. The RI characterization trial was carried out and the intensity of the selected interference dip as the RI of the ambient environment varied is plotted in Fig. 6-7 (hollow circles) as well. Data values for this CNT-deposited PCF-MZI were similarly fitted along a single line and a similar nonlinear trend was observed. The sensitivity was calculated to be approximately 13.9 dB/RIU and 26.0 dB/RIU for the RI ranges of 1.33-1.38 and 1.38-1.42, respectively. The similarity in sensitivities for the

respective RI ranges as well as the observable nonlinear behavior displayed by both CNT-deposited PCF-MZIs indicates the good reproducibility of the sensor. The deviation in sensitivity for the first PCF was measured to be approximately 11% for the two trials, which shows that the sensor has relatively good repeatability. Hence the CNT-deposited PCF-MZI has been shown to demonstrate good repeatability and reproducibility.

6.4 Sensing Behavior of MG-deposited PCF-MZI

The MG-deposited PCF-MZI was similarly connected to an ASE and OSA in the transmissive configuration. In the subsequent sub-sections, the results of the sensing behavior of the CNT-deposited PCF-MZI would be presented and a short discussion of the results would be given.

6.4.1 RI Characterization of MG-deposited PCF-MZI

Fig. 6-8 shows the results of the RI characterization trial. The magnitude of the intensity of the interference dip at a wavelength of 1579.24 nm exhibited a clear and distinct decrease as the RI of the ambient environment increased. The ripple like effect is similarly due to the equipment used as discussed in section 6.3.1. The reason for the decrease in magnitude of the intensity is also due to the decrease in reflectance of the evanescent wave and thus decrease in intensity of the excited higher order cladding modes, leading to the diminishing in the amount of interference between the excited higher order cladding modes and the core mode.

It can be observed that the variation in intensity of the MG-deposited PCF-MZI is lower than that of the CNT-deposited PCF-MZI. The exact reason is still under investigation but the observed difference is most likely due to the properties of the two carbon nanomaterials. The smaller CNTs would have formed a more porous layer over

Fig. 6-8 Behavior of the interference dip at a wavelength of 1577.68 nm to variations in RI of the ambient environment.

the PCF-MZI allowing for better interaction of the evanescent waves with that of the ambient environment. Whereas as seen in Fig. 4-7(b), the MG overlay is made up of overlapping layers of graphene flakes which would have confined most of the evanescent waves within the overlay which would decrease the amount of interaction of the evanescent waves with the ambient environment.

6.4.2 Wavelength Behavior of MG-deposited PCF-MZI

The variation in wavelength of the interference dip with respect to the variation in RI of the ambient environment is shown in Fig. 6-9. From the figure, it can be observed that the wavelength exhibited no variations and remained at a constant value of 1579.72 nm within the RI range of 1.33 to 1.44. This is considerably more stable than that of the CNT-deposited LPFG and the CNT-deposited PCF. The same evanescent wave confinement properties of the MG overlay from the previous sub-section can be invoked to explain the improved stability of the MG-deposited PCF-MZI over that of the CNT-deposited PCF-MZI.

Fig. 6-9 Variation in wavelength of the interference dip at 1580.20 nm to variations in RI of the ambient environment.

6.4.3 Repeatability, Reliability and Sensitivity of MG-deposited PCF-MZI

Fig. 6-10 shows the variation intensity (diamonds) of the interference dip to a variation in RI of the MG-deposited PCF-MZI. The results of a second trial (triangles) are also shown in the figure. It can be seen that both trials show a consistent nonlinear variation in the intensity of the interference dip as the RI of the ambient environment increased. A single fitting line was plotted through the data values from both trials and from there, the maximum error due to repeatability was calculated to be approximately 0.1 dB which is better than that of the CNT-deposited LPFG and CNT-deposited PCF-MZI. The achieved sensitivity was calculated to be approximately 9.4 dB/RIU and 17.5 dB/RIU for the RI ranges of 1.33-1.38 and 1.38-1.43, respectively. From here, the error corrected minimum achievable resolution for the RI range of 1.33-1.38 was calculated to be approximately 1.07×10^{-3} RIU. The poor sensitivity and thus, low resolution of the sensor is likely due to the thickness of the deposited MG overlay. A thick MG overlay

Fig. 6-10 Variation in intensity of the attenuation band at 1463.2 nm to variations in RI of the ambient environment for two separate trials.

would lead to a higher absorption of the evanescent wave resulting in a decrease in the amount of light interacting with the ambient environment. The sensing performance of the MG-deposited PCF-MZI would be expected to improve with the optimization of the thickness of the MG overlay.

The reliability of the MG-deposited PCF-MZI can also be inferred from these results. The repeatability of the RI sensing behavior over repeated trials, and over an extended period of time, indicates that there was no degradation of the overlay. This is due to the fact that the adherence of the MG onto the fiber surface and between individual MG flakes is due to van der Waal's forces which have to be overcome for any overlay degradation to occur.

6.5 Discussion of Results

Both carbon nanomaterial-deposited PCF-MZIs showed a considerable amount of variation in intensity the intensity of the interference dips as the RI of the ambient

environment varied. From Fig. 6-5 and Fig. 6-8, it can be observed that the interference dips of both sensing probes were able to demonstrate clear and distinguishable variations in intensity as the RI of the ambient environment varied. The encoding of the variations in RI of the ambient environment can be concluded to be solely in that of the magnitude of the intensity of the attenuation bands. Due to this modification in sensing scheme, both sensing probes were able to continuously measure variations in RI of their ambient environment with no need for additional reference measurement points.

As shown in sections 6.3.2 and 6.4.2, there was a fluctuation in wavelength of approximately 1.3 nm for the CNT-deposited PCF-MZI while no detectable fluctuation in wavelength was detected for the MG-deposited PCF-MZI. This enhanced stability can also be contrasted with that of the uncoated PCF-MZIs discussed in section 3.3.2. It can be seen that for the uncoated PCF-MZIs, there is random fluctuation in intensity as the RI of the ambient environment varies. The superior stability of the MG-deposited PCF-MZI over that of the CNT-deposited PCF-MZI however, comes at the cost of a lower sensitivity. Despite this trade off, based on the calculated resolution, both carbon nanomaterial-deposited PCF-MZIs were still able to demonstrate an acceptable performance in terms of their resolution. The repeatability tests have also shown that there was no overall degradation in sensing performance over time for both sensors which satisfies one of the criteria for sensors in general.

There is however, one key disadvantage of such a sensor. PCF-MZIs have been shown to exhibit minimal response to variations in temperature of the ambient environment [117]. This property is particularly detrimental to RI sensors as the RI of a particular medium is cross-coupled with the temperature of its ambient environment. With a near-immunity to variations in temperature of the ambient environment, a PCF-MZI would not be able to de-couple the two parameters for absolute RI sensing. Based

on equation (4.21), the carbon nanomaterial-deposited PCF-MZIs would not exhibit any spectral variations in output as the temperature of the ambient environment varied and thus have the same inherent temperature immunity as the bare PCF-MZIs. Hence a dual parameter sensor with the ability to de-couple the cross-relation between temperature and RI would not be achievable for these sensing probes. To achieve such a sensing probe, a second temperature sensitive sensing probe like a FBG or LPFG has to be cascaded with the individual carbon nanomaterial-deposited PCF-MZIs which would increase the fabrication complexity and costs as well. Nevertheless, PCF-MZIs have been shown to respond to variations in strain of the ambient environment [117], hence dual parameter sensing can still be achieved in fields which would require the measurement of variations in RI and strain of the ambient environment.

6.6 Concluding Remarks

This chapter has discussed the fabrication and sensing behavior of PCF-MZIs deposited with CNTs and MG. Like the CNT-deposited LPFG, the carbon nanomaterial-deposited PCF-MZIs also experienced a modification in sensing mechanism. Despite the fact that PCF-MZI offers the advantage of a relatively low fabrication complexity as compared to the LPFG, the complex fabrication process and cost of fabrication of a PCF would not make it an ideal sensing probe for large-scale applications.

Chapter 7 - CNT-Deposited MMF-MMI

The MMF-MMI described in section 3.4 has been shown to exhibit good sensitivity to variations in RI of the ambient environment. This sensing probe however, is limited in RI measurement range due to the constraints set by the Fresnel relation (equation (2.9)).

This chapter will present the results of the last carbon nanomaterial-deposited sensing probe. CNTs would be deposited onto the end-face of a MMF-MMI. The fabrication process of the CNT-deposited MMF-MMI and the enhancement of its sensing capabilities compared to that of the bare MMF-MMI to variations in RI of the ambient environment would be presented.

7.1 Sensing mechanism of the CNT-deposited MMF-MMI

Fig. 7.1 shows a schematic of the light propagation mechanism of the CNT-deposited MMF-MMI. As previously discussed in section 2.4.5.2, light propagating as the fundamental mode (solid arrows) would excite the higher order modes (dashed arrows) as it enters the MMF segment. The excited higher order modes would co-propagate with the fundamental mode along the MMF segment. Upon interaction with

Fig. 7-1 Schematic of the light propagation mechanism of a CNT-deposited MMF-MMI.

the fiber-CNT interface, the fundamental mode and the excited higher order modes would both experience a reflectance given by equation (4.21). The back-reflected modes can then be monitored by a detector. RI variations in the ambient environment would modulate the intensity of the reflected modes resulting in a variation in intensity of the spectral features in the output spectrum.

7.2 Fabrication of CNT-deposited MMF-MMI

The same procedure as that described in section 3.4.1 was used to fabricate a MMF-MMI with a MMF segment of 21 mm. The optical deposition method described in section 4.5.2 was then carried out to deposit a CNT overlay onto the end-face of the MMF-MMI. Fig. 7.2 shows the output spectrum of the MMF-MMI before and after deposition of CNTs. The RBWs before and after the deposition of CNTs was calculated to be approximately 17.6 nm and 19.1 nm, respectively. As can be seen from the figure,

Fig. 7-2 Output spectrum of the MMF-MMI before (dotted line) and after (solid line) deposition of a CNT overlay.

there was an increase in intensity with almost no variation in wavelength of the output

spectrum after the optical deposition of CNTs. The increase in intensity is due to the high RI of the CNT overlay which from equation (4.21), can be seen to increase the amount of reflectance. The interference peak at 1558.36 nm was chosen due to its high extinction ratio and it can be seen that there was a red shift to 1558.96 nm and an approximately 6 dB increase in intensity after the deposition of the CNT overlay. The variation in wavelength was small and can be attributed to errors from the detector and also to fluctuations in the temperature of the ambient environment.

The microscopy image of the CNT-deposited MMF-MMI before and after the optical deposition process is shown in Fig. 7-3(a) and Fig. 7-3(b), respectively. It can be seen in Fig. 7-3(b) that CNTs were deposited on the end-face of the MMF-MMI and from here, the thickness of the deposited CNT overlay was estimated to be approximately 20 μm . The adhesion of CNTs onto the end face of the MMF is due to van der Waal's forces which have to be overcome before any degradation in CNT would

Fig. 7-3 Microscopy images of the MMF-MMI (a) before and (b) after deposition of CNTs.

occur. Unless exposed to some form of mechanical or thermal shock, it would be unlikely that the CNTs would be detached from the MMF fiber. The reliability of the fabricated CNT-deposited MMI can be later seen in section 7.3.3 through the repeatability of the RI sensing behavior over repeated trials and over an extended period of time.

7.3 Sensing behavior of CNT-deposited MMF-MMI

The CNT-deposited MMF-MMI was connected to an ASE and an OSA via a circulator in the reflective configuration. The subsequent sub-sections would discuss the RI characterization procedures, wavelength behavior, sensitivity, repeatability and stability of the fabricated sensing probe.

7.3.1 RI characterization of the CNT-deposited MMF-MMI

The response of the CNT-deposited MMF-MMI to variations in RI of the ambient environment is shown in Fig. 7-4. It can be seen that there was a clear and distinct decrease in the magnitude of the intensity of the interference peak at a wavelength of 1558.84 nm as the RI of the ambient environment increased. As discussed previously in section 4.2, this behavior is to be expected as the amount of reflectance would decrease as the RI of the ambient environment increased. A decrease in reflectance would decrease the intensity of the back-reflected modes resulting in a decrease in intensity of the interference peak.

Fig. 7-4 Behavior of the interference peak at a wavelength of 1558.84 nm to variations in RI of the ambient environment.

7.3.2 Wavelength Behavior of the CNT-deposited MMF-MMI

Fig. 7-5 shows the variation in wavelength of the interference peak at a wavelength of 1558.84 nm to a variation in RI of the ambient environment. From here it can be seen that there was little variation in the wavelength of the interference peak

Fig. 7-5 Variation in wavelength of the interference peak at a wavelength of 1558.84 nm as the RI of the ambient environment varied.

with slight fluctuations within the wavelength range of 1558.72-1558.84 nm. This is to be expected as the variation in RI of the ambient environment would lead to a variation in reflectance as shown in equation (4.21) with no variation in wavelength. The fluctuations can be attributed to measurement errors from the detector. The relatively high wavelength stability implies that variations in RI of the ambient environment would be encoded solely in the intensity of the interference peak.

7.3.3 Sensitivity, Repeatability and Stability of the CNT-deposited MMF-MMI

A second RI characterization trial was carried out and the change in intensity of the interference peak at 1558.84 nm for both trials (diamonds for the first trial and circles for the second trial) is shown in Fig. 7-6. It can be seen that there was a nonlinear variation in intensity of the interference peak as the RI of the ambient environment increased. A line was fitted through the data values and the maximum error due to repeatability was calculated to be approximately 0.8 dB. From here, the sensitivity of

Fig. 7-6 Variation in intensity over two trials of the interference peak at 1558.84 nm as the RI of the ambient environment varied.

the CNT-deposited MMF-MMI was calculated to be approximately 35.8 dB/RIU and 98.8 dB/RIU for the RI ranges of 1.33-1.40 and 1.40-1.46, respectively. The minimum achievable resolution for the RI range of 1.33-1.40 can then be calculated to be approximately 2.79×10^{-4} .

To characterize the stability of the CNT-deposited MMF-MMI, the sensing probe was completely immersed in a test solution with a RI of 1.3334 and the variation in intensity of the interference peak was observed over 60 mins. The intensity of the interference peak at 1558.84 nm was recorded at 5 min intervals as shown in Fig. 7-7. It can be seen that there was only a 0.08 dB fluctuation in intensity over the 60 minutes which is negligible and can be attributed to fluctuations from the source and measurement errors from the detector. From here, it can be concluded that the performance of the CNT-deposited MMF-MMI would be stable and not fluctuate over time.

Fig. 7-7 Variation in intensity of the interference peak at 1558.84 nm over 60 mins for the CNT-deposited MMF-MMI immersed in a test solution with a RI of 1.3334.

7.4 Discussion of results

It can be concluded from the results that the noise floor limit which affected the bare MMF-MMI as discussed in section 3.5.2 has been overcome by the CNT-deposited MMF-MMI. From Fig. 3-10(a) and Fig. 3-11(a), it can be seen that at a RI of 1.43, the interference peaks start to experience noise from the detector. The noise is however absent in the output spectrum of the CNT-deposited MMF-MMI for RIs as high as 1.44 (Fig. 7-4). This can be explained by comparing equation (2.9) for the bare MMF-MMI and equation (4.21) for the CNT-deposited MMF-MMI. As described briefly in section 2.4.5.2, it can be seen from equation (2.9) that the amount of reflectance for the bare MMF-MMI would decrease exponentially as the RI of the ambient environment increased. This decrease would occur continuously until the RI of the ambient environment matches that of the MMF fiber core which is approximately 1.4446. At this point, the reflectance would be zero, meaning that there would be no back-reflected signal. Further increasing the RI of the ambient environment further would lead to an increase in reflectance again. At this point, the bare MMF-MMI would experience a limitation similar to that of the FSR limitation where reference measurements of the intensity have to be taken to measure any variations in RI of the ambient environment above that of the fiber core. From equation (4.21), it can be concluded that the CNT-deposited MMF-MMI would also experience the same limitation. However, this would only occur when the RI of the ambient environment matches that of the RI of CNT, which is considerably higher than that of the fiber core. Apart from that, the high RI of CNTs also resulted in a higher reflectance of the back-reflected light, increasing the SNR of the output spectrum as shown in Fig. 7-2. Hence the CNT-deposited MMF-MMI would have the added advantages of an improved RI measurement range and SNR as compared to the bare MMF-MMI.

7.5 Concluding Remarks

The MMF-MMI has the advantage of having the least fabrication complexity, highest cost effectiveness and application versatility, making it an ideal candidate for environmental sensing applications. This chapter has presented the fabrication and sensing performance of the CNT-deposited MMF-MMI. It can be seen that the deposited CNT overlay enhanced the RI sensing capabilities of the bare MMF-MMI, improving its measurement range and stability. A discussion of all the sensing probes and a comparison of their sensing performance was provided and it can be seen that the CNT-deposited MMF-MMI was able to demonstrate the best sensing performance in terms of the achievable resolution, RI measurement range, stability and repeatability.

Chapter 8 – Discussion, Conclusion and Future Work

The first seven chapters of this thesis have discussed the need for optical fiber environmental sensors and how these optical fiber sensing probes would behave to perturbations in the ambient environment. This chapter would conclude this thesis by first presenting a discussion and comparison of all the sensing probes discussed in the thesis. It would then go on to give a summary of what has been done and the future directions of this study.

8.1 Discussion and Comparison of all Sensing Probes

The detection mechanisms of the various sensing probes discussed in this thesis rely on the interaction of light propagating through the optical fiber with the ambient environment. With the tapered fiber, LPFG and PCF-MZI, this involves the excitation or coupling of a portion of the light near the fiber surface. The behavior of these excited higher order modes of light would depend on the RI of the ambient environment, resulting in a variation in wavelength of the output. For the MMF-MMI, the variation in output depends on the intensity of the reflected light at the end face of the sensing probe.

With the deposition of a carbon nanomaterial overlay, the sensing mechanism would be altered due to the high RI of the overlay. For the CNT-deposited LPFG, this would involve the coupling of the energy in the core mode to radiation modes as described in section 2.2.1. Variations in RI of the ambient environment would affect the magnitude of this coupling, thus varying the intensity of the attenuation bands. For the carbon nanomaterial-deposited PCF-MZIs, deposition of a carbon nanomaterial overlay would lead to an increase in effective indices of the excited cladding modes. The high effective indices of the cladding modes would lead to little variations in phase of the cladding modes as the RI of the ambient environment varied. The sensing mechanism would then

depend on the reflection of the evanescent waves due to the variation in effective index of the carbon nanomaterial overlay as the RI of the ambient environment varied. For the CNT-deposited MMF-MMI, the reflected intensity of the light at the end face of the sensing probe would depend on the variation in reflectance of the CNT as the RI of the ambient environment varied. From the above discussion, it can be seen that the deposition of a carbon nanomaterial has led to an overall increase in performance of the sensing probes.

A possible explanation for this improvement in sensing performance due to the deposition of a carbon nanomaterial overlay can be inferred from Fig. 4-5. It can be seen that the reflectance of the carbon nanomaterial overlay decreases exponentially as the RI of the ambient environment increases. Hence, at lower RI ranges, there is little variation in reflectance as the RI of the ambient environment varies. However, at higher RI ranges, a larger variation in reflectance of the carbon nanomaterial overlay would be observed. Hence the performance of these carbon nanomaterial-deposited sensing probes would be expected to be better than their bare counterparts. Contrasting this performance with that of references [27], [33], [106] and [119], it can be seen that the carbon nanomaterial deposited sensing probes performed better in various aspects. The resolutions for that of the bare LPFG, PCF-MZI and MMF-MMI for a RI range of 1.3-1.38 were reported to be 6.33×10^{-2} , 1.0×10^{-2} and 3.8×10^{-4} . Comparing these values to that in Table 2, it can be seen that the carbon nanomaterial-deposited counterparts to these bare sensing probes performed better. For the upper RI range of 1.38-1.44 however, the carbon nanomaterial-deposited sensing probes performed slightly poorer than their reported bare counterparts. As previously discussed, the poorer performance is likely due to measurement errors and a better approximation of the behavior can be obtained through obtaining more measurement data which would be the subject of future

work (see section 8.3). In all cases however, the continuous measurable RI range of the carbon nanomaterial-deposited sensing probes were higher than that of their bare counterparts both presented in this thesis and reported in the literature.

As discussed in sections 5.4 and 6.5, the modification of the sensing scheme through deposition of a carbon nanomaterial overlay onto LPFG and PCF-MZI sensing probes has opened up the possibility for dual parameter sensing. As also discussed in chapter 2, perturbations like strain, RI and temperature from the ambient environment would result in a shift in wavelength of the various spectral features in the output of the LPFG and PCF-MZI. The deposition of a carbon nanomaterial overlay onto these sensing probes has enabled the decoupling of the RI perturbations, allowing the encoding of a second perturbation in the variation in wavelength of the spectral features. As seen from equation (4.21), the amount of reflectance only varies with a variation in RI of the ambient environment. Hence variations in temperature or strain in the ambient environment can be encoded in the wavelength, leading to the possibility for dual perturbation sensing. Dual parameter sensing is also possible for the CNT-deposited MMF-MMI. It has been shown that an increase in temperature of the ambient environment would lead to an increase in the length of the MMF segment of a bare MMF-MMI [104, 106]. This would result in a red shift in the wavelength of the interference peaks and dips which can be used to characterize the variations in temperature of the ambient environment. The response of the CNT-deposited MMF-MMI to variations in temperature of the ambient environment would be expected to be similar to that of the bare MMF-MMIs. The ability to encode variations in RI and temperature through variations in intensity and wavelength, respectively, of its output would enable the CNT-deposited MMF-MMI to de-couple the temperature-RI cross-relation and function as a dual parameter sensor as well.

It can also be seen from the results in chapters 5, 6 and 7 that the carbon nanomaterial-deposited optical fiber sensors encode variations in RI of the ambient environment through variations in magnitude of the intensity of their spectral features. This has resulted in an improvement in sensing capabilities, allowing these sensors to overcome the limitations discussed in section 3.5. The FSR limitation discussed in section 3.5.1 is no longer a limitation for the carbon nanomaterial-deposited PCF-MZIs. Comparing the results from section 3.3 and sections 6.3 and 6.4, it can be seen that higher RIs can still be continuously measured by the carbon nanomaterial deposited PCF-MZIs as compared to the bare PCF-MZIs. Due to the FSR limitation, the highest RI of approximately 1.42 could be measured by the bare PCF-MZI before reference measurements have to be taken to extend the measurement range of the bare PCF-MZI. Whereas for the carbon nanomaterial-deposited PCF-MZIs, RIs as high as 1.43 could still be distinguished without the need for reference measurements.

An interesting feature of these carbon nanomaterial-deposited sensing probes is that the modification of the sensing scheme would allow for an increase in compactibility of the sensor as a whole. The entire sensor configuration can be modified by replacing the OSA with a power meter and cascading a band pass filter between the sensing probe and the power meter. This would increase the cost effectiveness and portability of the sensors. For the MG-deposited PCF-MZI which has exhibited no change in wavelength to variations in RI of the ambient environment, the cost effectiveness and compactibility can be further increased by omitting the band pass filter.

Tables 2 and 3 compare the RI range, sensitivities and the achievable resolution of all the optical fiber sensors that were fabricated. Table 2 shows the performance of these sensors for a lower RI range while that shown in Table 3 is for a higher RI range.

It can be seen from Table 2 that for the lower RI range the tapered fiber sensor displayed the highest sensitivity and resolution as compared to the other optical fiber sensors. The high sensitivity and resolution is to be expected for the tapered fiber due to the exposure of a large portion of the light propagating through the tapered region to the ambient environment. However, this is at the cost of a limited continuous measurement range due to the FSR as discussed in section 3.5.1. The wavelength of the interference dip corresponding to ambient environmental RIs above 1.3345 could not be determined without the use of reference wavelengths as the change in wavelength had exceeded the FSR. Apart from this, the decrease in diameter of the tapered fiber also decreases the mechanical strength of the tapered fiber which makes it prone to failures and erroneous measurements.

From tables 2 and 3, it can be seen that the carbon nanomaterial-deposited LPFG and PCF-MZI demonstrated better resolutions in the upper RI ranges. From table 2, both the CNT-deposited and MG-deposited PCF-MZIs demonstrated resolutions lower than that of the bare PCF-MZI. From table 3, it can be seen that there is a reversal in

Fiber Sensor	RI Range	Sensitivity	Minimum Resolution
Tapered Fiber	1.3332-1.3345	3373.4 nm/RIU	2.00×10^{-5}
PCF-MZI	1.33-1.38	144.1 nm/RIU	1.97×10^{-4}
MMF-MMI	1.33-1.40	93.19 dB/RIU	6.88×10^{-4}
CNT-deposited LPFG	1.33-1.38	30.7 dB/RIU	7.14×10^{-4}
CNT-deposited PCF-MZI	1.33-1.38	19.4 dB/RIU	1.08×10^{-3}
MG-deposited PCF-MZI	1.33-1.38	9.4 dB/RIU	1.07×10^{-3}
CNT-deposited MMF-MMI	1.33-1.40	35.8 dB/RIU	2.79×10^{-4}

Table 2 Table comparing the sensitivities and the minimum achievable resolution of all optical fiber RI sensors studied in this thesis.

Fiber Sensor	RI Range	Sensitivity	Maximum Resolution
PCF-MZI	1.38-1.42	433.0 nm/RIU	7.08×10^{-4}
MMF-MMI	1.40-1.46	216.3 dB/RIU	6.13×10^{-4}
CNT-deposited LPFG	1.38-1.42	46.8 dB/RIU	3.36×10^{-4}
CNT-deposited PCF-MZI	1.38-1.42	24.2 dB/RIU	4.88×10^{-4}
MG-deposited PCF-MZI	1.38-1.43	17.5 dB/RIU	5.72×10^{-4}
CNT-deposited MMF-MMI	1.40-1.46	98.8 dB/RIU	1.02×10^{-3}

Table 3 Table comparing the sensitivities and the maximum achievable resolution of all optical fiber RI sensors studied in this thesis.

performance with both the CNT-deposited and MG-deposited PCF-MZIs demonstrating a better sensing resolution than that of the bare PCF-MZI. The performance of the MG-deposited PCF-MZI was slightly lower than that of the PCF-MZI at higher RI ranges. It does however have the advantage of having the highest stability amongst all the sensing probes studied.

For the case of the MMF-MMI, it can be seen that the CNT-deposited MMF-MMI performed better than the bare MMF-MMI at the lower RI range but not at the higher RI range. This is likely due to the fluctuations in measurement data for the CNT-deposited MMF-MMI which resulted in a poor error corrected resolution. This can be overcome through collecting a wider body of measurement data to better approximate the behavior and resolution of the CNT-deposited MMF-MMI. With sufficient data, it is believed that the resolution of the CNT-deposited MMF-MMI would theoretically be able to achieve a resolution as low as 1.0×10^{-4} for the RI range of 1.40-1.46. The bare MMF-MMI also exhibited the poorest wavelength stability and also experienced a limitation in its RI measurement range. This would affect its application as a dual

parameter sensor as the encoding of temperature variations of the ambient environment in the wavelength of its output spectrum would be affected by the fluctuations. Its CNT-deposited counterpart however, was able to overcome these stability and RI measurement range issues with only a fractional trade off in resolution in the upper RI ranges. Hence, it can be concluded that as compared to its bare MMF-MMI counterpart, the CNT-deposited MMF-MMI would overall demonstrate a better performance as a sensor. The CNT-deposited MMF-MMI also possesses an inherent advantage as compared to the other carbon nanomaterial-deposited sensing probes. As discussed in section 2.1, due to its usage in the reflective configuration, it would be possible to use the CNT-deposited MMF-MMI as a probe to access certain small and inaccessible environments. Upon functionalization of the CNTs for detection of certain analytes (see section 8.3), this application can be further extended to chemical or gas leak detection in these remote environments thus increasing the versatility of the CNT-deposited MMF-MMI.

From the above discussion and the data presented, it can be seen that the sensing capabilities of various sensing probes would be enhanced. In general, the deposition of the various carbon nanomaterials has improved the resolution of the various sensing probes. Apart from that, they have also demonstrated observable variations in intensity with little fluctuations in wavelength as the RI of the ambient environment varied. This is in contrast to that of the bare sensing probes which demonstrated an observable variation in wavelength but random fluctuations in intensity as the RI of the ambient environment varied. From here, it can be seen that the carbon nanomaterial-deposited sensing probes would have the added advantage of performing as dual parameter sensors as the wavelength can be used to quantify a secondary parameter like temperature or strain. Apart from that, the added potential for functionalization of the carbon

nanomaterial overlay would also allow for the extension of the sensing capabilities of these sensing probes.

8.2 Conclusion

Due to their physical and light confinement properties, optical fibers have been shown to be applicable as environmental sensors. Various optical fiber sensing probes have been demonstrated over the years which have been shown to be able to achieve good refractive index (RI) sensing performance of their ambient environment. However, some of these sensing probes still face limitations from their sensing scheme or sensing mechanism. To overcome these limitations and thus enhance the sensing capabilities of some of these sensing probes, this thesis has proposed the deposition of a carbon nanomaterial overlay onto these sensing probes.

Two carbon nanomaterials, carbon nanotubes (CNTs) and multilayer graphene (MG), were studied as overlay materials to be deposited onto long period fiber gratings (LPFGs), photonic crystal fibers in a Mach-Zehnder Interferometer configuration (PCF-MZIs) and a multimode fiber (MMF) segment fusion spliced to a single mode fiber (SMF) to form a multimode interferometer (MMI). The carbon nanomaterials had to be specially dispersed into a solution before they could be cold transferred onto the various sensing probes. The spray coating method was used to deposit a CNT overlay onto a LPFG and a CNT and MG overlay onto the PCF-MZIs. The optical deposition method was used to deposit a CNT overlay onto the end-face of MMF-MMI. The deposited carbon nanomaterial overlays were able to adhere strongly to the fiber surfaces without the need for any additional physical or chemical treatment. Both methods were found to be reliable, cost effective and low in complexity which would partially satisfy the fabrication criteria for an optical fiber sensing probe.

The sensing mechanism of each of the carbon nanomaterial-deposited sensing probes were investigated. The behavior of the spectral features in the output of these sensing probes were monitored for variations in their intensity and wavelength. The stability, repeatability and reproducibility of these sensing probes were also investigated. It was found that these sensing probes were able to encode variations in RI of the ambient environment through variations in intensity of the spectral features in their output. Any variations in RI of the ambient environment could be continuously measured within the range of RIs studied without the need for any reference points. This allowed for the encoding of a secondary parameter through variations in wavelength of the spectral features, opening up potential for dual parameter sensing.

Of the four carbon nanomaterial-deposited sensing probes investigated, it was found that the CNT-deposited MMF-MMI achieved the best overall sensing performance. It exhibited the best sensitivity and achievable resolution to variations in RI of the ambient environment and was also able to characterize the largest RI range. Apart from that, the CNT-deposited MMF-MMI also had the advantage of having a relatively low fabrication complexity and material cost. As compared to the fiber-based sensors presented in this thesis, the CNT-deposited MMF-MMI was also able to detect a change in RIs for the widest range of RIs without any decrease in the quality of the output spectrum. This coupled with its theoretically achievable resolution of 1.0×10^{-4} and theoretically achievable detection range to RIs as high as that of the RI of CNT has made it comparable to that of the currently commercially available non-fiber based refractometers. Hence, the CNT-deposited MMI would be a viable sensing probe for industrial-based applications.

8.3 Recommendations for Future work

As the current study is only a pilot demonstration of the sensing capabilities of various optical fiber sensing probes deposited with a carbon nanomaterial overlay, more research can be done to look into improving the various aspects of these sensing probes.

Due to the encoding of the variations in RI of the ambient environment in the intensity of the various spectral features in the output spectrum, these carbon nanomaterial-deposited sensing probes would be susceptible to any intensity fluctuations in output from an unstable light source. Hence further improvements to the sensing configurations can be explored. Intensity fluctuations in the output of the light source can be accounted for by modifying the transmissive and reflective configurations shown in Fig. 2-2 to include an additional reference arm. The reference arm would generate a reference spectrum and the change in the spectral features in the sensing arm can be measured relative to the reference arm and recorded instead. This would allow for the compensation of any fluctuations in intensity, but with the disadvantage of increasing the complexity of the sensor as a whole.

The sensitivity of the various carbon nanomaterial-deposited sensing probes can also be further optimized. For the CNT-deposited LPFG, it has been previously shown that in order to achieve a higher sensitivity, there is an optimum overlay thickness for any material deposited on any LPFG with any grating period [249, 253]. This can also be extended to the other sensing probes. As also shown in equation (4.21), the thickness of the carbon nanomaterial overlay would affect the amount of reflectance. Hence the thickness of the deposited carbon nanomaterial overlay can be optimized to improve the performance of these carbon nanomaterial-deposited sensing probes. This can be done in several ways. For the sensing probes fabricated by the spray coating method, this can

be done by optimizing interdependent parameters like the spray duration, the pressure of the nitrogen gas, the aperture of the spray gun and the distance between the spray gun and the sensing probe. For the sensing probes fabricated by the optical deposition method, the thickness of the CNT overlay can be optimized through varying the intensity of the amplified light from the EDFA and the duration of deposition. Apart from this, the morphology of the carbon nanomaterial overlay and its effect on the sensitivity of the various carbon nanomaterial-deposited sensing probes would also be explored.

The current range of RIs measured is limited by the type of test solution used. It has been shown in chapters 5 to 7 that the various carbon nanomaterial-deposited sensing probes have the potential to measure a larger range of RIs. Also as discussed in section 7.4, the theoretical limit on the range of RIs would be that of the RI of the carbon nanomaterials used. Hence, test solutions with a higher RI would be obtained and used to test for the maximum continuous detection range of the various carbon nanomaterial-deposited sensing probes.

Future work involving other carbon nanomaterial solutions would also be explored. The current CNT solution consists of a mixture of MWCNTs and SWCNTs. The RI sensing characteristic of the various sensing probes deposited with a CNT overlay consisting solely of SWCNTs or MWCNTs can be explored and a comparison in sensitivity can be made with the current CNT-deposited sensing probes. A similar study can be conducted using a monolayer of graphene prepared by methods such as CVD. In this case, the monolayer graphene would be in the form of a single sheet and can be mechanically wrapped around the sensing probe, presenting a simpler fabrication process than the currently used methods. Based on the optical properties of graphene discussed in section 4.1.1, a monolayer of graphene might be able to achieve a better sensing performance than the sensing probes currently being studied.

Applications into chemical sensing would also be explored. It has been shown that CNTs can be functionalized to allow certain molecules to bind to the functionalized sites on the CNTs [254, 255]. By functionalizing the CNTs before deposition, various analyte specific optical fiber-based sensing probes can be fabricated, opening up the possibility for applications in chemical sensing.

Finally, the multiplexing capabilities of the sensing probes would also be explored. This would involve the arraying of the sensing probes into a sensing network using wavelength division or time division multiplexing techniques to monitor a large area or several localized regions simultaneously. This can be extended even further by converting the entire network into a wireless network similar to that of the telecommunications networks of today.

List of Publications

Y. C. Tan, W. B. Ji, V. Mamidala, K. K. Chow, and S. C. Tjin, "Carbon-nanotube-deposited long period fiber grating for continuous refractive index sensor applications," *Sens. Actuators, B*, vol. 196, pp. 260-264, Jun. 2014.

Y. C. Tan, Z. Q. Tou, V. Mamidala, K. K. Chow, and C. C. Chan, "Carbon-nanotube-deposited photonic crystal fiber for refractive index sensing," in *Proc.SPIE-OFS23*, pp. 91577Y-91577Y-4, Jun. 2014.

Y. C. Tan, Z. Q. Tou, V. Mamidala, K. K. Chow, and C. C. Chan, "Continuous refractive index sensing based on carbon-nanotube-deposited photonic crystal fibers," *Sens. Actuators, B*, vol. 202, pp. 1097-1102, Oct. 2014.

W. B. Ji, **Y. C. Tan**, B. Lin, S. C. Tjin and K. K. Chow, "Nonadiabatically tapered microfiber sensor with ultrashort waist," *IEEE Photon. Technol. Lett.*, vol. 26, no. 22, pp. 2303-2306, Nov. 2014.

Y. C. Tan, J. Y. Huang and K. K. Chow, "Continuous Refractive Index Sensing based on Carbon-nanotube-deposited joint single-mode multi-mode fiber segment," *2015 IEEE Photon. Conf. (IPC)*, pp. 343-344, Oct. 2015.

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Y. C. Tan, Z. Q. Tou, K. K. Chow and C. C. Chan, "Graphene-deposited photonic crystal fibers for continuous refractive index sensing applications," *Opt. Express*, vol. 23, no. 24, pp. 31286-31294, Nov. 2015.

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Appendix A - MATLAB code of the Variation in Reflectance to a variation in RI of the Ambient Environment for Normal Incidence of Light at the Carbon Nanomaterial Overlay

```
lambda = 634;

n_fiber = 1.4446;

n_CNT = 2.71 + 1j.*(1.41);

d_CNT = 40;

n_ext = 1.33: 0.01 : 1.50;

k_CNT = (4.*pi.*n_CNT.*d_CNT)./lambda;

r_23 = (n_fiber - n_CNT)./(n_fiber + n_CNT);

r_34 = (n_CNT - n_ext)./(n_CNT + n_ext);

A = (r_23 + r_34.*exp(-1j.*k_CNT))./(1 + r_23.*r_34.*exp(-1j.*k_CNT));

R = A.*conj(A);

plot (n_ext,R);

xlabel('external refractive index'), ylabel('reflectivity');

hold all;
```

Appendix B - MATLAB code of the Variation in Reflectance to a variation in RI of the Ambient Environment for Light at an Incident Angle of 30° at the Carbon Nanomaterial Overlay

```
lambda = 1550;

n_fiber = 1.4446;

d_CNT = 40;

n_ext = 1.33: 0.01 : 4;

n_CNT = 2.71 + 1j.*1.41;

T1=0.523;

T2 = asin((n_fiber./2.71).*sin(T1));

T3 = asin((n_CNT./n_ext).*sin(T2));

k_CNT = (4.*pi.*n_CNT.*(d_CNT.*cos(T1)))./lambda;

r_23=(n_fiber.*cos(T1)- n_CNT.*cos(T2))./(n_fiber.*cos(T1) + n_CNT.*cos(T2));

r_34 = (n_CNT.*cos(T2) - n_ext.*cos(T3))./(n_CNT.*cos(T2) + n_ext.*cos(T3));

A = (r_23 + r_34.*exp(-1j.*k_CNT))./(1 + r_23.*r_34.*exp(-1j.*k_CNT));

R = A.*conj(A);

plot (n_ext,R);

xlabel('external refractive index'), ylabel('reflectivity');

hold all;
```