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Fibre Optic Long Period Gratings with Nano-Structured
Overlays

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Fibre Optic Long Period Gratings with Nano-Structured Overlays

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ABSTRACT

The influence of nano-structured overlays on the properties of fibre optic Long Period Gratings (LPGs) has been investigated. The presence of overlays of thickness of order 100 nm is shown to significantly alter the response of the LPG to environmental parameters, such as the surrounding refractive index. An LPG with a nano-structured overlay was observed to exhibit enhanced sensitivity to refractive indices lower than silica, and was observed to exhibit sensitivity to refractive indices greater than that of the silica cladding, not observed in the absence of the overlay. A theoretical model predicts the observed behaviour. The observation was exploited to enhance the sensitivity of an LPG based chemical concentration sensor.

The response of the transmission spectrum of concatenated long period gratings (LPGs) to the deposition of nano-structured overlays was also investigated. Concatenated LPGs act as a Mach-Zehnder interferometer and the phase of the interference fringes that appear within the LPGs' attenuation bands were shown to be highly sensitive to the optical thickness of the overlay, for thicknesses of order 100 nm. Several devices were fabricated with increasing path lengths to demonstrate effect of overlay thickness and refractive index on the transmission spectrum of the LPG.

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For my father,

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Chapter 1 Introduction to Long Period Gratings (LPG)

1.1 Introduction

Optical Fibres were first proposed as alternative waveguides for telecommunication applications by Charles Kao and George Hockham in the early 1960s and this idea was developed into a competitive product in 1970 when the benchmark loss level of 10dB/km was achieved. Without doubt this had a revolutionary impact on the telecommunications industry and the ensuing demand for optical fibres in engineering and research applications spurred further applications and so today a great variety of commercial optical fibre technology has become available¹. Notably this has given rise to an entirely new type of measurement device based on modern optical fibres; Fibre optic sensors (FOS).

1.2 Fibre Optic Sensors

FOS have been used in a number of applications and are slowly being converted into commercially available products^{2,3} where they offer viable alternatives in niche applications. Their importance and success has been the ability to overcome limitations of conventional electrical and mechanical sensors and to offer new additional advantages. For instance, whereas interference due to electromagnetic induction (EMI) restricted the applications of a conventional sensor, an optical sensor can come into its prime due to their inherent immunity to EMI. They offer other advantages over traditional sensors as they are compact, robust and chemically inert⁴. As optical fibres are non-conducting they can operate at zero potential, which makes them useful for safe operation in explosive environments. Fibre sensors can survive hazardous or inaccessible environments, for example, even stringent radiation tests. In addition to behaving as sensors the optical fibre can be used for data transmission and multiplexing, allowing large numbers of sensor elements to be placed in series using the same length of fibre².

Fibre optic sensors can be classified as either extrinsic or intrinsic¹; sensors based on fibre gratings are classified as intrinsic and have been studied extensively³. Fibre gratings are introduced within an optical fibre by periodic modulation of the refractive index core of an optical fibre. They have been one of the most useful developments in the field of fibre optics and can be classified in accordance with the period of the grating. The periodicity of a Long Period Gratings (LPG) is typically tens to thousands of microns rather than the periodicity of less than 1µm for the majority of Fibre Bragg Gratings (FBG). Both devices may be used as sensors but their sensing mechanism is based on different effects¹.

1.3 Fibre Bragg Gratings

The principle of operation commonly used in an FBG-based sensor system is to monitor the shift in the wavelength of the reflected “Bragg” signal due to the changes in the measurand. The Bragg wavelength or resonance condition of a grating is given by the expression³

$$\lambda_B = 2n_{\text{eff}}\Lambda$$

Equation 1.1

where n_{eff} is the effective refractive index (RI) of the fibre core and Λ is the grating period. Figure 1.1 illustrates the principle of operation. The optical fibre containing an FBG is illuminated with a broadband optical source. Light transmits through the fibre except at a wavelength where the forward mode couples with the reverse propagating mode and is reflected, at the Bragg wavelength. This spectral component is missing in the transmission spectrum, as shown. FBGs have been studied extensively as sensors⁵ and although offer clear advantages, they are limited by either sensitivity to strain or to temperature. Separating the response to either strain or temperature adds to the cost of the sensing system⁶ and additionally they require expensive demodulation schemes.

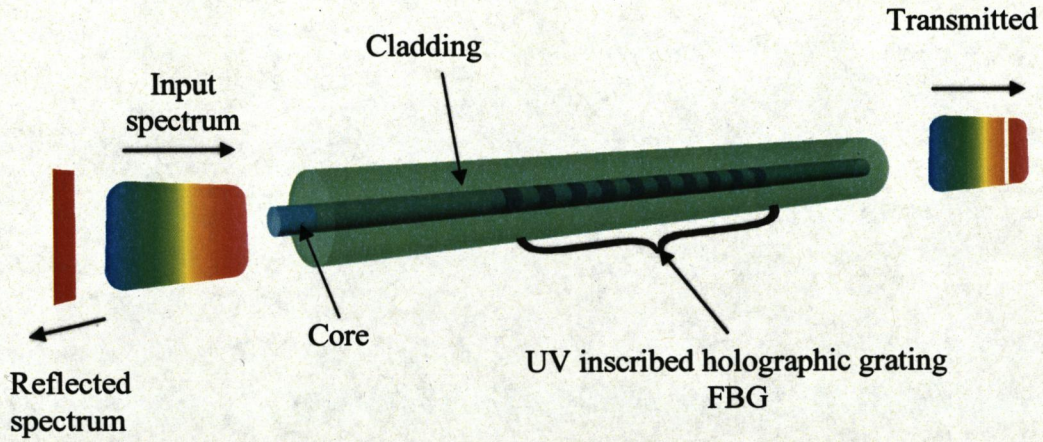


Figure 1.1: Schematic diagram of an FBG illustrating that only the wavelength of light, λ_B , that satisfies the Bragg condition, is reflected. Diagram reproduced from⁷.

1.4 Long Period Gratings

LPGs are a relatively new class of periodic structure within a fibre and were originally proposed in 1995⁸. These devices show sensitivity to the local environment surrounding the optical fibre. The LPG couples light between the guided core mode and the co-propagating cladding modes. The coupling occurs at discrete wavelengths when the phase matching condition is satisfied⁹,

$$\lambda = [n_{eff}(\lambda) - n_{cladd}^i(\lambda)] \Lambda \quad \text{Equation 1.2}$$

where $n_{eff}(\lambda)$ is the effective refractive index of the propagating core mode at wavelength, λ , $n_{cladd}^i(\lambda)$ is the refractive index of the i^{th} cladding mode and Λ is the period of the LPG.

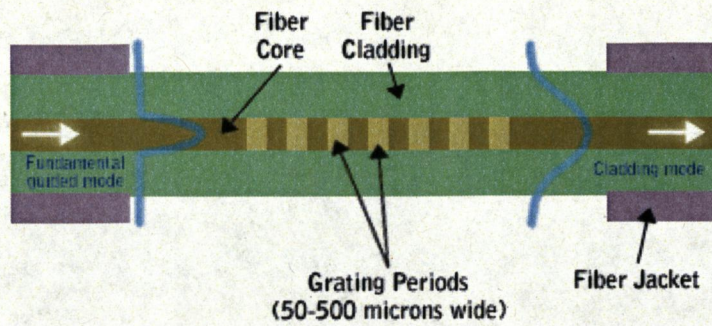


Figure 1.2: Schematic diagram to show the modes in an LPG. Diagram reproduced from¹⁰.

Figure 1.2 shows a cladding mode within the optical fibre. Once light is in the cladding, it decays rapidly due to high attenuation suffered by the cladding modes, resulting in attenuation bands or resonances in the transmission spectra of the fibre. Figure 1.3 illustrates the principle of operation. The centre wavelengths of the LPG resonances depend on the refractive index difference between the core and cladding and on the period of the LPG, as apparent from equation 1.2. External perturbations, temperature, bending, strain or, with particular relevance to this thesis, surrounding refractive index (SRI), can cause shifts in the resonances. Changes in these parameters can modify the period of the LPG and/or the differential refractive index of the core and cladding modes. Measurement of the change in central wavelength of attenuation bands, or changes in the extinction of the attenuation of the bands forms the basis of the operation of LPG sensor systems. An LPG is unique as it can have a different sensitivity for different fibre type, grating period and for each loss band making them ideal for multi-parameter¹¹ sensing using a single sensor element.

The attractive properties of LPGs have been exploited for a number of sensing applications¹¹. Refractive index measurements are required in chemical and biomedical applications and previous techniques have involved etching the cladding to gain access to evanescent field of a guided mode. LPG sensors circumvent the etching process to maintain the strength and integrity of the sensor¹¹. LPGs have been demonstrated as sensors capable of measuring the refractive index of a solution, allowing concentrations

of sodium chloride and ethylene glycol to be determined¹². Such sensors are not specific to a particular chemical species and are limited to use at only refractive indices less than or equal to the refractive index of the fibre cladding. Most LPGs are fabricated to operate at telecommunications wavelengths (1300nm and 1550nm) but fibres with lower cut-off wavelengths, 650nm, allow the use of low cost CCD spectrometers to monitor the response of the LPG during sensor applications.

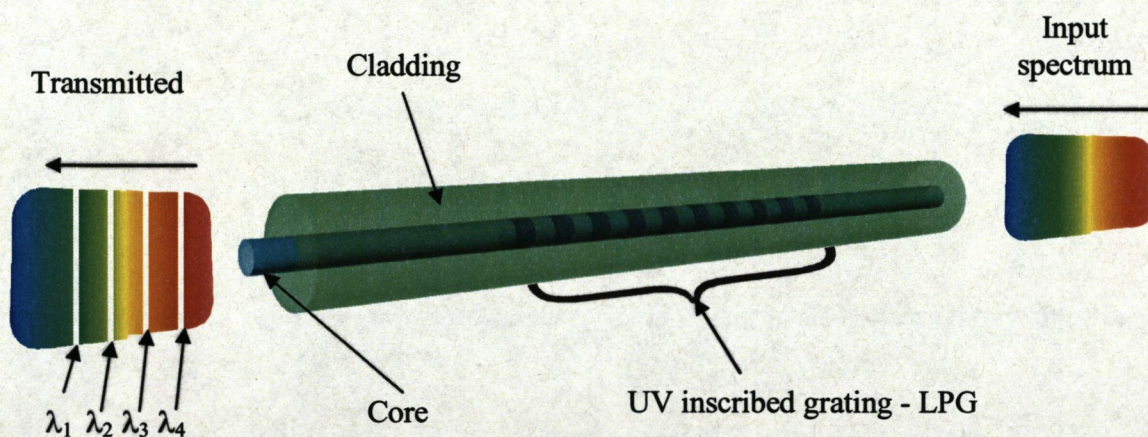


Figure 1.3: Schematic diagram of an LPG illustrating the attenuation bands situated in the transmission spectrum at wavelengths $\lambda_1 - \lambda_4$ which satisfy the phase matching condition. Diagram reproduced from⁷.

1.5 Thesis Objectives

LPGs have been introduced as a fibre optic sensor elements with several advantages that make them an attractive class of sensor and could lead to commercial applications, for instance as strain sensors in smart structure systems³. In particular the sensitivity LPGs exhibit to SRI is of interest and will be the focus of the research presented here. The use of active and passive nano-structured overlays deposited onto optical fibres containing LPGs is an exciting means to control the transmission spectrum. The interaction of the guided modes with the overlay offers potential for development of a new generation of

fibre optic sensors. The possibilities of depositing overlays which can undergo a change in RI itself due to exposure to local environment indicates the potential in this area to form, for example a tuneable loss filter¹³ or a chemical concentration sensor only responsive to, the chemical under observation¹⁴.

The research is centred on fabricating high quality optical fibre LPGs and depositing nano-structured overlays, using the Langmuir-Blodgett (LB) technique, directly onto the fibre. The refractive index and thickness of the overlay were used to influence the response of the LPG and the effect was investigated. The LB technique allows high resolution control of 3nm by depositing one molecular layer at a time of an organic overlay. Previous work shows that the transmission spectrum of an LPG is highly sensitive to the optical thickness of an overlay material¹⁵ and it is this sensitivity which will be exploited to develop and extend the sensor applications of this type.

In addition, the fabrication and use of concatenated LPGs, where two identical LPGs were fabricated in-series in the same fibre, will also be investigated. The cascaded LPGs function as a Mach-Zehnder interferometer and result in interference fringes within the transmission spectrum. The introduction of finer scale features within the relatively broad attenuation bands offers potentially higher resolution in the measurement of measurand induced changes in the mode propagation constants than is possible with a single LPG. The phase of the interference pattern is dependent upon a phase difference imposed by the LPGs, and upon the optical path difference between the core and cladding modes¹⁶. Deposition of overlays to investigate the interaction of the optical properties of the overlay with the varying optical path lengths was investigated, to demonstrate enhanced sensitivity.

1.6 Thesis Summary

Chapter One presents an introduction to fibre optical sensors and in particular fibre grating sensors are discussed. Emphasis is given to LPGs and the objectives of the thesis are outlined.

Chapter Two gives a concise theoretical description of LPGs. The coupling of power from the core mode to the cladding modes is discussed. The transmission spectrum of an LPG fabricated in-house is presented, showing the attenuation bands observed.

Chapter Three presents a review of the sensitivities of LPGs to a range of measurands. Emphasis is given to the response of LPGs to SRI as this provides the basis for the research presented in this thesis.

Chapter Four reviews the current fabrication methods available to fabricate an LPG, focusing on the UV irradiation technique as this was used at Cranfield for the fabrication of LPGs and concatenated LPGs.

Chapter Five outlines the LB technique for deposition of overlays on to optical fibres and discusses in detail the practical modifications required to allow deposition onto a cylindrical substrate. The technique is also compared with the competing technique of Electro-static Assembly.

Chapter Six presents the response of an LPG as a function of increasing thickness of an overlay. The experimental results are compared to a theoretical model. The characterisation of the LPG to SRI with and without overlays is investigated to identify the optimum thickness of overlay for enhanced response. An enhanced chemical sensor is then demonstrated using an overlay of a few hundred nm thickness.

Chapter Seven then extends the research by investigating the response of concatenated LPGs to overlays. A number of devices were fabricated with varying interference fringes in the attenuation. The overlay was deposited onto the optical fibre in two experimental configurations and the observed response was compared to theoretically predicted response.

Chapter Eight presents the conclusions of the research and discusses the proposed further work in this field.

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Chapter 2 Theory of Long Period Gratings

2.1 Introduction

Optical fibres act as waveguides as a result of their construction, whereby they are comprised of a core of refractive index higher than the surrounding cladding, enabling light to experience total internal reflection (TIR). The cladding also acts as a waveguide, as the surrounding air is of lower refractive index. The short grating wave-vector of an LPG promotes coupling between the propagating core mode and the co-propagating cladding modes. The transfer of power between co-directional modes is an effect particular to LPGs.

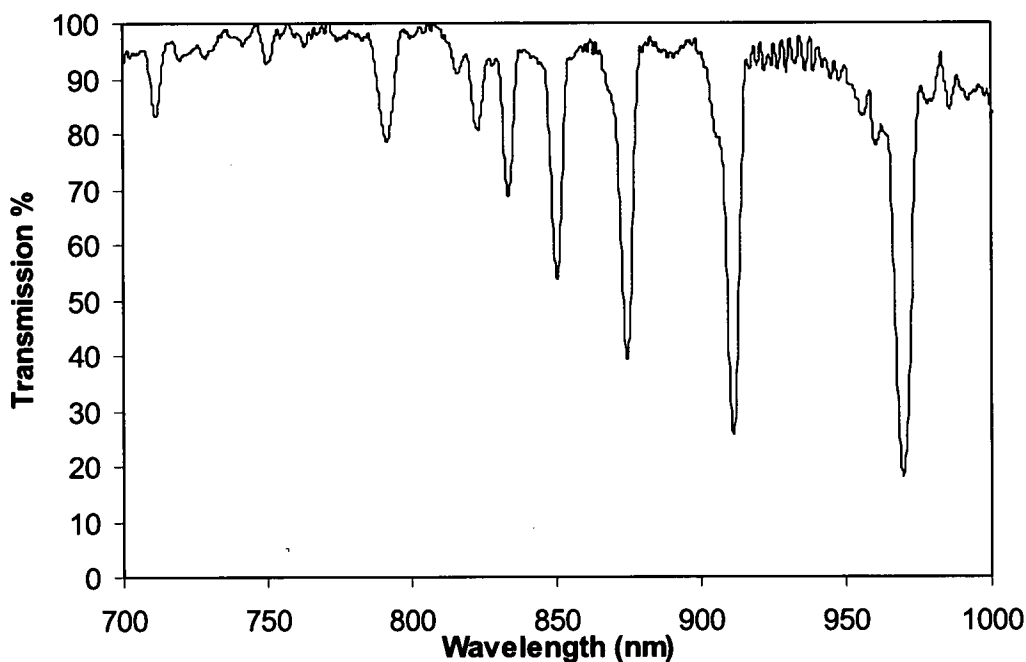


Figure 2.1: Transmission spectrum of an LPG showing several attenuation bands centred at discrete wavelengths. The LPG was 40mm in length and period 400 μ m, written using UV irradiation at 266nm through an amplitude mask. The fibre was photosensitive with a cut-off wavelength of 650 nm.

LPGs were introduced in Chapter One as optical devices formed by the result of periodic RI perturbations in the core of the fibre, with a longer period than that of fibre Bragg gratings. The result of these perturbations are attenuation bands within the transmission spectrum of an LPG, a typical spectrum shown in Figure 2.1.

The central wavelength of the attenuation bands are determined by the phase-matching condition, which in turn is dependent on the difference in the effective refractive index of the cladding and the core modes, and on the period¹. The minimum transmission, T_0 , and the width of the attenuation bands are governed by the coupling efficiency between the core and the cladding modes and by the length of the LPG². This chapter briefly presents the theoretical background of LPGs and discusses the transmission spectrum observed.

2.2 Principles of operation

An optical fibre can be considered as a dielectric waveguide. Dielectric waveguides have been studied extensively in the past³. Optical fibres consist of a central core surrounded by a cladding with a refractive index lower than the core. Light incident on the interface between the core and the cladding of the optical fibre undergoes total internal reflection (TIR), provided that the angle of incidence is greater than the critical angle, θ_c , as shown in Figure 2.2. The propagating light is said to be guided and forms the basis for communication and sensor systems employing optical carriers. The majority of the light remains confined within the core and an exponentially decaying tail extends into the cladding⁴.

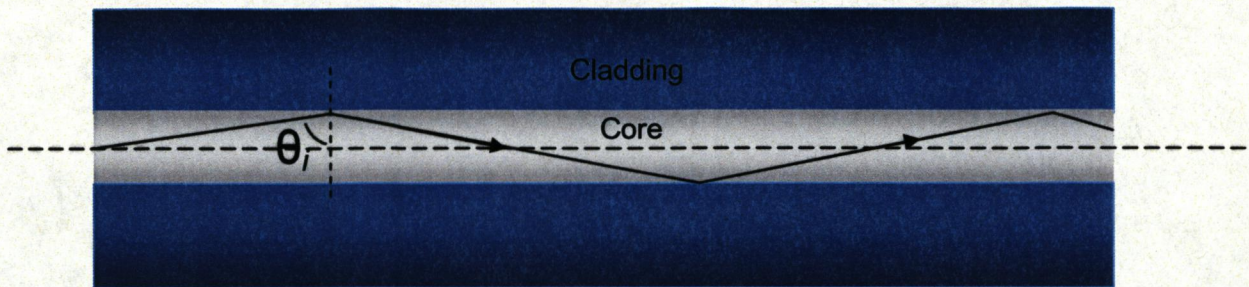


Figure 2.2: A schematic to show a light ray propagating within the core of an optical fibre when experiencing TIR, where the angle of incidence $\theta_i > \theta_c$. Also shown is the cladding of lower refractive index surrounding the core and the normal plane.

The propagating light rays cannot take any arbitrary angle greater than the critical angle. The rays must propagate at discrete angles associated with guided modes⁵ due to the phase of the rays, which are not depicted by this simple ray diagram³. The phase change suffered by the wave upon reflection will contribute to the propagation of light only at discrete values of θ_i . In an unperturbed optical fibre system with no loss and at any angular frequency, ω , optical fibres can support a finite number of guided modes. A mode in an optical fibre is a set of electromagnetic waves that participate in the propagation of energy in the fibre⁶. The guided modes travel in the fibre, without the loss or gain of power, with discrete propagation constants, β , that are functions of the optical source's wavelength and the properties of the fibre itself; such as the shape of the core, size of the aperture and nature of the optical fibre. The propagation constant can be determined using Maxwell's equations and the electric and magnetic field boundary conditions at the core and cladding interface^{5,6}. Fibres with a large core and large numerical aperture, such that they support a large number of modes, can be adequately analysed by the tools of geometrical optics⁵.

This research used step-index single mode fibre for fabrication and characterisation of the LPG devices. Fibres which exhibit a discontinuity in the index of refraction at the boundary between the core and the cladding are termed step-index fibres. A single mode fibre allows one mode to propagate through the core of the fibre. This is achieved by a reduction of the diameter of the core to eliminate all other modes, transmission of

appropriate wavelengths greater than cut-off wavelengths of the fibre and reduction of the refractive index difference between core and cladding. Figure 2.3 shows the schematic cross section of a single mode step index fibre.

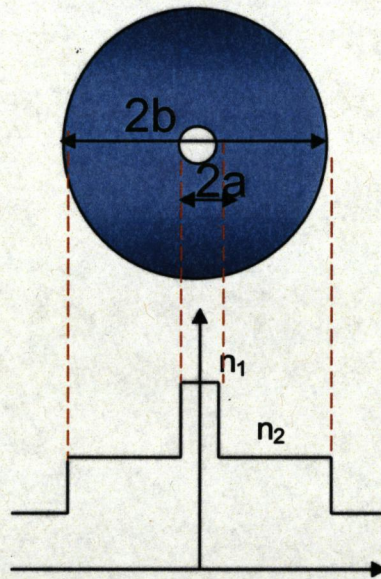


Figure 2.3: A schematic of the cross section and RI profile of the fibre configuration of step-index, single mode fibre. Where n_1 and n_2 are the refractive indices of the core and cladding respectively and $n_1 > n_2$. The radius of the core is represented by a and that of the cladding is represented by b .

2.3 Guided and Radiation modes

The propagation constants for the guided modes have previously been shown to take discrete values confined to $n_2 k_0 < \beta < n_1 k_0$, where k_0 is the free space propagation constant at a wavelength of light λ , $k_0 = 2\pi/\lambda$. Figure 2.4 illustrates the propagation constants of the forward ($\beta > 0$) and reverse propagating ($\beta < 0$) guided modes as functions of the optical frequency in a lossless unperturbed fibre. The β plot represents the modal propagation constants in terms of frequency⁸ and the propagation constant is given by Equation 2.1⁷.

$$\beta = \frac{\omega n_{\text{eff}}}{c} = \frac{2\pi n_{\text{eff}}}{\lambda} \quad \text{Equation 2.1}$$

Where n_{eff} is the effective index of refraction, $n_2 < n_{\text{eff}} < n_1$, from the cladding to the core and c is the speed of light in a vacuum.

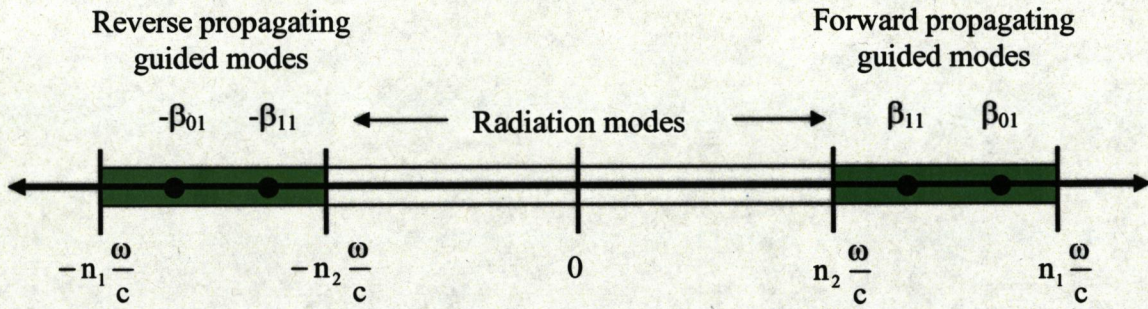


Figure 2.4: Propagation constants of the forward and reverse propagating guided modes in an optical fibre. β_{01} and β_{11} are the propagating constants of the two lowest order forward propagating guided modes. Diagram reproduced from ⁸.

In addition to the guided modes, another set of modes also satisfies the electromagnetic Maxwell's equations and the boundary values³. These modes are called radiation modes and continuous loss of power from these modes is observed⁹. The infinite number of radiation modes present have continuous complex propagation constants in the range, $0 < \beta < n_2 k_0$ ⁷, as illustrated in Figure 2.4. The infinite radiation modes are strongly influenced by the outer cladding boundary so that some of the radiation modes are trapped by the cladding-air interface and result in modes called cladding modes³. If the RI of the medium surrounding the cladding is n_3 , the cladding modes' propagation constants are bound within the range, $n_3 k_0 < \beta < n_2 k_0$.

Cladding modes may travel in either the forward or reverse direction and have discrete propagation constants as illustrated in Figure 2.5. The cladding modes attenuate rapidly due to bends, scattering losses and absorption of cladding. The periodic modulation in the core of an optical fibre, corresponding to the LPG, causes the coupling of energy propagating in the guided core modes into discrete cladding modes. For a typical LPG, different cladding modes satisfy the phase-matching condition at different wavelengths, shown in Equation 1.2, and so a number of attenuation bands are normally observed. The difference between the propagation constant of the guided mode and the wave-vector of the grating is equal to the propagation constant of at least one cladding mode. Thus co-directional coupling occurs between the core and cladding modes.

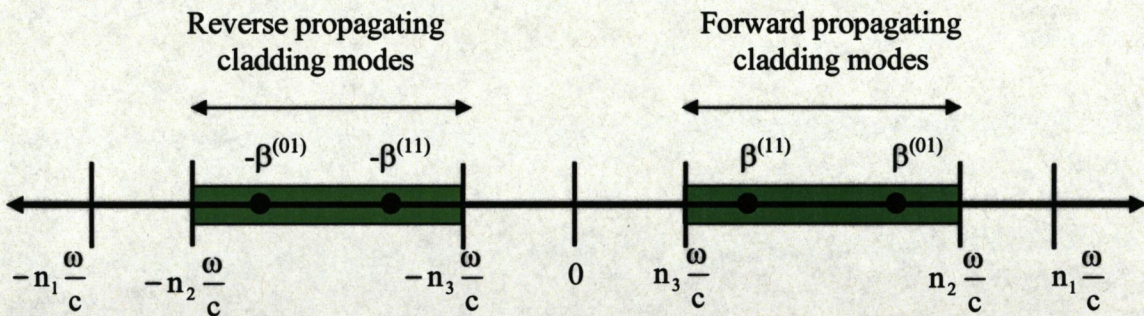


Figure 2.5: Propagation constant distribution of the cladding modes in an optical fibre.

The coloured region represents the cladding modes, where β_{01} and β_{11} are the propagation constants of the two lowest order forward cladding modes.

Diagram reproduced from ⁸.

2.4 Coupling to guided modes in long period gratings

The LPGs presented within this thesis are designed to couple the guided mode to the cladding modes. However, LPGs can also couple light to other guided modes and these will be briefly discussed here. LPGs that couple light from the fundamental LP_{01} mode to other forward-propagating guided modes were first fabricated to couple to the LP_{11} mode¹⁰. The coupling in this case occurs from a circular symmetrical mode to an asymmetric mode and therefore to achieve efficient coupling, the grating needs to be

blazed¹¹. Blazing is achieved by orientating the fibre axis at an angle to the corrugations of an amplitude mask rather than orientation to the normal⁵. The mode coupling occurs at wavelengths where the phase matching condition, $\Delta\beta = 2\pi/\Lambda$, is satisfied. Changing the periodicity or the blaze angle of the grating can change the wavelength that coupling occurs and LPGs with 96% conversion efficiency have been demonstrated by changing these two parameters¹⁰. LPGs that couple the fundamental guided mode to the LP₀₂ mode have also been demonstrated¹². However since both LP₀₁ and LP₀₂ modes possess circular symmetry, blazing was not required to achieve coupling.

2.5 Grating parameters

The parameters that determine the central wavelengths and the minimum transmission values of the attenuation bands will now be discussed. The dependences of the transmission spectrum upon the length of the LPG and the coupling coefficient are also considered.

2.5.1 Wavelength separation

The separation of the central wavelengths of two attenuation bands in the transmission spectra of an LPG can be determined. The central wavelengths separation can be observed to increase with the increasing order of cladding mode to which coupling occurs, as illustrated by Figure 2.1. The wavelength separation between two attenuation bands of order m and $m+1$ at wavelength $\lambda^{(m)}$ and $\lambda^{(m+1)}$, can be determined⁸. Firstly the wavelength of the m^{th} order attenuation band relative to the cut-off wavelength of the fibre, λ_{cut} , is calculated using Equation 2.2,^{8,13}

$$\lambda^{(m)} - \lambda_{\text{cut}} \approx \frac{\lambda_{\text{cut}}^2 \Lambda}{8n_2} \frac{m^2}{b^2} \quad \text{Equation 2.2}$$

where the effective RI of the guided mode (n_{eff}) is assumed to be equal at $\lambda^{(m)}$, and λ_{cut} ⁸. The RI of the cladding mode is given by n_2 , m is an integer and b is the cladding radius of the optical fibre. Equation 2.2 shows that the wavelength separation between $\lambda^{(m)}$ and λ_{cut} is an increasing function of the order, m , and of the periodicity, Λ , of the LPG. In an LPG with a constant period, as in the case for the LPGs used in this investigation, the wavelength separation would be expected to increase for higher order cladding modes⁷.

When m is small, as is the case for the first few cladding modes, $\lambda^{(m)}$ and λ_{cut} can be assumed to be very close and the wavelength separation $\delta\lambda^{(m, m+1)}$ between the attenuation bands of order m and $m+1$ can be obtained from Equation 2.3⁷,

$$\delta\lambda^{(m, m+1)} = \lambda^{(m+1)} - \lambda^{(m)} \approx \frac{\lambda_{\text{cut}}^2 \Lambda}{8n_2} \frac{2m+1}{b^2} \quad \text{Equation 2.3}$$

As can be seen from Equation 2.3, the wavelength separation between the attenuation bands increases as the value of m is increased. This is consistent with the central wavelengths of the attenuation bands observed in the transmission spectrum of an LPG.

2.5.2 Power transmission

Coupled mode analysis can be used to determine the power transmitted through an LPG and the minimum transmission values of the attenuation bands. This allows the fabrication of LPG devices with the characteristics that are suitable for a given application. The minimum transmission of the attenuation bands at the phase matched is governed by the expression¹⁴,

$$T_0 = 1 - \sin^2(\kappa^{(m)} L) \quad \text{Equation 2.4}$$

where $\kappa^{(m)}$ is the coupling coefficient for the m th cladding mode and L is the length of the

LPG. Equation 2.4 shows that minimum power in the guided mode is dependant on the coupling coefficient and the length of the LPG. Power is completely transferred to the cladding mode when Equation 2.5 is satisfied⁸.

$$\kappa^{(m)} L = \frac{\pi}{2} \quad \text{Equation 2.5}$$

The fibre can be exposed to the UV irradiation until the required Δn , the refractive index change in the fibre core, has been achieved; and the exposure stopped when the minimum transmission value of the central wavelength of the attenuation band is achieved.

The power in the cladding mode can start to couple back into the guided mode when the following condition exists, $\kappa^{(m)} L > \pi/2$. This leads to an increase in the guided mode transmitted power. For a fixed value of Δn , the complete power transfer occurs for a particular cladding mode and all other cladding modes are either under or over coupled. Hence, it is expected that all the attenuation bands will have different minimum transmission values, as illustrated in Figure 2.1. The central wavelength of the attenuation band most suitable for the final application, can be monitored during fabrication to achieve the required minimum transmission⁷.

2.6 Modelling fibre optic long Period Gratings

LPGs can be modelled in order to predict the coupling wavelengths and minimum transmissions as a function of the LPG's periodicity to assist in the design of the LPG prior to fabrication¹⁵. The parameters for determining the coupling between the modes of an LPG can be determined using coupled mode theory¹⁶. Although an exact theoretical treatment of optical waveguides is possible¹⁷ it must be performed in cylindrical coordinates and becomes very complicated leading to 6 component equations of the homogenous wave equations¹⁸. Therefore, a simplified model of propagation within optical fibres is usually used using the weak field approximation as reported by Gloge⁹.

2.6.1 Cladding mode analysis

An exact analysis of the effective indices and the mode profiles of the cladding have been carried out using the vector field method¹⁹. This takes into account the presence of the core and therefore the overlap between the mode propagating in the core and the cladding can be calculated accurately. A simpler investigation of the cladding modes uses the approximation that the fibre can be considered as an ideal step index structure and ignores the presence of the core^{8,3}. For a weakly-guided fibre with closely matched values of core and cladding refractive indices, this assumption has provided a good approximation of the cladding mode propagation constants⁸. However, since this approach predicts that the majority of the energy propagating in the cladding mode is outside the core, there is little overlap between the propagating core and cladding modes. The approximation cannot be used to predict the coupling efficiency because the value for the overlap integral is highly inaccurate.

The central wavelength, λ_i , of the attenuation band corresponding to coupling to the i th mode are given by the solutions of the phase matching equation²⁰ as shown in Equation 1.2. To determine the coupling wavelengths (λ_i), the effective refractive indices of the core and cladding modes are calculated as functions of wavelength and this is used in the phase matching equation to solve for the wavelengths as a function of the periodicity. Using the method presented in [20], the graphs shown in Figure 2.6 and Figure 2.7 were produced²¹. The figures show the dependence of the coupling wavelength for low order cladding modes and high order cladding modes upon the period of the LPG, assuming that the fibre's cut-off wavelength is 650nm.

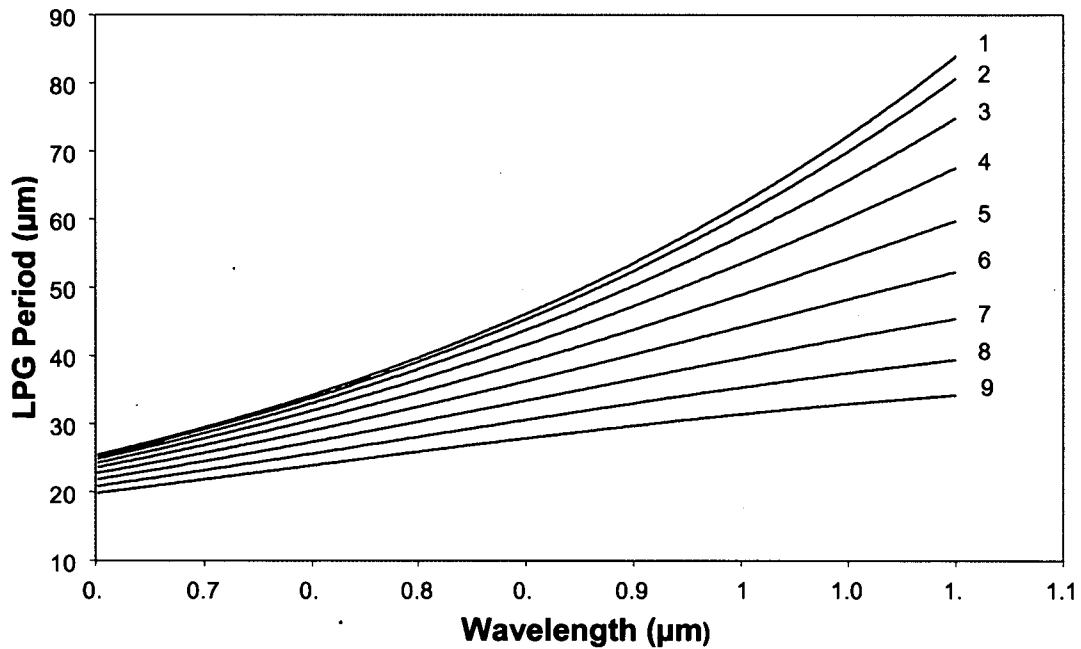


Figure 2.6: Graph of the resonant wavelength as a function of LPG period for coupling between the guided core mode and the cladding modes of order 1-9. Reproduced from²¹.

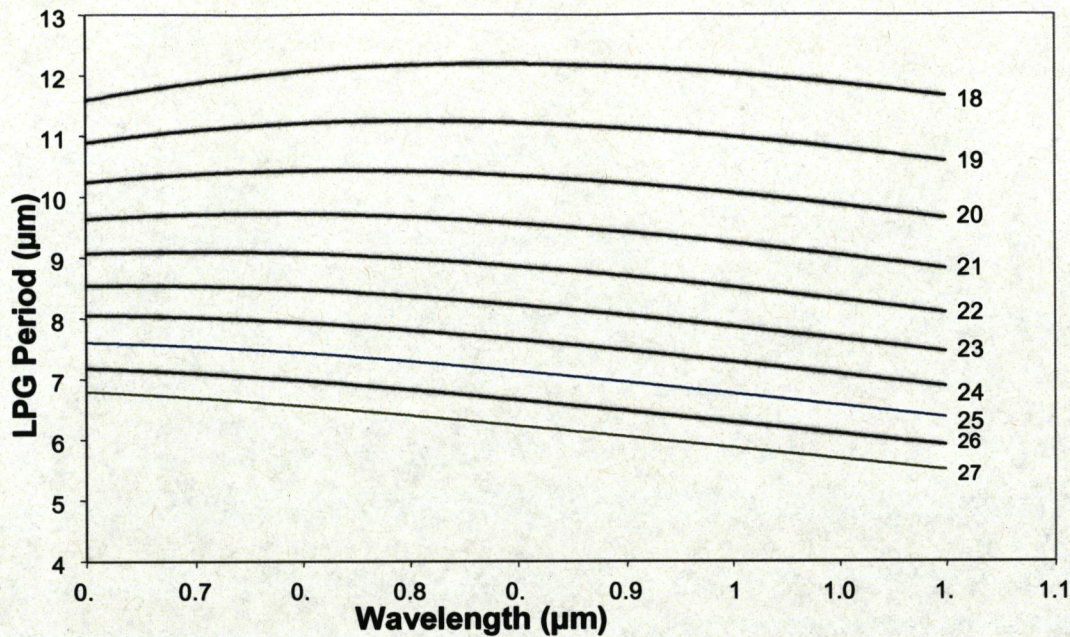


Figure 2.7: Graph of the resonant wavelength as a function of LPG period for coupling between the guided core mode and the cladding modes of order 18-27. Reproduced from²¹

Figure 2.6 shows that coupling to low order-cladding modes is achieved using longer periods, while Figure 2.7 shows that shorter periods facilitate coupling to higher order modes. It is also possible to choose the period of an LPG to generate attenuation bands with positive or negative responses to the measurands, as required. For positive responses to the measurands where the gradient, $d\lambda/d\Lambda$, of the graph is positive, the LPG's period should be $> 200\mu\text{m}$ and for negative responses, where the gradient of the graph is negative, the period should be $< 90\mu\text{m}$. It can be seen from Figure 2.7 that coupling to higher order cladding modes, for instance cladding mode 18, is possible at two wavelengths. The concomitant formation of two attenuation bands would therefore be observed²².

Depending on the operating wavelength, the attenuation band will exhibit either a positive or a negative response to the measurands. At the lower operating wavelength the slope and hence the response exhibited is positive, while at higher wavelengths the opposite conditions and response are observed. Thus small changes in the coupling conditions could result in large changes in the separation of the two attenuation bands. For LPGs fabricated such that their period coincides with the peak of the curve, for example, mode 18, it has been observed that changes in the coupling conditions result in a change in the coupling efficiency, but not in the wavelength. These devices were shown to offer greater resolution and overcame the need for spectrum analysers²³.

2.7 Chapter Summary

This Chapter has presented the principles of operation of LPGs and discussed the transmission spectrum of an LPG. The central wavelengths and the minimum transmissions of the observed attenuation bands within the spectrum were shown to depend on some key parameters. Knowledge of the dependence of the transmission spectrum on these parameters allows the fabrication of s with characteristics optimised for specific applications.

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Chapter 3 Review of LPG Sensors

3.1 Introduction

Fibre optic sensors (FOSs) offer a number of advantages over conventional sensors, as discussed in Section 1.2, including inherent immunity to electromagnetic interference and chemical inertness¹. However, as yet they have not penetrated the market to displace conventional electro-mechanical sensors which are well established, have proven reliability records and have low manufacturing costs².

Since the first report of LPGs in 1995 by Vengsarker *et al*³, this new type of fibre grating has been investigated for many applications. They were initially developed for use as band rejection filters^{3,4} and as such have been used for gain-flattening of erbium-doped fibre amplifiers (EDFA's)⁵. LPGs have also presented a unique opportunity for application FOSs and these sensor applications will be reviewed here.

In single mode optical fibres, an LPG couples the fundamental core mode and multiple forward propagating cladding modes and therefore can offer greater sensitivity to external measurands than Fibre Bragg Gratings. The exact form of the transmission spectrum and the centre wavelengths of the attenuation bands are sensitive to local environmental parameters such as temperature, strain, bend radius and refractive index of the medium which is surrounding the fibre⁶. Changes in these environmental factors can modify the core or cladding refractive indices, or the period of the LPG, which in turn modifies the phase-matching conditions for coupling to the cladding modes. This manifests itself as a change in the coupling wavelengths.

The sensed information is encoded directly into wavelength, an absolute parameter that does not depend on source power, losses associated with connectors and couplers^{7,8}. This gives the sensor devices based on LPGs a built-in self-referencing capability and good repeatability. The sensitivity to particular measurands is dependant on parameters such as the grating period and the material attributes of the optical fibre used, including the refractive index and the dopants used, to name two. It is therefore important to choose carefully the parameters when the sensor is being

designed and manufactured to ensure the sensitivity exhibited is in the region required. Appropriate selection can be used to enhance sensitivity to a particular measurand whilst reducing sensitivity of another, overcoming the problem of cross sensitivity.

3.2 Sensitivity to Temperature

The transmission spectrum of an LPG contains several attenuation bands, as illustrated in Figure 2.1. Each band displays distinct sensitivities not only to different measurands but also to the same measurand. Generally, the bands at longer wavelengths tend to exhibit greater sensitivity⁹.

An attenuation band can have different responses depending on the grating period, the order of the cladding mode to which coupling takes place⁹ and the fibre composition¹⁰. It is therefore possible to fabricate sensors which display either enhanced temperature sensitivity or are temperature insensitive. Such insensitivity is useful, for example, to enable single parameter measurement where multiple effects are acting simultaneously on the grating, or in the telecommunications industry where spectral stability is important. LPGs fabricated in standard telecommunications optical fibre have been shown to exhibit sensitivities in the range 3nm/ 100°C⁹, an order of magnitude larger than Fibre Bragg Gratings (FBGs) making thermal response an important factor to consider. Therefore the correct choice of the period and fibre composition is vital in assisting the optimisation of the thermal sensitivity for the given application, and some methods used to achieve this are discussed later in this chapter.

The sensitivity of long period gratings to temperature can be examined by differentiating Equation 1.2. to yield⁹:

$$\frac{d\lambda}{dT} = \frac{d\lambda}{d(\delta n_{\text{eff}})} \left(\frac{dn_{\text{eff}}}{dT} - \frac{dn_{\text{cl}}}{dT} \right) + \Lambda \frac{d\lambda}{d\Lambda} \frac{1}{L} \frac{dL}{dT} \quad \text{Equation 3.1}$$

Where λ is the central wavelength of the attenuation band, T is the temperature, n_{eff} is the effective refractive index of the core mode, n_{cl} is the effective refractive index of the cladding mode, $\delta n_{eff} = (n_{eff} - n_{cl})$ the differential effective refractive index, L is the length of the LPG and Λ is the period of the LPG.

There are two terms on the right-hand side of the equation which contribute to the overall wavelength shift exhibited by the LPG and hence the grating's thermal sensitivity. The first of these terms can be thought of as the material contribution as it relates to a change in the differential effective index due to the thermo-optic effect and is a function of the fibre composition. It is strongly dependant on the order of cladding mode to which coupling occurs. The second term represents the waveguide dispersion contribution related to the thermal expansion of the fibre and concomitant changes in the LPG period¹¹.

When coupling to low-order cladding modes occurs, by using LPGs with longer periods ($\Lambda > 100\mu\text{m}$), the material effect dominates since $dn_{eff}/dT > dn_{cl}/dT$. As the thermal expansion coefficient of silica is very small ($\sim 0.5 \times 10^{-6}/^\circ\text{C}$), the waveguide contribution effect can be ignored (central wavelength shift of less than $0.001\text{nm}/^\circ\text{C}$)¹² because it is far less than the spectral shift caused by the thermo-optic effect⁹. For coupling to higher-order modes, accessed using shorter periods ($\Lambda < 100\mu\text{m}$), the material effect for standard germanosilicate fibre can be considered negligible. This is because dn_{cl}/dT approaches dn_{eff}/dT as the modes undergo larger changes to their refractive indices. The magnitude of the material effect is a function of the relative change between the effective indices and can have either polarity⁹. The strong dependence of the material contribution to the order of coupling for standard long period gratings with periodicity of a few hundred micrometers is what provides the distinct wavelength shifts observed.

The waveguide contribution similarly can be negative or positive depending on the slope $d\lambda/d\Lambda$ of the characteristic curve corresponding to the appropriate cladding mode. For coupling to low-order cladding modes $d\lambda/d\Lambda$ is positive as can be seen from Figure 2.6 ($\Lambda > 200\mu\text{m}$) and negative for higher-order modes as apparent by Figure 2.7 ($\Lambda < 90\mu\text{m}$). Since the period of the gratings dictate the modes to which

coupling occurs, it is feasible to vary temperature response considerably by appropriate choice of the LPG period.

Careful selection of various parameters effecting the two contributions can be used to either balance the sensitivity to produce a temperature-independent loss bands or heightened sensitivity loss peaks (positive or negative) as required.

In addition to the shifts in the central wavelength, a change in amplitude of the resonance wavelengths can be observed for LPGs with varying strengths of κL . As can be seen from Equation 2.4, the minimum transmission of the attenuation bands is dependant on the value of κL and the magnitude of sensitivity is amplified by this factor. For LPGs with $0 < \kappa L < \pi/2$ the amplitude increases with temperature while for LPGs with $\pi/2 < \kappa L < \pi$ the amplitude decreases. For LPGs with $\pi < \kappa L < 3\pi/2$, the transmission spectra results in the splitting of the loss peaks into two comparable peaks¹².

The response of LPGs to varying temperature has been found to be linear at non-cryogenic temperatures as illustrated by Figure 3.1 and Figure 3.2; a linear fit to the wavelength shift yields a sensitivity of 9.3nm/100°C in LPGs with a period of 280µm written in Corning SMF-28 fibre using a UV laser. This is almost an order of magnitude higher than that observed in an FBG. Intensity variation with temperature at a particular wavelength is also an important characteristic which enables simple demodulation schemes for the long-period gratings⁹.

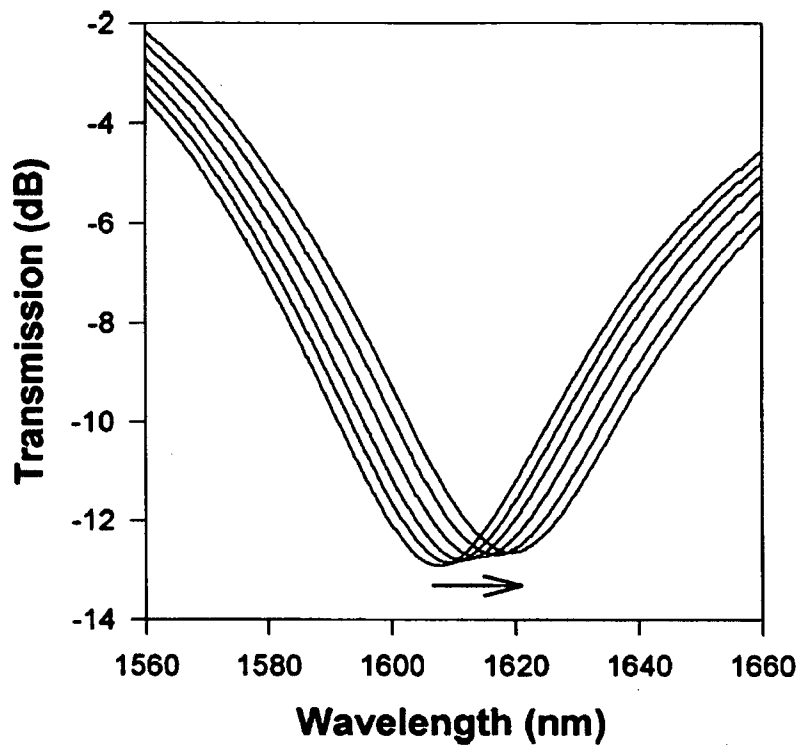


Figure 3.1: Shift in the central wavelength of an LPG attenuation band with temperatures at 22.7°C , 49.1°C , 74.0°C , 100.9°C , 127.3°C and 149.7°C from left to right. The LPG was fabricated with a period of $280\mu\text{m}$ in Corning SMF-28 fibre. Diagram reproduced from⁹.

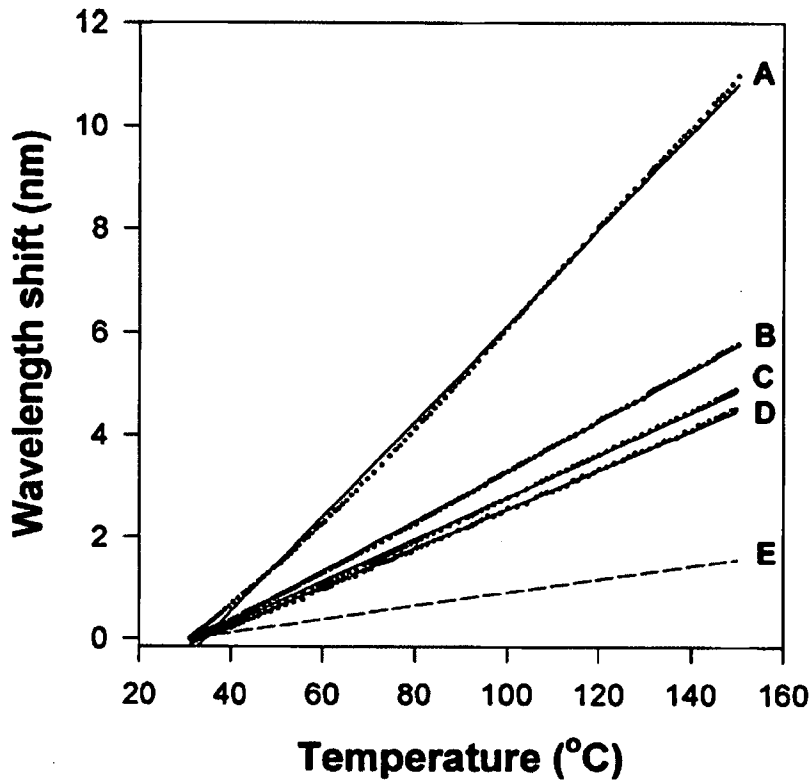


Figure 3.2: Shift in the central wavelength of four attenuation bands A-D, as a function of temperatures at 22.7°C, 49.1°C, 74.0°C, 100.9°C, 127.3°C and 149.7°C from left to right. The dashed line, E, is the wavelength shift for a Bragg grating at 1550nm with temperature coefficient 1.3nm/100°C for comparison. The LPG was fabricated with a period of 280µm in Corning SMF-28 fibre. Diagram reproduced from⁹.

The temperature response of an LPG has been shown to be non-linear at cryogenic temperatures below 77 K and to still be measurable at temperatures down to 20 K¹³. In comparison, the thermal response of fibre Bragg gratings has been shown to be largely temperature insensitive below 100 K¹⁴. The extended range in the thermal sensitivity displayed by LPGs is a result of the dependence of the coupling wavelength upon the differential index of the core and cladding, whereas the thermal response of a FBG arises largely due to the thermal expansion of the fibre material².

Several methods have been reported to enhance the thermal sensitivity to demonstrate a temperature sensor with high resolution, or to produce widely tuneable spectral filters,. As mentioned in the introduction, the choice of periodicity, the order of mode

to which coupling occurs and the operating wavelength all contribute in effecting the thermal response exhibited.

The doping of the core of the fibre with boron and germanium to increase the photosensitivity and thus provide a greater differential thermal response was shown to increase the sensitivities up to $275 \text{ nm}/100^\circ\text{C}$. The LPG was fabricated with a periodicity of $240\mu\text{m}$ in the conventional B-Ge co-doped fibre and a total of 27.5nm spectral shift was achieved from only a 10°C change in temperature. Such a large enhancement of the thermal response could lead to extremely high resolution temperature sensors and the large shift over a small range temperature range is well suited for efficient tuneable loss filter applications¹⁵.

Other techniques to enhance the thermal response have involved surrounding the fibre by a material of large thermo-optic coefficient. This results in the LPG responding to the compounded effect of changes in temperature and to the temperature induced RI change of the surrounding medium. Temperature sensitivities of up to $19\text{nm}/^\circ\text{C}$ over a temperature range of 1.1°C have been reported employing this method¹⁶. A novel procedure involving uniquely forming two cladding layers on the fibre has been presented recently. This consisted of HF-etching the fibre to form an ultra-thin ($28 \mu\text{m}$) original fibre cladding onto which a second cladding was deposited. The ultra-thin LPG was placed into a cell containing a liquid crystal, and, by controlling the temperature to manipulate the refractive index of the liquid crystal, a wavelength shift of 46nm was observed over a temperature range of 22°C . This provides an excellent method to make a highly sensitive tuneable filter, with tuning efficiency of $2.1\text{nm}/^\circ\text{C}$ in a temperature range from 48°C to 70°C ¹⁷.

Enhanced temperature sensitivities have also been reported using polymer coatings. Gratings coated with a polymer exhibit a nearly linear temperature shift of approximately $80\text{nm}/100^\circ\text{C}$ compared to the temperature dependence of $5\text{nm}/100^\circ\text{C}$ displayed by the LPG with no polymer coating. Although these results were obtained using standard dispersion shifted fibre, special fibres were also investigated. Microstructured fibre, which consists of an inner cladding region surrounded by a ring of air was used. These air pockets were filled with the same polymer followed by subsequent UV-irradiation. A nearly linear temperature response of $74\text{nm}/100^\circ\text{C}$ was

observed which was enhanced from just 3nm/100°C when no polymer was inserted into the air-cladding¹⁸.

Recently highly temperature sensitive LPGs have been fabricated in single-mode photonic crystal fibre using focused pulses from a CO₂ laser, which results in a periodic stress relaxation. This method did not geometrically deform or elongate the fibre but achieved thermal sensitivities of 10.9pm/°C with strain-insensitivity¹⁹.

The parameters in Equation 3.1 can be employed to enhance the sensitivity and conversely can also be used to design temperature-insensitive LPGs^{20,21,11}. Utilisation of a small grating period (40µm) has been shown to reduce the temperature sensitivity such that it may be considered negligible at 1.8pm/°C, an order of magnitude smaller than that of an FBG, thus allowing temperature independent strain measurements to be undertaken²⁰. There have been reports of the manipulation of the refractive index of the outer cladding to completely eliminate the thermal response. By using a polymer to recoat the cladding and inducing a shift equal and opposite of the thermal response, a temperature insensitivity of 0.07nm/100°C was achieved²². Another method to reduce the temperature sensitivity employed a multi-layer RI profile optical fibre. However the expense associated with the fibre would make it unsuitable for commercial use²³.

A different approach involving the use of athermal packaging has been demonstrated to very successful effect. This method consisted of bonding the LPG to a substrate whose thermal expansion coefficient induced a strain that produced a wavelength shift of the opposite sign to the temperature induced shift. The thermal response was reduced by approximately 45% by bonding the LPG to an aluminium cylinder²⁴.

In addition to using refractive index to modify thermal response, it is also possible to exploit the bend sensitivity to achieve similar results; in fact a reduction by two orders of magnitude has been shown²⁵. This was achieved by attaching a bent LPG onto a mount with a suitable thermal expansion coefficient which acted to cancel the effect of temperature induced shift.

Metal-coated LPGs have been used to develop low power tuneable filters based upon resistive heating. This has been achieved by depositing thin films of copper (Cu)²⁶, or platinum (Pt)²⁷, of thickness approximately 300nm onto the LPG. To provide good adhesion for the thin metal films a 15nm layer of titanium (Ti) was deposited initially. The RI of the surrounding layer of titanium produced a 1nm wavelength shift in the attenuation bands and a small reduction in the coupling efficiency. The Cu-coated fibre exhibited a 4nm wavelength shift for electrical power of 0.5W²⁶, while for the Pt based filter an 11nm wavelength shift was reported for 0.67W, with a linear efficiency of 16nm/W²⁷. The indices of refraction of Ti and Pt at 1.55µm are 4.04 and 5.31 respectively. These are greater than that of silica thus, the metal coating was found to make the performance of the LPG insensitive to the RI of the medium surrounding the metal coating²⁷.

In this section the sensitivity of an LPG to temperature was reviewed. The techniques for fabricating temperature sensitive or insensitive LPGs was discussed.

3.3 Sensitivity to strain

The sensitivity of a LPG to axial strain ϵ can be predicted by expanding the phase matching equation and rearranging to give⁹:

$$\frac{d\lambda}{d\epsilon} = \frac{d\lambda}{d(\delta n_{\text{eff}})} \left(\frac{dn_{\text{eff}}}{d\epsilon} - \frac{dn_{\text{cl}}}{d\epsilon} \right) + \Lambda \frac{d\lambda}{d\Lambda} \quad \text{Equation 3.2}$$

The sensitivity to strain can be described as comprising of material and waveguide contributions which can be manipulated to yield strain insensitivity or enhanced sensitivity. Appropriate choice of the LPG's periodicity, the composition and the geometry of the fibre allow LPGs to exhibit either positive, negative or zero sensitivity to strain²⁰ and offers a convenient method for isolating the response of the LPG to one or other of the measurands. LPGs can also be fabricated to exhibit a change in coupling strength as a result of strain, whilst remaining at fixed wavelengths, especially appropriate for wavelength filtering application²⁸.

The material effect, first term on the right hand side, arises from the change in transverse dimensions of the fibre due to Poisson's effect and the strain-optic effect which results in a change in refractive index²⁰. The waveguide effect, the second term, can be significant and is a function of the slope of the dispersion term, $d\lambda/d\Lambda$, for a particular cladding mode²⁰. The two contributions to the total strain induced wavelength shift can be either positive or negative depending on the period of the LPG and the order of the cladding mode to which coupling occurs. For LPGs with a periodicity $> 100\mu\text{m}$, the material contribution is negative, whilst the waveguide contribution is positive.

An LPG of period $340\mu\text{m}$, written in Corning Flexcore fibre exhibited an attenuation band with a strain sensitivity of $0.04\text{pm}/\mu\epsilon$ ²⁰ which is an order of magnitude less than that of a FBG. This reduced sensitivity can be understood as the negation of the two opposing contributions acting on the central wavelength of the attenuation band. Strain sensitivities as large as $-2.2\text{pm}/\mu\epsilon$ with a good degree of resolution have been demonstrated using an LPG with a period of $40\mu\text{m}$ since in this region, $\Lambda < 100\mu\text{m}$, both contributions to the strain sensitivity are negative and the effects are compounded²⁹. A typical response to strain is shown in Figure 3.3, where the wavelength shifts in the centre wavelength of several attenuation bands are plotted as a function of strain. Each attenuation band exhibits a linear response to applied strain.

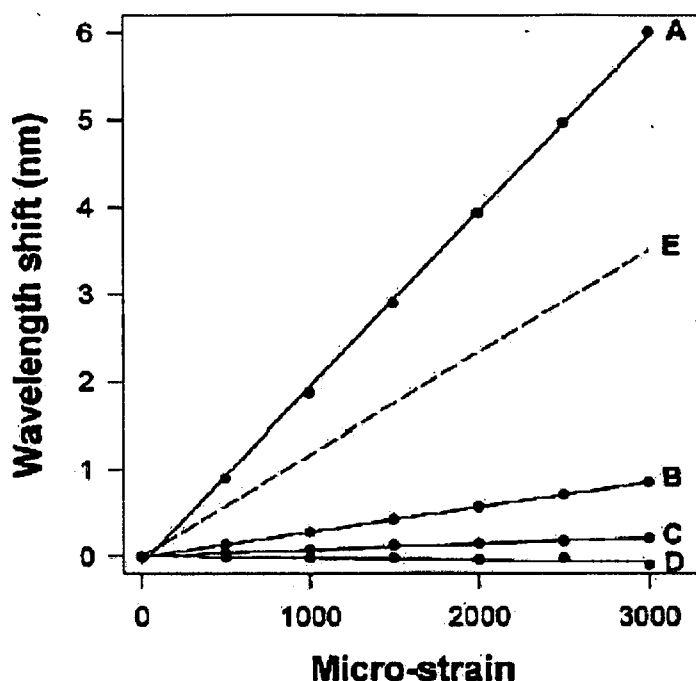


Figure 3.3: Shift in the wavelengths of four attenuation bands of an LPG of period $280\mu\text{m}$, written in Corning SMF 28 fibre, A-D as a function of strain. The dashed line, E, represents a strain induced response for a FBG fabricated at 1550nm for comparison. Diagram reproduced from⁹.

The effect of strain has been shown to decrease the coupling strength of the LPG and hence to change the minimum transmission of the LPG attenuation bands, as shown in Figure 3.4. This provides an effective method for sensing applications as it eliminates the requirement of spectrometers and time consuming spectral processing. A quadratic-dispersion LPG sensor ($d\lambda_{\text{res}} / d\Lambda = 0$) written in a QPS-1355T fibre with a period of $50.1\mu\text{m}$ and length 1cm has been shown to reduce linearly the efficiency of coupling to an attenuation band from over 80% with no strain to 0% for an applied strain of $9410\mu\epsilon$. Measurements were made with a resolution of $1\mu\epsilon$, which equals or exceeds that of considerably more complex sensor systems based on Bragg gratings. The dynamic range was greater than $5000\mu\epsilon$, comparable to most other conventional sensor systems. The resolution can be improved further by use of an amplitude-stabilized light source, although an inexpensive LED would be suited for the majority of applications²⁸.

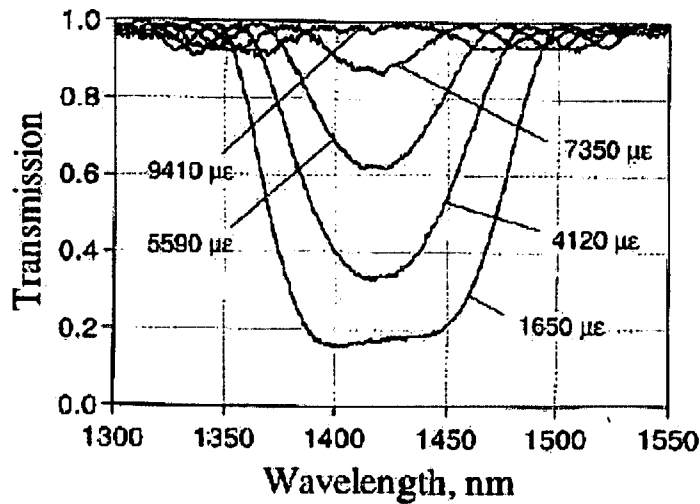


Figure 3.4: Change of the transmission spectrum of a quadratic-dispersion LPG written in QPS-PFBG-1355-T fibre from QPS Tech under strain ranging from 1650 to 9410 $\mu\epsilon$. The strength of the grating decreases whilst the resonant wavelength remains at 1420nm. Diagram reproduced from²⁸.

Strain insensitive attenuation bands have been demonstrated using special fibres and by careful selection of the period of the LPG, $\Lambda > 100\mu\text{m}$, to counteract the material and waveguide contributions³⁰. An important limitation to LPG strain sensors is the cross-sensitivity to temperature which can usually be overcome by selection of parameters, such that the sensitivity to strain is enhanced, $\Lambda < 100\mu\text{m}$, to allow differentiation^{30, 31}. The dependence of the sensitivity of the LPG to strain and temperature upon the order of cladding mode may be used to allow decoupling of the temperature and strain responses. This allows the simultaneous measurement of both parameters without issues related to cross-sensitivity³².

In addition to the measurement of axial strain, the sensitivity of LPGs to transverse loads has been investigated^{33, 34}. LPGs were fabricated in non-high birefringence fibre and a transverse load was introduced. The induced birefringence resulted in each attenuation band dividing into two smaller attenuation bands, corresponding to coupling to the two orthogonal polarisation states, as shown in Figure 3.5.

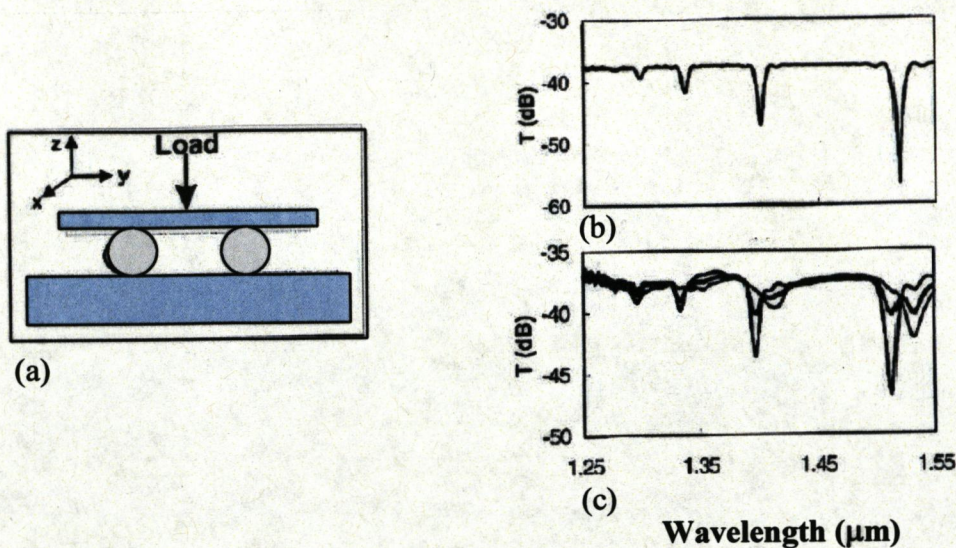


Figure 3.5: (a), Loading system for an LPG transverse load sensor used for an LPG of period $490\mu\text{m}$ written in B-Ge co-doped fibre. The transmission spectrum is shown under (b) unloaded and (c) loaded conditions, the attenuation bands can be observed to split as a result of the induced birefringence. Diagram reproduced from³⁴.

The magnitude of the birefringence, and thus separation of the sub-bands, increased with the load. A linear response was obtained, with a sensitivity of 500nm/kg/mm , 800 times larger than the response of a FBG³⁴. The generation of two narrow resonances rather than the single broad spectral resonance provided improvement to the spectral response and thus sensitivity to transverse strain. These types of devices where the spectral response is narrowed intentionally will be discussed further in Section 3.6, where concatenated LPGs will be reviewed.

Corrugated LPGs have been used to form axial strain and torsion sensors³¹, created by mechanical forces such that a periodic strain distribution is induced when an external mechanical force is applied. Due to the photoelastic effect, any applied strain causes a periodic modulation of the refractive index and thus increases the coupling strength of the LPG. A corrugated fibre experiencing torsion is exposed to different twisting rates in the etched and unetched regions because of the difference in diameter. This results in a stress concentration at the etched boundary. The effect of torsion is to change the coupling strength and to induce a wavelength shift³⁵.

This section has reviewed the response of LPGs to axial strain, transverse loads and torsion sensors. It has highlighted that the sensitivity to strain can be enhanced or suppressed by selection of parameters to obtain the sensor most suitable for the application. The cross-sensitivity to temperature has been presented as a long standing issue with fibre optic sensing schemes and the opportunity to overcome these were also discussed.

3.4 Sensitivity to Bending

An LPG is highly sensitive to bending and an overview is presented here. The sensitivity to bending results from a modification of physical parameters of the LPG. The expansion of the outer edge of the LPG and a compression of the inner edge allows the LPG to be considered as a non-uniform tilted grating³⁶. Structural bend sensing has applications in a number of engineering areas, such as monitoring the bending of rotating helicopter rotor blades, in observing the bending of reinforcement girders in structures and in robotic manipulation⁷.

The sensitivity of an LPG to bending is apparent as either a shift in the central wavelength of the attenuation bands^{37, 38}, or as a subdivision into two of each attenuation band. During the second manifestation, the wavelength separation of the two components increases with increasing curvature^{39, 40}.

The first observation of the influence of bending upon the transmission spectrum of a LPG noted that the shift in the central wavelength of the attenuation bands is accompanied by a reduction in the coupling strength and a broadening of their spectral width³⁷. The magnitude of the bend induced wavelength shift was observed to depend upon the orientation of the optical fibre with respect to the plane in which the bend is applied, with the maximum and minimum sensitivities separated by 180°³⁸. This rotational dependency of the response gives the added advantage to this type of sensor to isolate the bends in different planes of a structure. The orientation dependency is believed to be due to an eccentricity of the fibre core, with the core no longer at the neutral axis of the fibre. Fibres with eccentricities of up to 14µm have

been used to form bipolar bend sensors which is a highly desirable effect as it allows one LPG to be used where two would be required for LPGs written in conventional fibres with a concentric-core^{41,6}.

The second manifestation is attributed to a breaking of the symmetry of the system that results in two degenerate spatial modes of the fibre becoming non-degenerate. This effect has been observed in B-Ge co-doped photosensitive fibre^{39,40} and in fibres with large core eccentricities^{42, 43}. It also has a dependence upon the orientation of the fibre^{41,42}. In this case the reference plane is defined by the orientation of the fibre during fabrication of the LPG. The largest sensitivity occurs when the surface of the fibre oriented towards the UV source during fabrication, is positioned on the outer surface of the bend^{40,6}. This is thought to be a result of an asymmetry introduced by a radially non-symmetric UV induced RI modulation. Such asymmetries have been used to account for similar effects in LPGs formed by CO₂ laser irradiation, where the asymmetry is more pronounced due to heating of the surface facing the laser beam. LPGs formed using this method should provide greater sensitivity to bending. Tuning of a resonance peak of over 30nm was observed as the axial rotational orientation of the fibre was changed from 0° to 180° in such a fibre⁴⁴.

As discussed in Section 2.6.1, higher order cladding modes can couple to the same cladding mode at two different wavelengths, which results in the formation of two attenuation bands. The wavelength separation between the two bands shows high sensitivity to external measurands, and this has also been exploited to perform bend sensing⁴⁵. The separation length between the split attenuation bands increases with increasing bend curvature. A bend sensitivity of 14nm/m⁻¹ was observed, corresponding to separations as large as 70nm for an applied curvature of 5m⁻¹, have been reported⁴⁶. This represents sensitivities of up to fifty times that offered by the previously reported LPG bend sensors^{47, 48} and nearly four times higher than previously reported wavelength shift detection method³⁹.

The sensitivity to bending in an LPG was discussed and several methods to demonstrate bend sensors were reviewed. The forms of the manifestation of the sensitivity to curvature were discussed and were explained.

3.5. Sensitivity to surrounding refractive index (SRI)

The sensitivity of an LPG to the RI of the material immediately surrounding it is an important effect and arises from the dependence of the phase matching condition upon the effective refractive indices of the cladding modes. These effective indices are dependent on the difference between the refractive index of the cladding and the medium surrounding the cladding, thus the central wavelength of attenuation bands is influenced by the material surrounding the LPG. This phenomenon only occurs where the refractive index of the cladding is higher than the surrounding medium. A review of the sensitivity of an LPG to SRI is given and the effect of overlays is discussed.

The sensitivity to SRI is manifested as a shift in the central wavelengths and also as a change in the minimum transmission of the attenuation bands, as shown in Figure 3.6 and Figure 3.7 respectively. The attenuation bands display varying degrees of sensitivity, with the higher order cladding modes exhibiting the greatest sensitivity as discussed in Chapter Two. Figure 3.6 shows how the sensitivity of one attenuation band changes with external refractive index and shows that the highest sensitivity is achieved at refractive indices approaching that of the cladding⁴⁹. The response is a function of the LPG period and cladding mode order to which coupling occurs, with certain combinations yielding wavelength shifts as high as 100nm²¹.

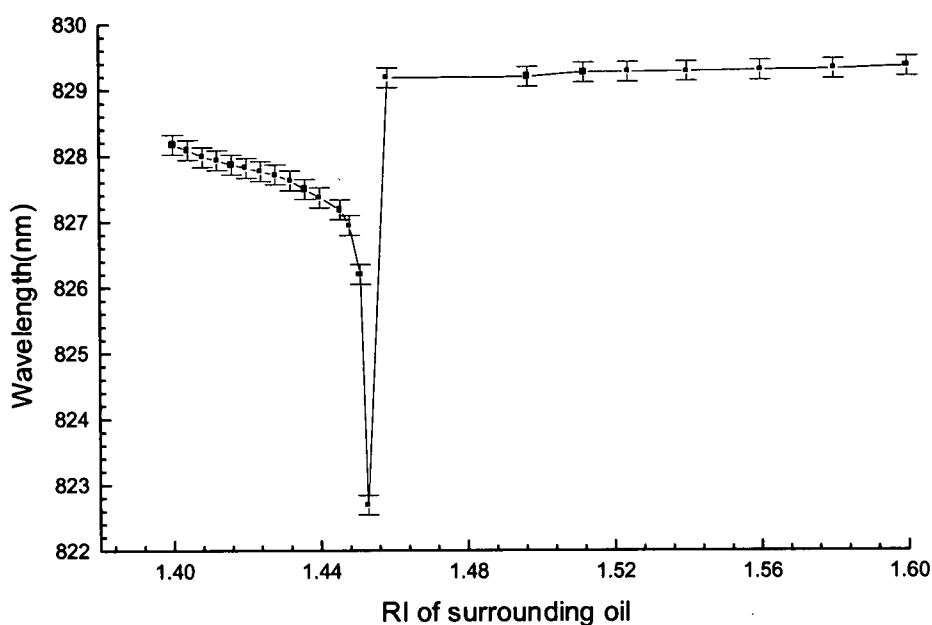


Figure 3.6: Plot of the central wavelength of an attenuation band as a function of the RI of the medium surrounding the LPG of period $400\mu\text{m}$ written in Boron-Germanium co-doped fibre with cut off wavelength of 650nm ; line is a guide for the eye only. Diagram reproduced from ¹⁶.

Figure 3.8 shows the response of the attenuation bands in the transmission spectrum to immersion in two liquids of different RI. It can be seen that the response is greatest when the LPG was immersed in an SRI approaching that of the silica cladding.

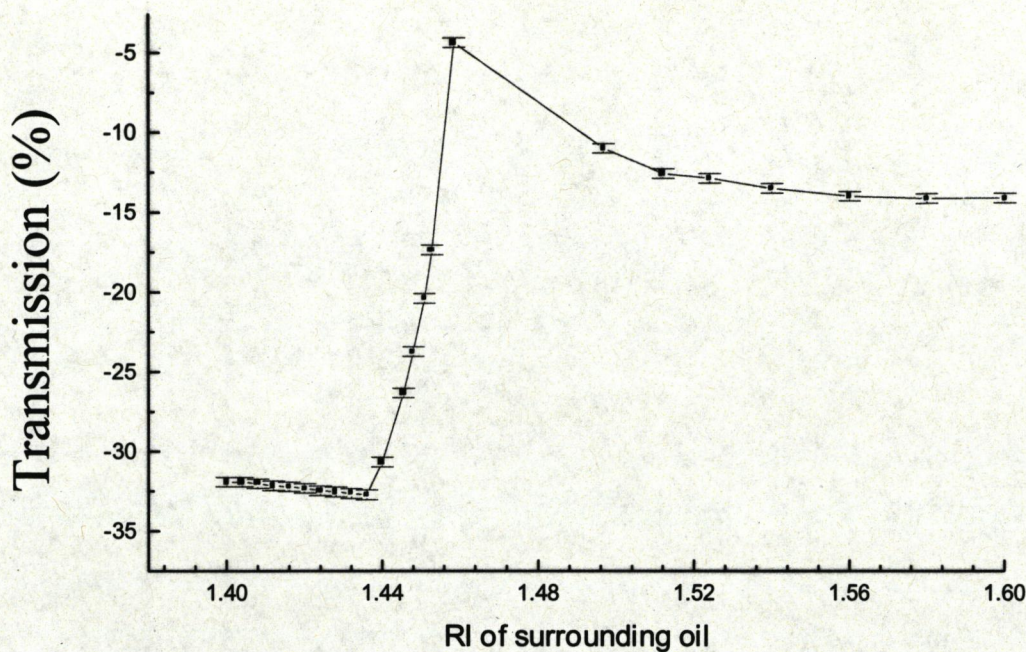


Figure 3.7: A plot of the minimum transmission value of the central wavelength of an attenuation band as a function of RI, the line is a guide for the eye only. Diagram reproduced from¹⁶.

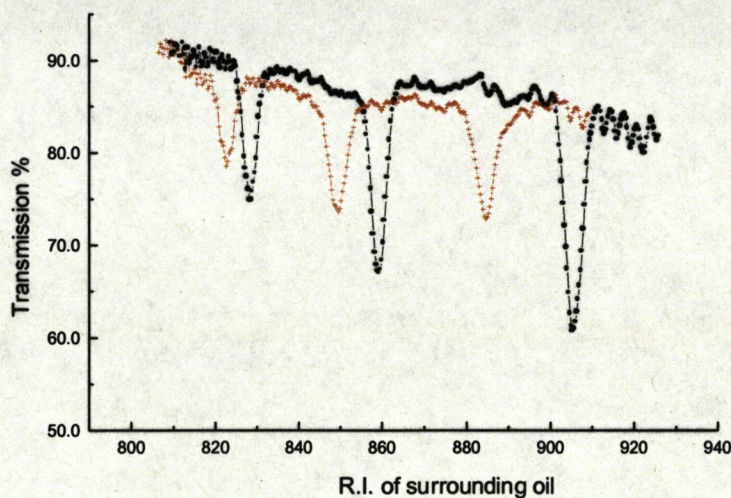


Figure 3.8: Plots of transmission as a function of wavelength for the LPG when it was immersed in liquids of RI, ● 1.400 and + 1.456. Diagram reproduced from⁵⁰

The sensitivity of the LPG is zero when the refractive index of the surrounding medium is equal to that of the cladding since the cladding behaves as if of infinite extent and thus no discrete modes can be supported. Broadband radiation-mode

coupling losses are then observed, with no distinct attenuation bands. For surrounding RI greater than that of the cladding, the centre wavelengths show a reduced sensitivity^{49, 51, 52}. Normally attenuation bands would be unsustainable in these conditions when the cladding is no longer acting as a waveguide. Their existence have been attributed to the existence of attenuated cladding modes arising from Fresnel reflections rather than the total internal reflection from the cladding and air interface⁴⁹.

The refractive index sensitivity offers many potential applications but also raise a number of practical concerns, which need to be overcome where well-controlled spectral characteristics are required. Packaging and protection of the LPG raises practical issues such as the effect of the properties of an adhesive used to attach the LPG to a substrate; or that the RI of any other materials used to protect it, does not interfere with the position of the central wavelength of the bands.

Where the SRI sensitivity is not required and needs to be suppressed, it is possible to fabricate the LPG in fibres with complex structures such as dual shaped core (DSC) dispersion-shifted fibre. This fibre contains an inner cladding region that has a higher refractive index than the remaining cladding layer and coupling to a mode in this cladding produces a refractive-index-insensitive attenuation band. This attenuation band is also insensitive to mechanical damage of the fibre surface⁵².

LPGs have been fabricated in an air-clad optical fibre, which prevents the cladding modes from coming in contact with the refractive index surrounding it. The cladding of the fibre is surrounded by a capillary tube, supported by thin silica webs⁵³. The capillary restricts the evanescent field of the cladding modes to extend beyond it and thus results in all the attenuation bands being insensitive to the external refractive index. This yields a key advantage in that it allows the fibre to be recoated to provide additional protection to the fibre and thus reduces the need for costly hermetic packaging. This should improve the design and flexibility of LPG gain flattening filters.

A similar structure has been proposed to form a tuneable filter, without the need for electrodes⁵⁴. The air between the capillary and the inner cladding is filled with a liquid crystal material. The refractive index of a dye-doped nematic liquid crystal was

controlled by realignment of the molecules using polarised laser illumination. Similar systems could be controlled using electrical or thermal techniques.

The refractive index sensitivity exhibited by the central wavelengths of the attenuation bands has many niche applications. It has been exploited to form refractive index sensors, a liquid level sensor⁵⁵, and chemical concentration sensors^{56,57,58}. Although LPGs have a response limited by the refractive index of the cladding and their response is not specific to the composition of liquid being monitored, it has still been shown to be of use in on-line monitoring of the concentration of aqueous solutions of materials. This application is especially useful in monitoring materials in inaccessible locations such as for industrial quality control⁵⁸. Concentrations of solutions of sodium chloride⁵⁸, calcium chloride⁵⁸ and ethylene glycol^{49,58} have been measured, with sensitivities equal to, or better than, that of conventional Abbe refractometry.

The feasibility of the use of LPGs to monitor the refining of kerosene was demonstrated and gave results comparable in accuracy to conventional liquid chromatography and UV spectroscopy techniques. This involved measuring the concentrations of organic aromatic compounds such as benzene and xylene. A binary solution of xylene and heptane was measured with a minimum detectable volumetric concentration of 0.04%, corresponding to a refractive index change of 6×10^{-5} ⁵⁷.

The majority of research into refractive index sensitivity has focused on the bulk immersion of LPGs into a solution. Some studies have looked at depositing overlay materials that exhibit changes in their refractive index in response to their local environment^{59,60}. These have attractive applications and have been used to form, a tuneable loss filter⁵⁹, a temperature-insensitive filter²² or a species specific chemical sensor^{60,6}.

LPG based biosensors have been investigated by monitoring the change in refractive index that occurs upon bond formation of an antibody with the antigen⁶⁰. This is the first reported example of the use of an LPG to form a species specific chemical sensor and offers comparable performance to other techniques including plasmon resonance

and interferometry but with the advantage of on-line monitoring⁶. Figure 3.9 shows an example of a reflection based biosensor as an example⁶¹.

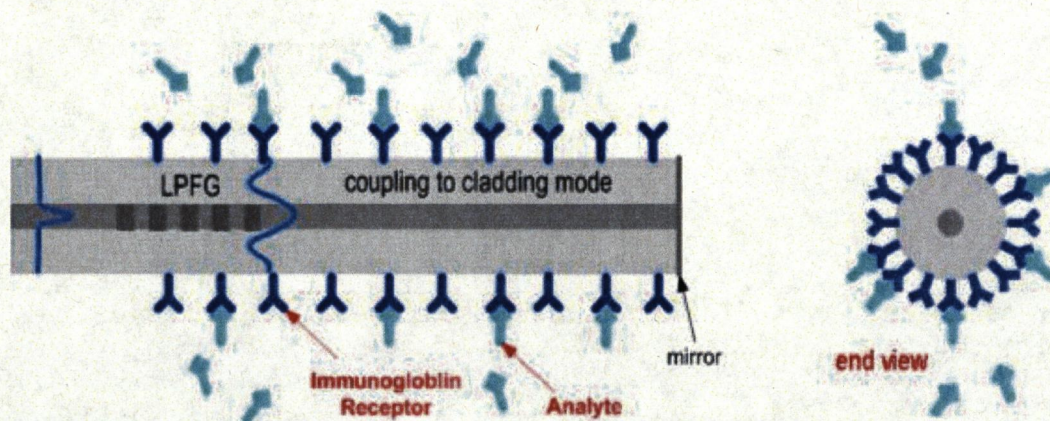


Figure 3.9: A schematic showing a reflection based LPG Biosensor .Diagram reproduced from ⁶¹.

The optical properties of an overlay deposited upon an LPG influence the response of the central wavelengths within an LPG. The response of an LPG as a function of increasing overlay thickness has also been investigated⁶². A thin amphiphilic film of organic material was deposited upon an LPG using the Langmuir-Blodgett (LB) technique, detailed in Chapter Five of this thesis. The overlay was deposited a single molecular layer at a time, with a corresponding thickness resolution of the order of approximately 3nm, on the optical fibre containing the LPG. It was shown that for an overlay with an RI of 1.57, higher than that of the silica cladding, when the overlay thickness reached a few hundred nanometres, the wavelength and amplitudes of the attenuation bands exhibited a very high sensitivity. This is different from the behaviour under bulk immersion, in which the highest sensitivity is observed for indices lower than that of the cladding, as shown in Figure 3.7 and is an important observation investigated further in this research. An example of this response is shown in Figure 3.10.

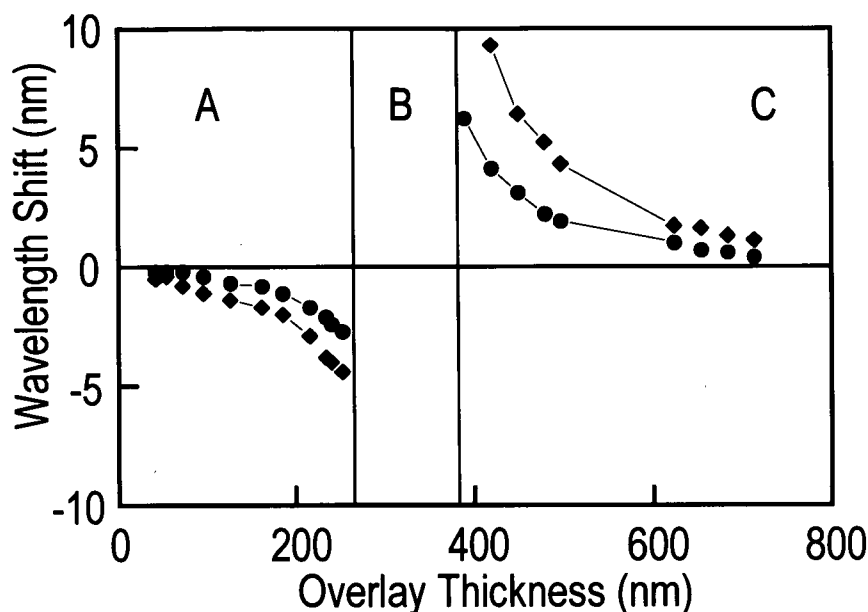


Figure 3.10: Response of the (●) 5th and (■) 6th order attenuation bands of an LPG to the deposition of thin film (ω -tricosenoic acid) of refractive index 1.57. Diagram reproduced from⁶².

The response shown in Figure 3.10 may be understood qualitatively as follows. As the film thickness is increased (region labelled A), both the LB film and the air influence the cladding mode. This increases the average external refractive index and causes a negative wavelength shift in the transmission spectrum, as expected from the response seen due to bulk immersion in refractive index oils, shown in Figure 3.6. As the film thickness is increased further (region labelled B), the average external refractive index is approximately equal to that of the cladding and so no cladding modes are supported. This is apparent in the disappearance of the attenuation bands in the transmission spectrum. Further increase to the film thickness (region labelled C) causes the average external refractive index to be higher than that of the cladding; however cladding modes are still supported. A more complex waveguide structure exists in this region and has been previously explained by leaky modes that exist in such an inverted waveguide⁶². This will be discussed in greater detail in Section 6.2.4 of Chapter Six.

Materials suitable for deposition by the LB technique have also been shown to be chemically sensitive, thus an opportunity to form species-specific chemical sensors exists^{63, 64}.

3.5.1 Refractive index based Physical sensors

The refractive index sensitivity of LPGs has been used to form other types of physical sensors. A flow sensor to monitor the arrival of resin within a liquid composite moulding system has been demonstrated^{65, 66}. A reduction of the extinction ratio of the attenuation band due to the higher refractive index material surrounding the LPG was observed during the flow. An LPG-based liquid level sensor has been reported based on the dependency of the depth of the attenuation bands upon the LPGs length⁵⁵. Partial immersion of an LPG held vertically in a liquid causes concurrent splitting of each attenuation band into two. The two central wavelengths correspond to coupling to the cladding surrounded by air and due to coupling of the same cladding mode but with its effective refractive index modified by the liquid. The relative depth of the attenuation band changes with changing levels of the liquid as the minimum transmission is governed by the expression given in Equation 2.4.

Figure 3.11 shows that the sensor has a linear response over 60% of the LPGs length and for an LPG of length 40mm, this corresponds to a sensitivity of 4.8% change in transmission per millimetre. This system can be configured to be independent of temperature by utilising a reference LPG fully immersed in the same liquid⁵⁰.

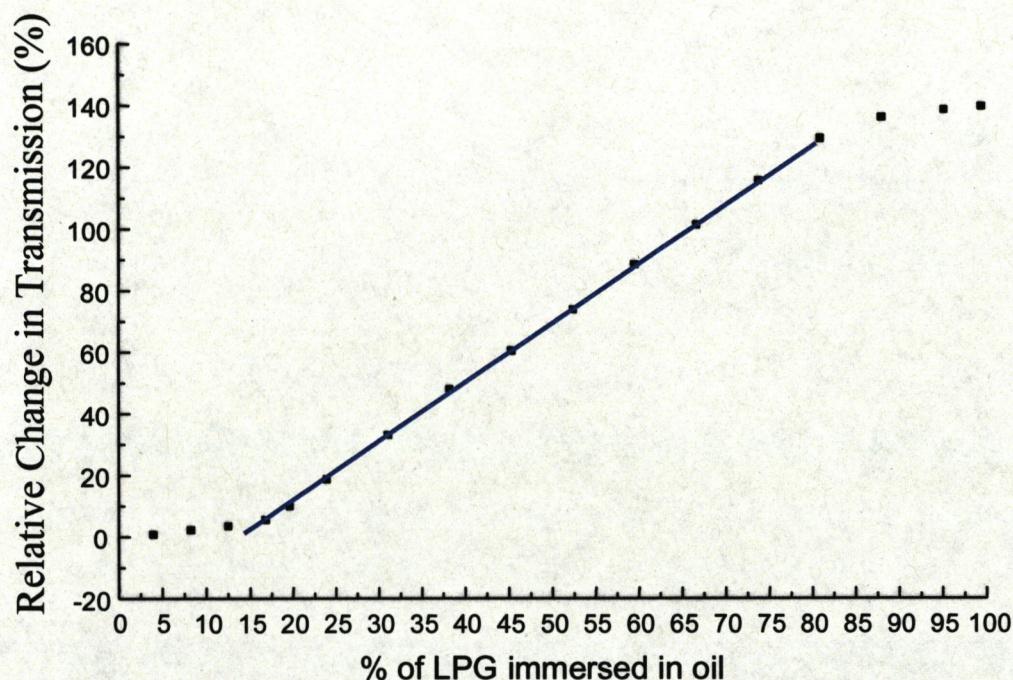


Figure 3.11: Relative change in the extinction of the attenuation bands of the liquid-level sensors, showing a linear response over 60% of the LPGs length. Diagram reproduced from⁵⁰.

The refractive index sensitivity has also been exploited to reduce or enhance the temperature sensitivity of the LPGs. A liquid with a large thermo-optic coefficient has been used to surround an LPG such that any changes in temperature cause a twofold response. One response is due to the change in temperature upon the LPG and the other is due to the temperature induced change in refractive index of the surrounding medium^{16,17,18}. Careful selection of the medium can provide the desired effect of either enhanced or decreased temperature sensitivity. For instance if a material is chosen with a negative thermal expansion coefficient, then for increasing temperature, the change in refractive index of the material would cause a red shift in the central wavelength of the attenuation band. If the attenuation band already displays positive temperature sensitivity, it is apparent that the two effects would add to give an increased temperature response. Conversely, if the material chosen has a positive

thermo-optic coefficient, then a reduced response to temperature would be observed for increasing temperature⁶.

Enhanced temperature sensitivity has been observed by recoating the LPG with a UV-curable acrylate-based polymer of refractive index approximately equal to that of the cladding. This polymer had a negative thermo-optic coefficient and increased the sensitivity of a dispersion-shifted fibre from 5 nm/100°C to 80 nm/100°C¹⁶; however this was accompanied by a reduced coupling strength and possible degradation of performance due to exposure of the polymer to the environment. An air-clad optical fibre in which the air regions were filled by the same polymer, was used to demonstrate a sensor with comparable sensitivity but with no change in the transmission spectrum of the attenuation bands¹⁸. This also had an added advantage of protecting the polymer from humidity and other environmental effects. A similar system was demonstrated where instead of coating the LPG with a polymer, a liquid crystal was used. This produced a large wavelength shift of 46nm over a 22°C change and corresponds to sensitivity of 210 nm/100°C⁵⁴. Ultra high sensitivity of up to 1900nm/100°C over a limited range of 1°C has been observed by immersing an LPG in a Cargille oil of refractive index approximately equal to that of the cladding. Careful selection of oils whose thermo-optic coefficient cause the refractive index to be approximately equal to the cladding, at the desired operating temperature, would produce highly sensitive sensors¹⁶.

The refractive index sensitivity of an LPG was utilised to reduce the temperature sensitivity. An LPG was coated with a material of positive thermo-optic coefficient. This gave a temperature response as low as 0.07 nm/100°C²² due to a blue shift of the attenuation band caused by the thermo-optic properties of the coating.

3.6 Concatenated LPGs

A concatenated LPG consists of multiple LPGs written in series in the core of a fibre. This results in the formation of interference fringes in the transmission spectrum, which provide the potential for some novel sensor applications. When a pair of identical LPGs are fabricated in such a way, separated by up to 10 cm⁶, a set of fringes are observed within the attenuation bands as seen in Figure 3.12. The effect of the two gratings produces an in-fibre Mach-Zehnder interferometer.

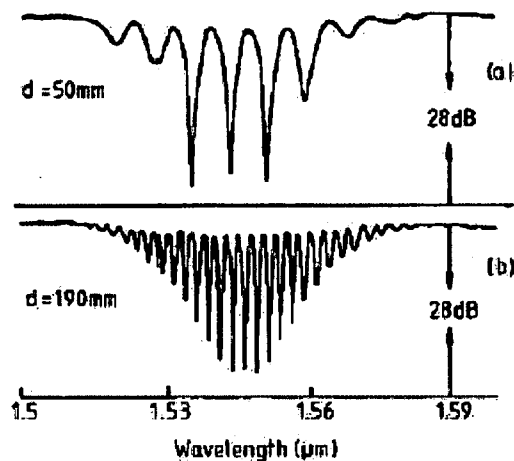


Figure 3.12: The effect on the transmission spectrum of an LPG by introducing a second LPG within the same fibre (a) separated by 50mm spacing and (b) separated by a 190mm spacing. Diagram reproduced from³⁴.

The formation of the fringes may be understood with reference to Figure 3.13, which shows a schematic of the principles of operation of in-series LPGs. The first LPG shown on the left in Figure 3.13 couples light into the cladding mode. Light propagates via two routes to the second LPG: from the core and from the cladding. The difference in the refractive indices of the core and the cladding modes results in the light, which is coupled back into the core by the second LPG, to be phase shifted with respect to the light propagating in the core. This gives rise to the interference pattern observed and results in narrower bandwidth peaks, offering greater resolution in the measurement of wavelength than conventional LPGs⁶.

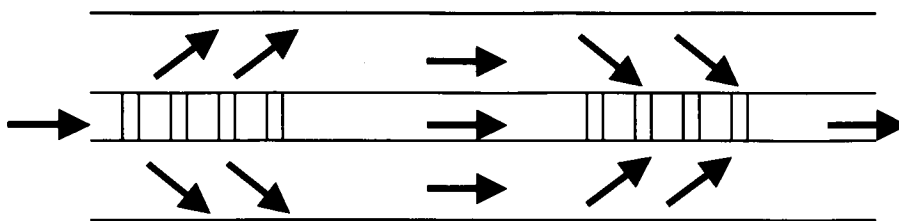


Figure 3.13: Schematic of light propagating through cascaded LPGs.

The channel spacing between the fringe patterns depends on the separation between the two gratings. The number of gratings also affects the shape of the fringe pattern and in order to obtain a clean fringe pattern the LPGs characteristics need to be identical as possible⁶⁷.

In-series LPGs have been used to sense bending⁶⁸, external index of refraction^{69,70}, temperature and transverse load⁷⁰. The bending sensitivity has been investigated in a LPG pair whose separation was wider than the grating length and exhibited an increase in the attenuation of the cladding modes, with a concomitant reduction in the fringe visibility. This was also accompanied by a shift in the central wavelength of the band⁶⁸. The grating pair was written in a hydrogen-loaded dual-shaped core-type fibre by a KrF excimer laser through an amplitude mask with a 400 μ m period and a 20mm length. Spectral analysis showed no appreciable change in any of the attenuation bands except the first, which exhibited interference fringes when no bending was present. As the bending was increased the interference fringes gradually degraded until they were no longer visible as shown in Figure 3.14.

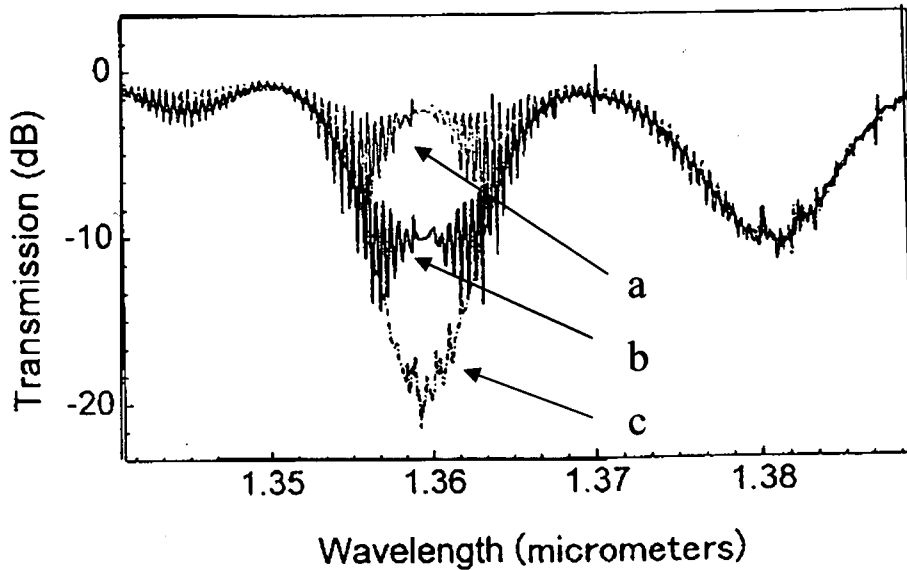


Figure 3.14: Plot of transmission spectrum of an LPG pair with a 50cm separation: (a) without bending; (b) with minimal bending; (c) with increased bending, measured with a 0.1nm resolution. Diagram reproduced from⁶⁸.

This device may be used as a sensor for bending measurements and as a removable multichannel narrow-band filter.

Transverse loading was observed to reduce the fringe visibility linearly until the fringe pattern disappeared⁷⁰. Further increase of the load causes the pattern to reappear with a π phase shift. The pair of LPGs were fabricated with a length of 2cm with a separation distance of 10cm, in an optical fibre adjusted to suppress the sensitivity to temperature to $0.002\text{nm } ^\circ\text{C}^{-1}$. Although a reduction in the depth of central peak was observed in the transmission spectra due to transverse load, a wavelength shift was not seen. This was because the applied load changes the mode field profiles, causing the variation in depth of the peak, but does not change the grating period significantly to cause a shift in the central wavelength.

The response of in-series LPGs to refractive index is exhibited as a decrease in fringe visibility and a change in the central wavelengths, similar to that demonstrated by single LPGs. This was investigated by bulk immersion of the separation length of the two LPGs in index matching oil; Figure 3.15 shows the response observed. The central wavelengths of the fringes were observed to decrease in visibility whilst the

inset shows that a shift in the wavelength, similar to that of a single LPG, was demonstrated⁶⁸.

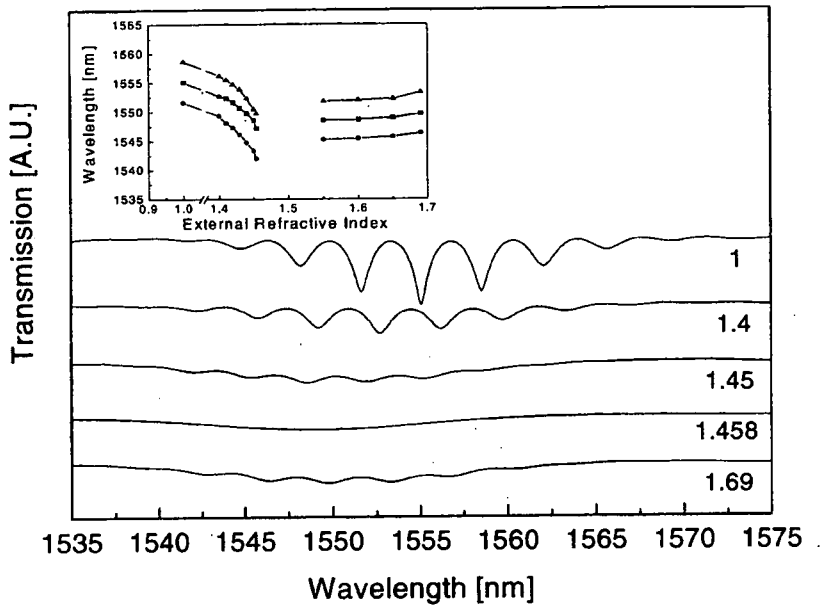


Figure 3.15: Changes in the transmission characteristics of a pair of LPGs due to change in the surrounding refractive index. The shift in one of the resonance fringe (inset) with change to external refractive index is shown. Diagram reproduced from⁷⁰.

Further investigation into refractive index sensitivity has demonstrated attenuated couplings in an LPG surrounded by a medium of index higher than silica in the central section between the fibre, the minimum transmission of the attenuation bands being twice as sensitive as a conventional LPG for refractive indices 1.5-1.55⁶⁹.

Coating the end of a fibre containing a single LPG with a metal film results in the double pass of the light through the LPG, allowing the generation of interference fringes associated with in-series LPGs⁷¹. This method also enables the monitoring of the LPG in reflection and overcomes the requirement to fabricate identical LPGs. This method has been used to form a temperature sensor⁷¹.

Other uses for concatenated LPGs include a novel all-fibre wavelength-division multiplexing (WDM) channel isolation filter which can easily be adjusted⁷². A series of notch filters have also been formed with three concatenated LPGs in series. The

free spectral range was measured to be 3.6nm with a finesse of 5. The shape of the filter can be tuned by changing the distance between gratings and by altering the total number of cascaded gratings⁷³.

3.7 Multi-parameter sensing using LPGs

Manipulation of the properties discussed earlier in this chapter can generate attenuation bands in LPGs which can be used for simultaneously sensing multiple measurands independently within the same fibre. Differential shifts in two or more resonance bands were used to measure the temperature and strain due to the difference in their sensitivities to the measurands³². Bend induced splitting of the attenuation bands has been utilised for the simultaneous measurement of bend radius and temperature. The wavelength separation of the split components of the attenuation band gave a measurement of the bend radius and the average wavelength was dependant upon temperature⁴⁰. LPGs have been used in conjunction with FBGs to separate their temperature and strain responses by virtue of their different sensitivities to these measurands⁷⁴. An LPG was also used as an edge filter to facilitate intensity based method to demodulate the reflection from an FBG⁷⁵.

3.8 Chapter Summary

The sensitivity of LPGs to strain, temperature, bending and external refractive index has been discussed; methods to influence the sensitivity by selection of parameters such as fibre composition and LPG period have been presented. Some applications have been outlined and compared to conventional sensing methods. The opportunity to develop sensors capable of simultaneously and independently monitoring a number of measurands has been discussed. This is due to different sensitivity exhibited by the different attenuation bands with the transmission spectrum.

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Chapter 4 Fabrication of fibre optic long period gratings

4.1 Introduction

Several methods have been used to fabricate LPGs. The different methods and processes are presented here and their respective advantages and disadvantages are discussed. The chapter also presents in detail the UV based amplitude mask fabrication method used in manufacturing the LPGs used for all work presented in this thesis. Details of how this method was modified to fabricate concatenated LPGs are also given here.

4.2 Induction of periodic modulation in optical fibre

4.2.1 Introduction

The fabrication of an LPG requires the production of a periodic modulation of the optical properties of the fibre. There are several methods available, and these have been categorised into two types in the discussion presented here. Some rely on introducing a physical deformity^{1, 2, 3, 4, 5, 6} and others depend on irradiation to modulate the refractive index of the core of the fibre. Irradiation of the fibre by femtosecond laser pulses in the infrared⁷ or by the focused output from a CO₂ laser^{8, 9, 10} have been used. A novel technique to electrically induce a periodic change in the refractive index of a hollow-core fibre filled with liquid crystal has been reported¹¹. The permanent modulation of the core RI has also been achieved by implantation of ions into the fibre core. The interaction of the ions with the atoms in the fibre produces changes in the structure and hence refractive index of the silica¹². Spatial redistribution of the chemical composition of the glass due to thermo-induced diffusion of the fibre's dopants has also been employed^{13, 14}, as well as the relaxation of the mechanical stress within the fibre^{15, 16}. Another method to fabricate LPGs has been the use of Electric discharges in fibre doped

with germanium^{17, 18, 19} and using air-silica microstructure fibre, which does not require the need for doping with germanium²⁰. Exposure of the fibre to ultra-violet (UV) light is perhaps the most widely used technique to create the periodic index modulation^{21, 22, 23, 24, 25, 26, 27}. Section 4.2.2 describes how LPGs can be fabricated by physically deforming the fibre and Section 4.2.3 describes techniques for LPG fabrication through the modulation of the fibre core's refractive index.

4.2.2 Physical deformation of the fibre

The techniques discussed in this section facilitate the induction of a periodic index modulation of the optical fibre via the transformation of the physical properties of the fibre. This is achieved by deforming either the core or cladding of the fibre or applying pressure points to achieve the loss bands of the LPGs..

A method has been used to mechanically induce the periodic index modulation via the photoelastic effect. A fibre is held and pressed between a periodically grooved plate and a flat plate. While the pressure is applied the transmission spectrum can be observed to contain attenuation bands. On complete removal of this pressure, the transmission spectrum returns to its original pre-pressure state. Attenuation bands with amplitudes upto 15dB have been achieved, which is comparable with photo induced LPGs. The position of the attenuation bands can be controlled by adjusting the angle between the fibre and the grooves. The extinction of the attenuation bands can be controlled by adjusting the pressure on the grooved plate. This method is therefore suitable for producing temporary LPGs.

Recently a new method of fabrication of lpgs has been shown which introduces periodic modulation of the core by twisting a standard single-mode fibre at high temperature²⁸. Due to core-cladding eccentricity which always exists in the optical fibre, when the fibre is twisted its core follows a helicoidal path inside the cladding. The transmission spectrum of the helicoidal long-period fibre grating that is produced contains several loss peaks that correspond to resonances with the fibre cladding modes.

An LPG has been produced by periodically tapering the fibre along its length. The fibre was pre-prepared by narrowing the section of fibre where the modulation was to be introduced. The fibre was narrowed by heating a length of fibre in a moving flame and then stretched to obtain a uniform fibre diameter of approximately $15\mu\text{m}$ [29]. A 0.8mm section of fibre is then further heated and elongated very slowly to narrow the fibre further in this section. This is repeated at equally spaced intervals along the narrowed fibre, where the intervals correspond to the period of the LPG formed. Attenuation bands of up to 17db have been achieved after 35 regularly spaced periods were formed. An LPG produced by this method is considerably more stable even at high temperatures because the perturbation is due to its macroscopic structure, unlike those induced by UV irradiation.

LPGs have also been made by deforming the core of the fibre. The removal of the buffer jacket is required prior to exposure to a focused single pulse of a CO_2 laser beam. The fibre is held firmly between two fixed points on a translation stage and then periodic incisions are made into its cladding by exposure, as shown in Figure 4.1. After each incision the fibre is translated along by a distance equal to the grating period and the process is repeated until the required numbers of incisions are obtained. The width and depth of the incision depends on the size of the beam and the diameter of the cladding respectively, which can be monitored ⁵.

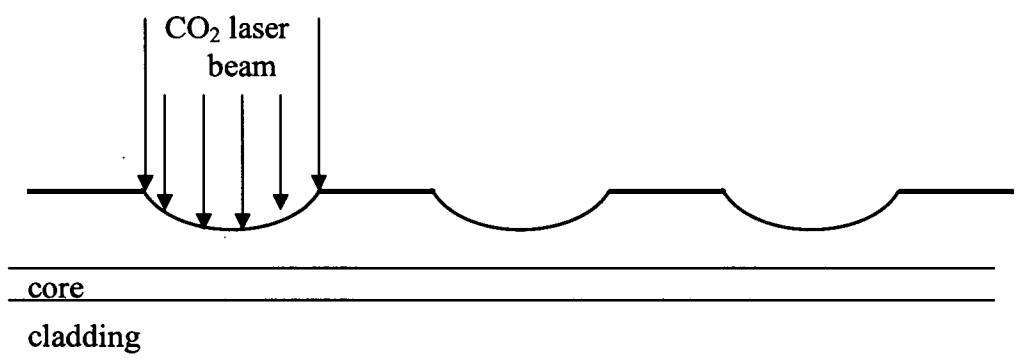


Figure 4.1: A schematic to show the formation of periodic incisions in the surface of the cladding using a focused CO_2 laser beam. Diagram reproduced from ³.

The fibre is then locally heated at each incision to its melting point for a short time shown in Figure 4.2, and this second step effectively introduces the required RI modulation at the core ⁵. Sufficient perturbation of the core is achieved after only 14 grating periods to enable coupling to the cladding mode to occur, creating compact devices. This is because the effective index changes introduced by this technique is approximately an order of magnitude greater than the largest permanent index changes achieved in photo-induced gratings ⁴. The immediate benefit of compactness is the reduced sensitivity to bending¹⁷. The LPG's response to external perturbations was measured and found to be comparable to photo-induced LPGs ³⁰.

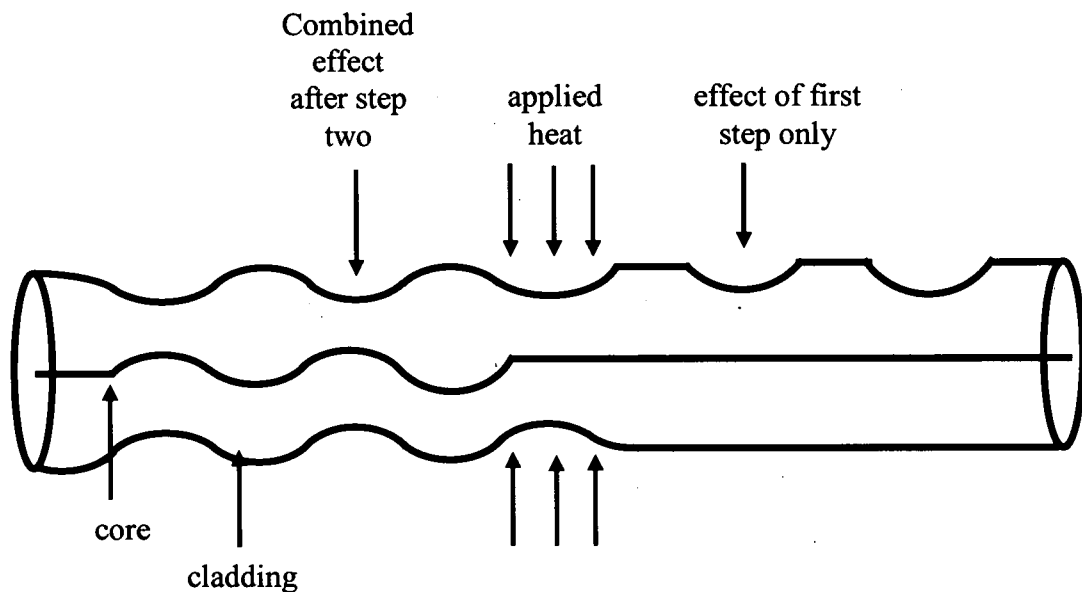


Figure 4.2: A schematic to show the induction of the required change in the RI of the core by locally melting the fibre, using the arc of a fusion splicer. The surface tension of the molten glass transforms the corrugation on the fibre surface into a sinusoidal deformation of the core. Diagram reproduced from ³.

Another method which is based on deformation of the fibre core relies on the induction of periodic micro-bends in the core by the use of electric arcs ^{5, 17}. The fibre is held firmly and untwisted between two fibre holders. A lateral stress is induced in the fibre by displacing one of the fibre holders by $\approx 100\mu\text{m}$ in a direction orthogonal to the fibre's axis. A section of the fibre, whose buffer jacket has previously been removed, is

heated by applying an electric arc. This causes a deformation in the fibre due to the lateral stress thus creating a micro-bend. The length of the bend, typically less than $1\mu\text{m}$, can be controlled by the duration of the arc. A series of micro-bends can be induced into the fibre without additional displacement of the fibre holders ⁵. To fabricate the LPG the electrodes are moved along the fibre by a distance equal to the grating period, and the process is repeated. The resonance wavelengths of LPGs produced using this method are determined by the grating period and are not affected by the coupling strength. This is because the micro-bending method of changing the index of the core does not induce a change in the beat length of two coupled modes ⁵. This method can be used in any type of fibre, including standard telecommunications single mode fibres. The LPGs have low insertion loss and high thermal stability, of up to 800°C . LPGs fabricated by this method offer flexibility in control of filter parameters and profiles by using simple apodisation techniques ⁵. The technique can be used to provide a low cost all fibre filters and mode converters for optical fibre communication and sensor systems ⁵.

Deforming the fibre cladding can also be used to introduce a periodic change of index profiles along the length of the fibre. The LPG consists of an etched corrugated structure. A section of the fibre has its cladding etched to reduce the cladding diameter whilst the adjoining section of the fibre remains unetched. Mode coupling occurs due to the difference in the stress fields induced within the corrugated structure when an equal external stress is applied ⁶.

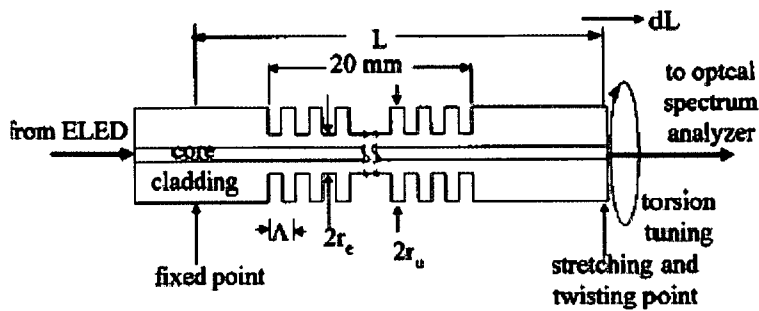


Figure 4.3: Schematic diagram showing the fabrication of an LPG made from a corrugated structure. Where Λ is the period of the corrugated structure and r_e and r_u are the etched and unetched radius of the cladding Diagram reproduced from⁶.

Each distinct region will experience different tensile stresses that are inversely proportional to the cross-section of the corresponding region ⁶. Therefore, by the photoelastic effect, the differential strain results in a periodic index modulation. The strength of this index modulation can be controlled by the applied tensile stress and thus the structure used as a tuneable loss filter ⁶. However, in the absence of any external stress no coupling can occur and no attenuation bands can be observed.

A similar technique is based on bonding a cladding etched fibre to a corrugated silicon fixture³¹. This produces a tunable LPG where the dynamic tuning range as large as 20 dB is obtained. The coupling to the cladding mode occurs due to a periodic shear strain on the fibre and fixture interface. Loss tuning is achieved by varying the temperature of the fixture over a temperature range from 25 to 245° C.

Liquid crystals have also been used to introduce a periodic refractive index modulation in the core of a liquid crystal filled hollow core fibre, as shown in Figure 4.4.

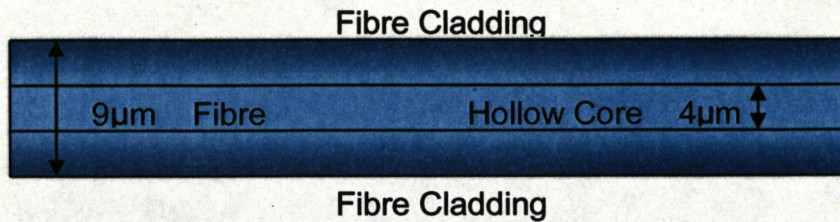
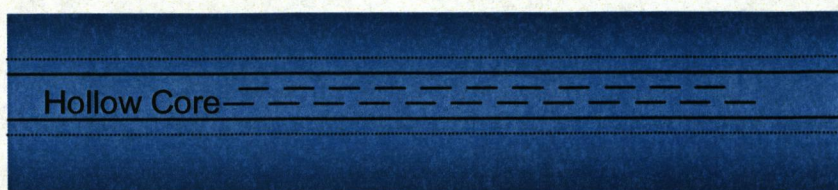
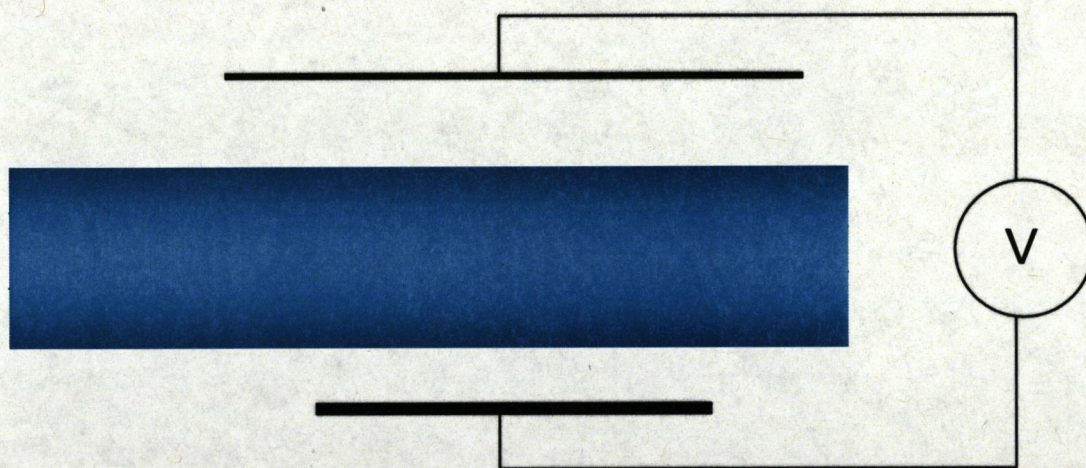


Figure: 4.4: A schematic to show the structure of a hollow core optical fibre.

The hollow core is filled with a nematic liquid crystal ¹¹ which in the absence of an applied electric field have molecules that align in a radial direction ³², as shown in Figure 4.5(a). If a voltage is applied the molecules align along the direction of the applied electric field, as shown in Figure 4.5(b). Utilising a comb-structured electrode to provide a periodic electric field it is possible to generate a modulation inside the hollow core since the effective index of the liquid crystal molecules depend upon the direction of poling. The effective index for the guided optical radiation changes with the direction change of the liquid crystal alignment and ²². The properties of the liquid crystal can be electrically controlled to manipulate a filter's characteristics³³.



(a)



(b)

Figures 4.5: A schematic to show the orientation of nematic liquid crystals when (a) no electric field is applied and the molecules align along the axis of the fibre and when (b) an electric field is applied and the molecules align themselves along the direction of the applied electric field.

4.2.3 Modulation of the core's refractive index

The techniques discussed in this section are based on the introduction of a periodic index modulation of the fibre via transformation of the physical properties of the core of the fibre, typically through irradiation. Several methods are discussed, with particular focus on UV irradiation, as this was the technique used for the fabrication of all LPGs used during the work presented here.

The refractive index modulation of the fibre's core can be achieved by high-energy ion implantation, using He^{2+} or H^+ ions implanted into the core of the fibre ¹². A change of refractive index of up to 1% is achieved by the compacting of the glass caused by the nuclear interaction of the implanted ions and the atoms in the fibre. This has been shown to occur mainly at the end of the travelling range of the ions which occurs when they come to rest in the host material ³⁴. The stopping distance of the ions is determined by the mass of the ion, its launch speed and the physical properties of the host material. The fibre may need to be prepared prior to ion implantation to ensure the ions can penetrate into to the core of the fibre. For example it is necessary to etch the cladding diameter from 125 μm , which cannot be penetrated by He^{2+} ion even at the highest possible launch speed, to a penetratable 53 μm . Etching is achieved using a strong acid such as Hydrochloric acid. For ion implantation using H^+ ions etching is not necessary as these ions can be launched at higher speeds.

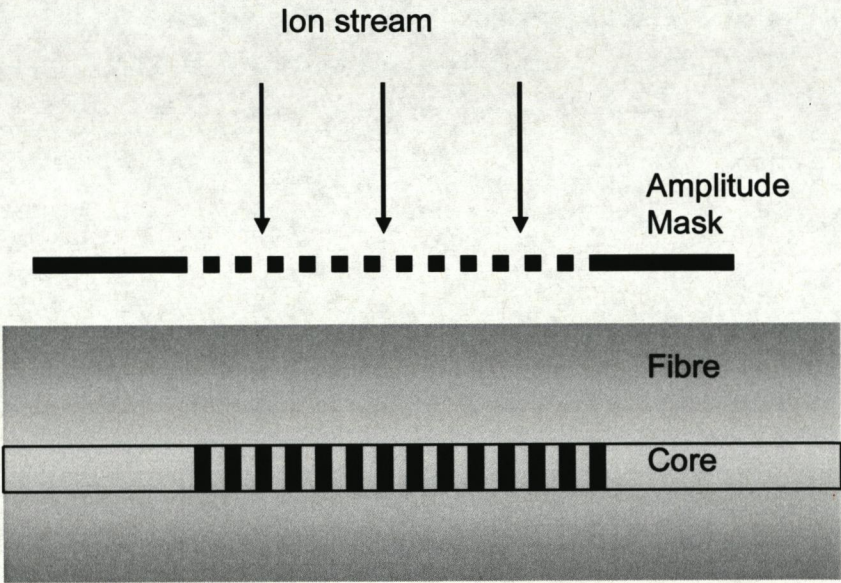


Figure 4.6: Schematic of LPG fabrication based on ion implantation through an Amplitude mask. Periodic modulation of the core is achieved where the fibre is exposed to the ion's through the amplitude mask.

The pre-prepared fibre is mounted inside a microprobe target chamber³⁵ and the ion beam is focused onto the fibre. The process uses an amplitude mask to irradiate specific areas of the core as the beam of ions cannot be sufficiently focused to bring about the desired modulation. A schematic of the alignment of the mask and the fibre is shown in Figure 4.6 .

There are several advantages associated with using ion implantation to fabricate LPGs. Firstly, since the fabrication process does not depend on the type of fibre used, it is possible to fabricate LPGs in almost all kinds of silica-based optical fibres³⁶. Secondly, the index change is approximately linear with ion dose at these concentrations of ions. This linear change eliminates the necessity for complex irradiation schemes of the type required for UV irradiation of photosensitive fibres³⁷. The implanting of ions into a fibre's core has been shown to produce significant changes in the RI of the optical fibre^{38,39}. Moreover the LPGs fabricated by this technique have been shown to be stable at above 500°C and thus would be suitable for temperature sensing applications⁴⁰.

Exposure of an optical fibre to the output from an infrared femtosecond laser, at a wavelength of 800nm has been used to fabricate LPGs⁷. The process induces a modulation of the RI of the core by light that penetrates through the cladding and the buffer jacket without being absorbed. An RI change of approximately 0.01% is observed and is brought about by a combination of factors. At present the detailed mechanism of this is not fully understood⁴¹. Local densification in the glass due to exposure is the main factor and since the densification of the glass is likely to result from thermal effects. The LPGs, based on the point-by-point fabrication process, show a high thermal stability below 500°C. This stability is associated to the different mechanism to achieve the refractive index change compared to UV induced RI changes. Another factor contributing to the RI modulation is a non-linear interaction through a multiphoton process and finally some of the modulation can be attributed to conventional heating by the laser in the localised regions⁷.

A three-photon absorption mechanism has been used to inscribe LPGs using high intensity 352nm femtosecond pulses to produce LPGs with significant polarisation properties⁴².

A refractive index change in the core of the fibre of 0.02% was achieved by directly exposing the fibre to 10.6 μ m wavelength CO₂ laser pulses⁸. The change was brought about by the release of residual stress in the fibre and the densification of the glass. Since the fibre was directly exposed to the CO₂ laser pulses, the absorption of silica at 10.6 μ m results in the majority of the energy being converted to heat in the first 10-20 μ m of glass. A focused CO₂ laser has also been used to fabricate multimode fibre LPGs for sensor applications⁴³

LPGs may be formed by thermal induction techniques to yield devices that have higher temperature stability in comparison with conventional photo-induced LPGs. The optical fibre is usually doped with chemical elements with a high diffusion coefficient in silica glass⁴⁴. The diffusion of Nitrogen to the cladding region has been proposed¹³. Since Nitrogen has a relatively small atomic weight, it can diffuse rapidly into the silica cladding at elevated temperatures. Periodic perturbations at the core and cladding interface can be induced by local heating of the fibre with a shaped beam of a CO₂ laser^{8, 9, 10} or by using a micro-arc discharge as a power source¹⁷. This can only be effectively initiated at temperatures in excess of 1400°C in order to successfully diffuse the Nitrogen. Thermo-induced LPGs retain their spectral properties even at temperatures of up to 1200°C¹³ thus offering distinct advantages over conventional LPGs.

Optical fibres contain inbuilt stresses and tension, induced during the manufacturing process. The release of the stresses can be used as another method to modulating the core. Dopants are employed to ensure that the periodic relaxation of this stress can be used to induce a RI change; typically a change of 0.1% is observed^{15, 16}. The dopant causes the cladding to have a lower softening temperature than the core and thus during the manufacture, when the fibre is drawn, the glass of the core solidifies faster than that of the cladding. The drawing tension remains in the core as a residual stress. To produce an LPG using this process the buffer jacket in the selected region of the fibre is removed and the fibre is held securely. The fibre is then locally heated at a selected point along its axis using CO₂ laser illumination¹⁵ or an electric arc¹⁶. The fibre is heated to the softening temperature of the cladding and the stress in the core is released. This results

in producing an increase in the local RI and the fibre is then translated by a distance equal to the grating period, Λ and the procedure is repeated.

Electric discharges have been used to produce periodic perturbations into the fibre core by this heating activated process. The main mechanism for the formation of the LPGs is considered to be the rapid heating and cooling process causing changes in the glass density, viscosity, index of refraction and Rayleigh scattering^{18,45}. The technique is a cost effective method of making LPGs, as expensive lasers and their associated equipment are not required. Another major advantage is that no special fibre, hydrogenised or pre-sensitised, are required for the fabrication⁴⁶. These LPGs have low insertion loss and high thermal stability enabling use at high temperatures.¹⁸. Simple apodisation techniques can be used to offer flexibility in control of filter parameters and profiles¹⁹. The technique can provide practical, low cost fibre filters and mode converters for optical fibre communication and sensor systems.

An index modulation of 1.8×10^{-3} in the RI of conventional dispersion shifted fibre with a Ge-doped core was achieved⁴⁷. Electric discharges have been used to fabricate LPGs into standard untreated telecommunications fibres, Corning (SMF- 28)⁴⁸, however an investigation into the mechanisms that forms these LPGs has yet to be conducted⁴⁸.

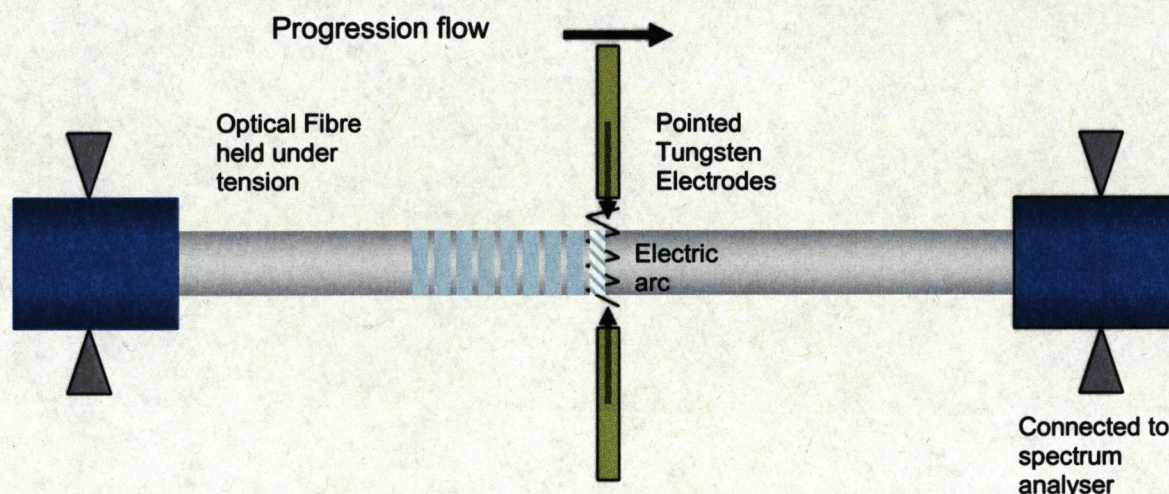


Figure 4.7: A schematic of the fabrication of a LPG using an electric arc formed between two electrodes using the point by point method

Figure 4.7 shows the fibre, with its buffer jacket removed and held longitudinally under tension, midway between two electrodes which are 2.54mm apart. The periodic modulation was induced by using pointed tungsten electrodes, which contained a trace amount of Thorium. When these were connected to a switched current source an electric arc formed between them. The fibre was exposed to approximately a 1.5mA current arc for a short period of time, typically about 2 seconds. Using the point-by-point technique, the LPG could be fabricated in a length of fibre that was not doped with hydrogen. Fabricating the LPG in hydrogen loaded fibre allows the current and exposure time to be reduced and thus produces a LPG that is mechanically stronger.

LPGs fabricated using electric discharges in S- or Ge-doped fibres have been shown to operate without any permanent degradation of their transmission spectrum at temperatures of up to 800°C¹⁸. LPGs fabricated in Ge-doped fibres were also shown to operate at temperatures of up to 1190°C for 30 minutes, thus demonstrating a unique method of monitoring these temperatures without the need for employing special fibres¹⁸. The transmission spectrum does suffer some irreversible change at these extreme temperatures but provides an important demonstration of the potential applications of LPGs fabricated by this technique. The irreversible degradation of the LPG's spectra at high temperatures is caused by two combined effects. Firstly, the annealing of intrinsic stresses within the fibre such as those induced during the fibre manufacturing process and secondly, by the local heating of the fibre during fabrication. At these temperatures the fibre undergoes plastic deformation and experience viscous flow¹⁸ which causes irreversible change in the physical properties of the fibre once the fibre is cooled.

Perturbations induced into standard telecommunication fibre, Corning (SMF- 28) by only 20 point-by-point steps produced a device length of between 10-14mm and gave 20dB isolation⁴⁸. Compared to a photo induced LPG in the same fibre loaded with Hydrogen, typically 100 steps would be required, forming a device of length 50mm. From a characterisation of the LPGs fabricated using this method, it was found that the resonance wavelength of the gratings depended only on the period and was independent of the arc current¹⁹.

4.3 UV irradiation to fabricate LPGs

4.3.1 Introduction

The method used to fabricate all the LPGs investigated within this thesis employed UV irradiation through an amplitude mask to induce periodic modulations into the core of a photosensitive Bo-Ge co-doped fibre. LPGs typically require periods in the region of 100 to 500µm and so are conveniently fabricated by UV exposure. A general overview of the UV irradiation source and the methods employed to fabricate gratings will be discussed.

4.3.2 UV Irradiation

The most common UV irradiation method utilises photosensitive fibre, generally germanium doped fibre, with UV irradiation through an amplitude mask or directly exposing the fibre point-by-point²¹⁻²⁷. The periodic modulation is achieved with UV radiation at a wavelength close to the absorption peak of germania related defects, at generally at a wavelength range of 240-250 nm⁴⁹. It has also been demonstrated that another second large defect band also exists at 195nm⁵⁰.

Fibres can be doped with elements other than germanium to exhibit photosensitivity. Fibres doped with europium⁵¹, cerium⁵² and erbium⁵³ also show varying degrees of sensitivity in a silica optical fibre, but not more sensitive than germanium doped fibres. The photosensitivity of the fibre can be enhanced further by co-doping with boron, producing gratings with saturated index changes approaching 10^{-3} ⁴⁹.

The use of dopants allows the alteration of the properties of silica, for example the refractive index. Fibres made with pure silica give rise to absorption band at around 160nm and exhibit low losses for light in the region of 820nm to 1600nm, as silica has a tetrahedral structure. Dopants such as germanium, also a group four element, can displace silicon in this tetrahedral structure. This results in the presence of absorption band centred at 240nm, with a bandwidth of around 30nm. Photosensitivity has been observed in fibres at wavelengths that can be absorbed at this band, either directly or via

a two photon resonant mechanism, thus it has received much attention from researchers⁵⁰.

Hydrogen loading or hydrogenation of fibres is a technique also used to increase the photosensitivity of the optical fibre. It involves the diffusion of hydrogen molecules into the fibre cores at high pressure and temperatures. The effect of the hydrogen is thought to enhance the defect density and thus the increased concentration of Ge-O deficiency centres absorbing near 240nm⁵⁴.

A UV laser beam at a wavelength of 157nm has been used to fabricate LPGs by exposing the fibre to the UV through an amplitude mask²⁵. A short wavelength was used^{55, 56, 57} after it was demonstrated that an improvement by a magnitude in the RI modulation in a low GeO₂ doped fibre, could be achieved by reducing the laser's wavelength from 248nm to 193nm⁶². The photosensitivity was attributed to two-photon absorption across the GeO₂ band. The fabrication at 157nm was attributed to access to a new single photon process near the band edge of GeO₂ and fused silica. The LPGs produced in this way have attenuation bands that have a minimum transmission value of -17dB²⁵. The use of lower wavelengths overcomes the requirement to enhance the photosensitivity of the fibre prior to exposure.

Although the mechanism responsible for the induced index change is not clear, it is certain that the germanium defect related absorption- band is strongly associated with the effect. Absorption of energy into this band causes a permanent change in the RI, which may be attributable to several mechanisms, in part through the Kramers-Kronig principle⁴⁹. The UV radiation causes bond breaking of point defects in the glass matrix^{58, 59} which is free to move within the matrix until it is trapped again⁶⁰. This increases the absorption in the UV spectrum and therefore a concomitant increase in the RI of the core⁶¹ is observed. The induced RI change can be increased by a factor of ten if UV radiation at a lower wavelength is used⁶².

Most fibres undergo UV induced RI modulation by the increase in GeE' centers due to the UV exposure and photoexcitation of oxygen vacancy defect states. This structural change, due to electron transfer from Germania related defects do not occur during

thermal processes⁶³. Defects related to the paramagnetic GeE' centres can relax even at temperatures below 100°C and so the LPG's performance degrades thermally.

The requirements of the UV source are less critical for the fabrication of LPGs than for the fabrication of FBGs. The limitations placed on the coherence length are reduced since the fabrication process does not require interferometric fringes. The mechanical stability requirements of the writing system are also less stringent because of the larger grating period used.

Several UV writing sources for fibre grating fabrication are available, such as CW frequency-doubled Ar lasers and pulsed excimer lasers. Recently, new types of UV or near-UV light sources have been reported for writing through the fibre jacket and for multiple-grating fabrication. A UV light source at 255nm generated from a frequency-doubled copper vapour laser (CVL) has been reported to fabricate LPGs with higher efficiency, despite not operating at the absorption band of the Boron-germanium co-doped fibre⁶⁴.

Although the UV based fabrication method is well-established in fabricating gratings, it has several disadvantages that need to be discussed. Firstly, it usually requires the preparation of the optical fibre by hydrogen loading for the enhancement of the photosensitivity of the fibre. Secondly, the optical fibre needs annealing to remove residual instability of the refractive index modulation which may otherwise result in further refractive index change and hence movement of the central wavelengths of attenuation bands. Thirdly, the control of filter parameters is more difficult as the resonance wavelength of the filter not only depends on the LPG's period, but also on the amplitude of the index modulation²³. The selection of the LPG's period must be made in view of both the coupling wavelength and the efficiency of the LPG to be fabricated. This makes it more difficult to implement the apodisation technique for tailoring a filter profile.

4.3.3 Fabrication of LPGs using the amplitude mask technique

The use of an amplitude mask to allow the simultaneous generation of multiple periods of modulation of the optical properties of the fibre is a major step towards easier inscription of fibre gratings. It is in keeping with the demand for mass production and repeatability during fabrication with controlled spectral response

The technique acts to simplify the manufacturing process, as it enables easier alignment of the fibre for photo-imprinting. Thus the stability requirements of the photo-imprinting apparatus are less stringent. It is however difficult to change the parameters of the LPG using this technique and therefore if greater control is necessary than the point-by-point fabrication technique is more suited. However the use of the amplitude mask enables the production of LPGs with identical parameters to be produced more rapidly.

There are four types of amplitude masks that are available for the production of gratings. These can be made from chrome-plated silica⁶⁵, metal masks⁶⁶, dielectric masks⁶⁷ and microlens arrays⁵. The low damage threshold of chrome masks and dielectric masks necessitates that low power is used and this increases the fabrication time. Metal masks consist of slots which are precision laser machined out of thin copper sheets. These act to spatially modulate the intensity of the writing beam. Since they have a higher threshold level, $> 1\text{J}/\text{cm}^2$, they can withstand high power and thus the writing time is reduced considerably. However all amplitude masks are exposed to a significant part of the laser beam which does not radiate through the mask and so are not energy efficient and their use with high power lasers shortens their lifetime⁶⁶.

Focusing micro-optics constructed with UV grade fused silica is a more suitable material as it has a similar damage threshold to the fibre in which the periodic modulation is to be induced. In addition it focuses the laser beam into a periodic pattern and so less energy is required from the laser. However, the extremely high cost of microlens arrays prohibits their use in LPG fabrication⁶⁶. Microlens arrays constructed using low cost UV grade fused silica (UVGFS) fibres have been demonstrated⁶⁶.

A dielectric mask has been produced by selectively etching off more than 99% of the reflecting layers of a commercial KrF excimer laser mirror⁶⁷. The mask enables the LPGs to be written quickly and with a high intensity laser output. The LPGs were formed in hydrogen loaded fibre by exposing it to the output of a UV adapted Argon laser, of wavelength 248nm and intensity of 120mJ/cm² per pulse. Using this method an LPG of length 210mm can be fabricated within only a minute. Since the exposure time can be increased, attenuation bands of depth up to 25dB can be observed.

A relatively inexpensive micro-lens array can be constructed for the mass production of LPGs by utilising commercially available UVGFS fibre. The array is placed between the fibre, to be exposed, and the UV beam of an excimer laser. The periodicity of the LPG is determined by the cladding diameter of the fibre used in the array. LPGs produced in this way give an attenuation of -11.7dB after 50 seconds, whilst under identical conditions but using a metal mask, an LPG with -10.9dB attenuation after 200 seconds produced⁶⁶. This shows that the microlens array method is far more efficient. Since the array has a similar damage threshold as the fibre to be exposed, less energy is used from the laser, however the array must be positioned near the fibre with micron precision.

4.3.4 Fabrication of LPGs using the point-by-point technique

The point-by-point configuration forms each modulation within the optical core of the fibre one point at a time and thus allows greater flexibility in the characteristics of the LPG fabricated⁶⁸, such as apodisation chirp and phase steps⁶⁹. Each grating plane is produced separately by a focused single pulse of UV light passing, for example through a mask containing a slit. A focusing lens is positioned to orientate the image of the slit onto the core of the fibre from the side. The refractive index of the core increases at the section of fibre irradiated and then the fibre is translated through a distance corresponding to the period of the grating, in a direction parallel to the fibre axis. This process is repeated to form the required grating structure. Alternatively the source can be translated rather than the fibre, however the stability and accuracy of the translation system is essential in either method.

The RI profile of the grating can be controlled to produce the required spectral response as the UV pulse energy can be varied between points of induced index change. However, LPGs fabricated using the point-by-point method are prone to errors in the grating spacing due to thermal effects²².

4.3.5 Annealing of long period grating

The thermal stability of LPGs is important for applications in telecommunications and sensing. The removal of the instability caused by the induced RI modulation results in a change in the wavelength and reflectivity of FBGs^{21, 70, 71, 72}. The effect has also been reported for LPGs, where a change in the central wavelength of the attenuation bands and the coupling efficiency is observed^{73, 74, 75}. Annealing of the LPG is carried out at temperatures above their maximum operating temperature as this promotes long term stability in the device by fixing the spectral characteristics of the fibre^{70, 72}. Annealed fibre gratings exhibit a linear wavelength response at high temperatures which can be exploited for accurate wavelength tuning and the development of sensor elements⁷⁶.

In an LPG the wavelength at which the coupling from the core to the cladding modes takes place is directly dependent on the effective UV induced RI change, Δn_{eff} , the period of the grating and on the difference in the RI of the core and the cladding, Δn . A large shift in the central wavelengths of the attenuation bands in the transmission spectra of a LPG would result if either Δn or Δn_{eff} decrease. For example, a 2% decrease in Δn_{eff} would have a negligible effect in a add/drop wavelength-division multiplexed (WDM) Bragg grating ($\Delta\lambda < 10 \text{ pm}$). However a similar decay in a LPG would shift the centre wavelength by $\approx 0.5\text{nm}$. Such a shift would be unacceptable for some applications, such as gain equalisation⁷⁶.

4.4 Fabrication of LPGs and concatenated LPGs used in this work

4.4.1 Introduction

This section describes in detail the procedure to fabricate the LPGs and concatenated LPGs used during the work presented in this thesis⁷⁷. The fabrication employed UV irradiation of Fibrecore's Boron-Germanium co-doped fibre photosensitive fibre (PS 750, cut-off wavelength of approximately 650nm), at a wavelength of 266nm through a copper amplitude mask.

4.4.2 Experimental description

4.4.2.1 Fibre preparation

Prior to the fabrication of the LPG the buffer jacket was removed by placing the fibre onto a glass slide and applying dichloromethane, on to the region of fibre to be irradiated by the UV source. After approximately one minute the buffer jacket was observed to detach from the fibre and was removed completely, along with excess dichloromethane by a lens tissue doused in Isopropanol. The clean fibre was terminated with FC301 connectors in preparation to be placed on the translation system.

4.4.2.2 UV Laser

The UV irradiation source used in the laboratory was a Spectra Physics, Quanta-Ray, GCR 100 Series injection seeded Neodymium doped Yttrium Aluminum Garnet (Nd:YAG), frequency-quadrupled, laser. The laser operates at a wavelength of 266nm, with a pulse width of 5nsec and a repetition rate of 10Hz. Prior to fabrication, the laser system required an hour to warm up, to enable the laser system to run at the maximum flash lamp energy. This ensured the optimum beam quality through the reduction of the pulse to pulse energy fluctuations of the Nd:YAG crystal. Phase matching for the Nd:YAG, fourth harmonic generating birefringent crystal is accomplished by rotating

the crystal. This achieves optimum conversion efficiency but since the optimum angle is temperature dependent, the angle of the crystal requires adjustment periodically during warm up until thermal equilibrium is reached.

4.4.2.3 The interrogation system

The transmission spectrum was monitored using this interrogation system throughout the work presented within this thesis. Described here is the system used for the on-line monitoring of the transmission spectra of the LPGs during fabrication.

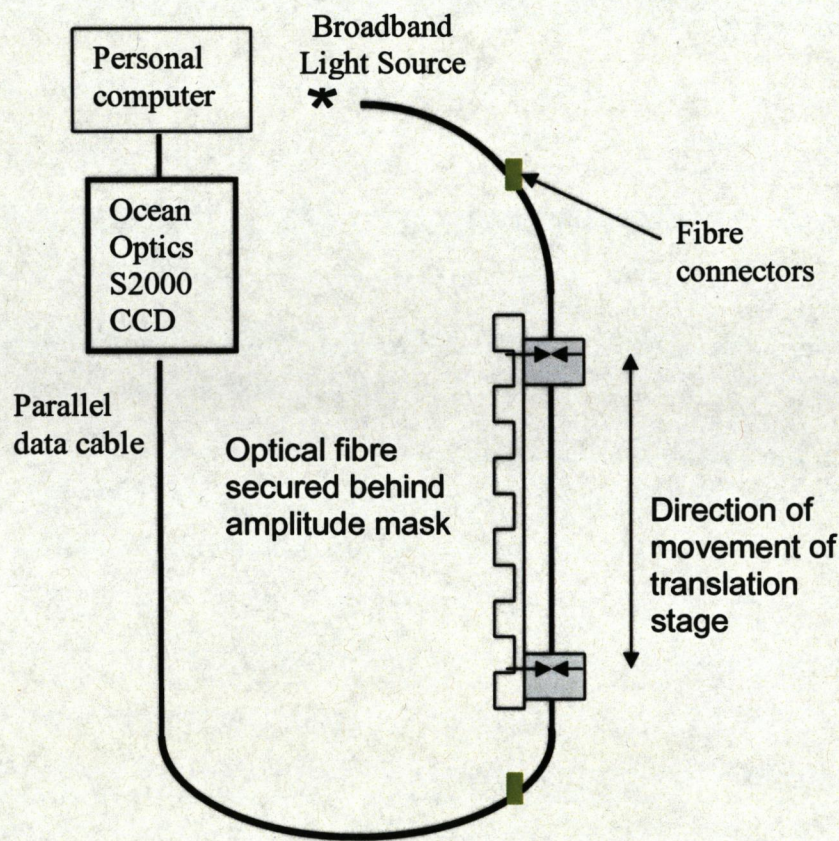


Figure 4.8: General experimental configuration of the major components of the interrogation System used to monitor the transmission spectrum of the LPG during fabrication.

Figure 4.8 illustrates the general experimental configuration of the major components in the interrogation system. The broadband light source used was an Ocean Optics, fan cooled Tungsten Halogen light source, HL-2000. It has a spectral range of $\approx 220 - 1100\text{nm}$ within a power a stability of $\pm 0.05\%$. An Ocean Optics S2000 CCD spectrometer is coupled to the distal end of the fibre. The spectrometer is interfaced to the PC where a Labview program allows the real time monitoring of the transmission profile. The spectrometer offers measurement of a spectrum extending from 500nm to 1100nm with a resolution of 0.7nm and a minimum integration time of 1ms .

The Labview program allows the transmission spectrum to be logged at a rate of up to 1Hz . The central wavelengths and the minimum transmission values of the attenuation bands were determined through a polynomial fit to the transmission spectrum of the band, thus enhancing the resolution to 0.3nm .

4.4.2.4 Fabrication system and components

The configuration of the UV based amplitude mask fabrication technique is shown in Figure 4.9. The process used a copper amplitude mask supplied by Oxford Lasers. The mask was 40mm long with a duty cycle of 50% and a periodicity of $400\mu\text{m}$. It was fixed onto the blocks 5mm in front of the v-grooves.

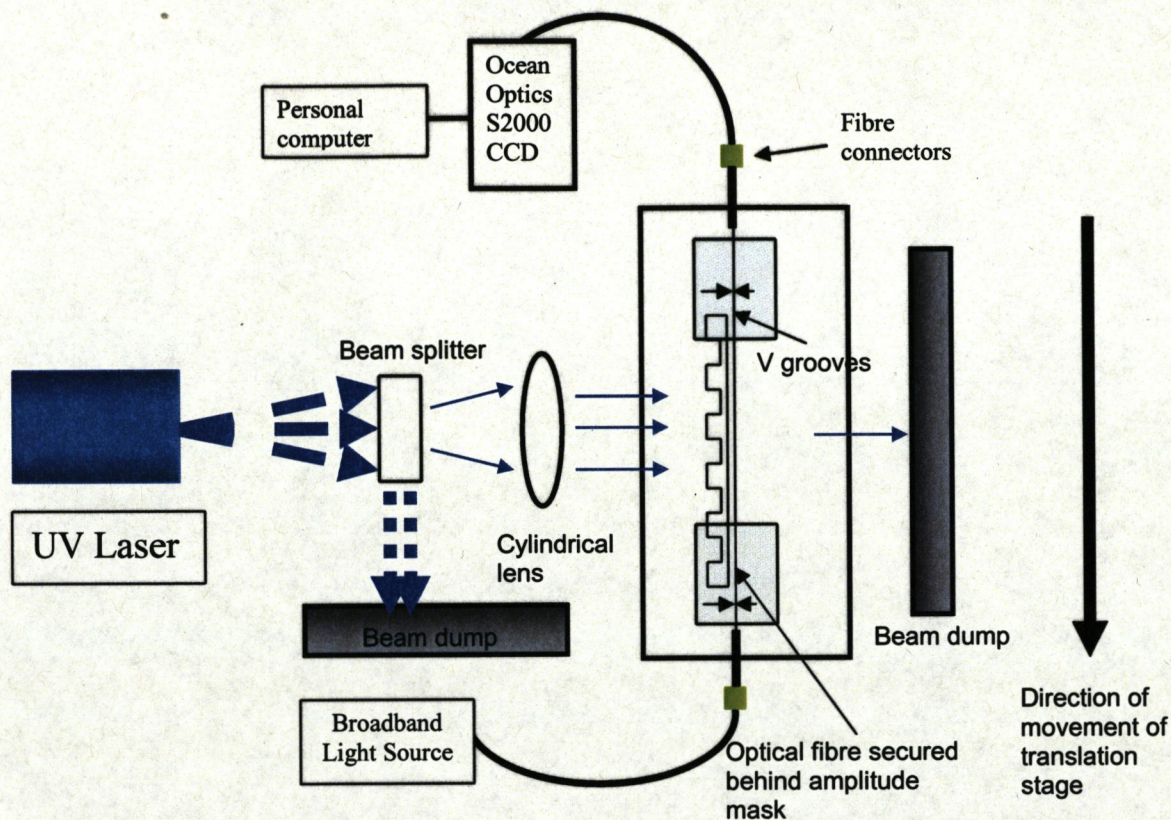


Figure 4.9: Configuration of the UV based fabrication using the amplitude mask technique

The output power of the UV laser was split using the 95%-5% beam splitter, where 95% of the laser beam was reflected into a beam dump whilst the remaining 5% was transmitted. The power was measured immediately after the beam splitter with an Ophir laser power meter, model N^o. AN/2 and UV detector head, model N^o. 30(150) A-HE-SH. The average power incident on the fibre was 50-60mW to ensure that the optical fibre did not experience mechanical failure during or post fabrication. The cylindrical lens, of focal length 100mm, was positioned to focus and orientate the UV into a line focus of length 10mm parallel to the axis of the optical fibre and to the motion axis of the translation stage. The beam was blocked whilst the prepared fibre was positioned on the v grooves. The fibre was positioned so that the region to be exposed was directly behind the amplitude mask. Magnets were used to secure the untwisted fibre, straight and taut, in the v-grooved blocks. The v-grooved blocks were mounted on a computer controlled stepping motor driven translation stage. The translation stage had a travel length of 50mm and a resolution of 2 μ m.

A NEAT 300 series programmable motion controller interfaced with a hyperterminal program allowed control over the number of steps and exposure time per step to define the spectral final parameters of the LPG. The translation stage is positioned at its starting point, the program executed and UV beam unblocked.

The fibre and amplitude mask traversed in a direction parallel to the fibre's axis in front of the stationary focused UV beam with incremental step sizes of about 100 μ m. The dwell time was 10 seconds at each position, these steps being repeated until a total of 40mm of length were exposed to the UV beam in approximately one hour. The RI perturbation using this method had a Gaussian profile.

The fabrication of concatenated LPGs was undertaken using the same configuration as shown in Figure 4.10. In order to produce two LPGs the translation stage was programmed to move quickly after the required length of the first LPG was achieved to the starting point of the second LPG. The computer program was used to fabricate the desired length of each LPG and the separation between them. The transmission spectra were monitored during the fabrication and the appearance of the attenuation bands and interference fringes, for the concatenated LPGs, was possible.

4.5 Chapter Summary

Several techniques have been discussed to induce a periodic modulation into the core of an optical fibre. These were compared and their respective advantages and disadvantages were outlined. The UV irradiation through an amplitude mask was the technique used for the fabrication of LPGs during the work presented in this thesis, so particular emphasis was given to describe this method. The experimental details to fabricate LPGs and concatenated LPGs were given.

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Chapter 5 Langmuir-Blodgett Technique

1 Introduction

Langmuir-Blodgett (LB) technique has been studied extensively^{1,2,3,4} and provides a means for overlay deposition with control over the overlay thickness on the nanometer scale. Although several thin overlay deposition techniques are available, such as Electrostatic Self Assembly (ESA)⁵ or covalent self assembly⁶, the LB method offers a number of unique advantages, discussed within this chapter, that make it particularly suitable for the development of the devices that are investigated here.

The LB technique has been used to facilitate an investigation of the influence of the presence of an overlay material upon the characteristics of LPGs and therefore a brief review of the technique will be given here. A brief review of other organic self-assembly methods, namely ESA and covalent self-assembly which have been used by other researches, will also be presented.

5.2 Electrostatic self-assembly

The ESA technique involves the adsorption, one layer at a time, of alternate anionic and cationic complexes from aqueous solutions. The method can be adapted to incorporate a wide variety of molecules, including nonlinear optical chromophores, conducting polymers, biological macromolecules, magnetic materials, dielectrics and metallic nanoparticles. The composition of each layer can be controlled by the incorporation of different molecules and the structure of each layer can be manipulated by adjusting deposition parameters⁷.

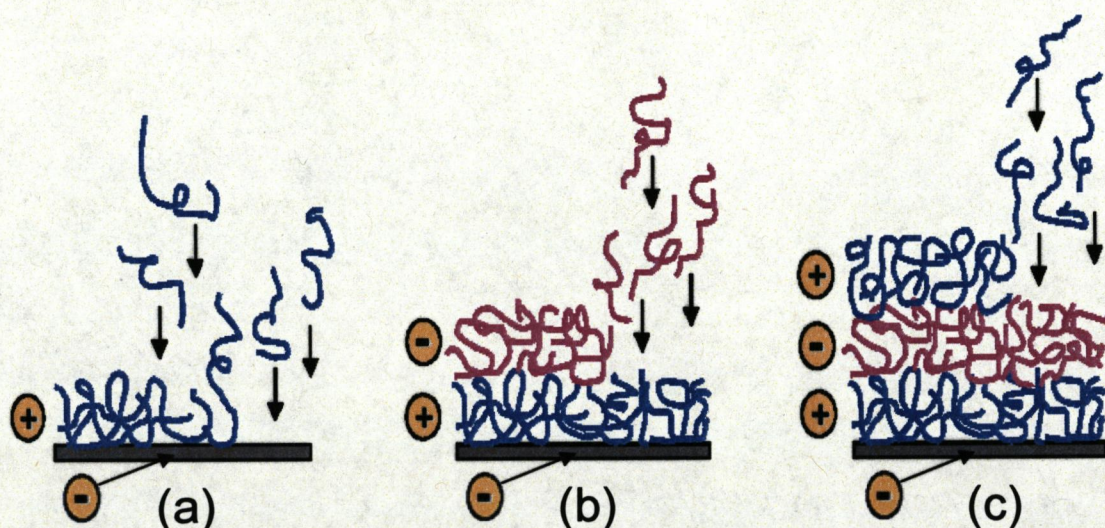


Figure 5.1: Schematic for the build up of multilayer assemblies by consecutive adsorption of anionic and cationic polyelectrolytes. (a) shows the charged substrate and assembly of the first polyelectrolyte monolayer, (b) shows the assembly of the second polyelectrolyte monolayer and shown in (c), the third monolayer assembly on the first bilayer. Diagram reproduced from ⁷.

Figure 5.1 shows the process used in the ESA technique to build overlays on substrates. Firstly the substrate is prepared for deposition of a layer by thoroughly cleaning and chemically processed to apply a negative charge, as shown in Figure 5.1. The charged substrate is then dipped into an aqueous solution containing polycations, shown in Figure 5.1 (a). These are attracted to the anionic substrate surface and assemble themselves into a molecular monolayer. The molecular order is near perfect due to the stability achieved by distributing electrical charge uniformly over the surface. The substrate is then removed from the solution and flushed with ultrapure water to remove any loose particles that may be held on the surface by weak van der Waals forces and that do not form part of the ionically bonded monolayer. This step is repeated, with the substrate now dipped into a polyanionic solution prior to flushing with ultrapure water. In such a way subsequent layers can be deposited.

5.3 Langmuir-Blodgett Deposition

LB deposition involves the transference of a molecular layer from an ultra pure sub phase onto a solid substrate. An LB consists of either a single molecular layer or a series of molecular layers of an organic material transferred one layer at a time onto dielectric substrates. The typical length of the molecule is approximately 3nm, offering high resolution control over the overlay thickness. The ability to control deposition thickness to such a high degree makes this technique ideal for fabrication of devices based upon planer waveguides. The coupling of light from the core of a single mode fibre to an LB waveguide overlay has previously been demonstrated to form channel dropping filters⁸, pH sensors⁹ and an effective method for producing second harmonic generation¹⁰. For the purpose of this research, the equipment was modified to enable deposition onto optical fibre as discussed in Section 5.4.

Molecules suitable for deposition using the Langmuir-Blodgett technique are amphiphilic surface active materials placed at the interface of two dissimilar phases such as liquid and gas^{1,4}. The molecules contain spatially separated hydrophilic and hydrophobic groups, illustrated in Figure 5.2, trapped at the interface due to the different types of bonding within the same molecular structure¹¹.

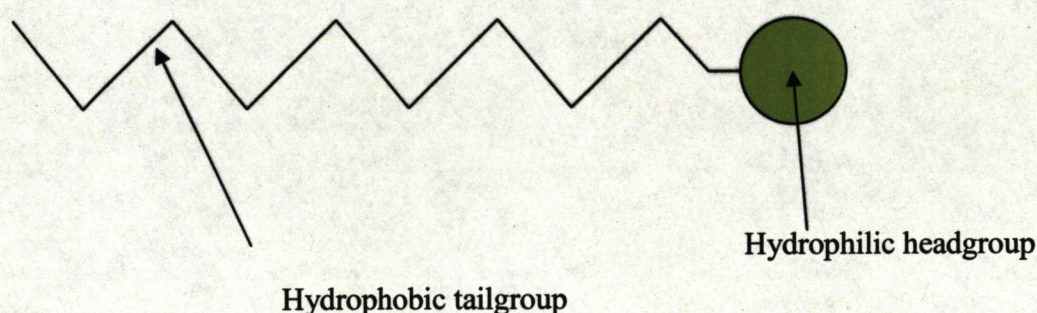


Figure 5.2: Schematic diagram of a typical molecule suitable for LB technique.

The molecules must be insoluble in water and readily soluble in a non-aqueous volatile solvent. The material is firstly dissolved in a solvent such as chloroform and the solution

is introduced onto the polar ultra pure water surface. The solvent evaporates leaving the molecules orientated such that the hydrophilic headgroups are attracted to the water surface whilst the hydrophobic tailgroups are directed towards the air. This forms a loosely packed disordered floating monolayer, as long as the amphiphatic balance of the molecule is correct. If the surface area of the water is subsequently reduced, by means of moving barriers, the molecules are forced into a more ordered, compressed monolayer as illustrated in Figure 5.3. The film at the surface is known as a Langmuir film and can be stacked sequentially onto a solid substrate.

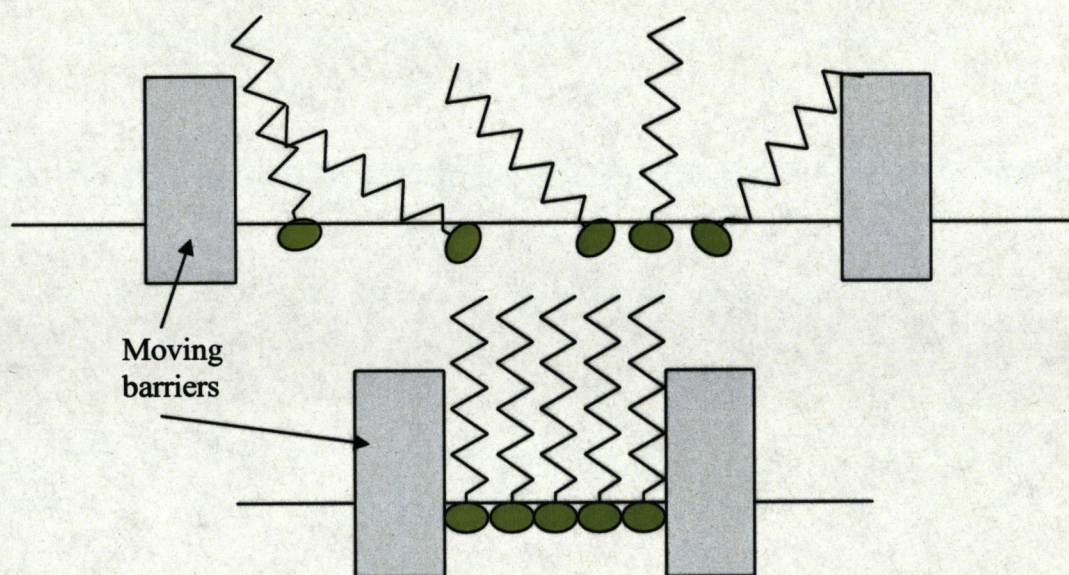


Figure 5.3: *Uncompressed and compressed monolayers of LB molecules on a water surface.*

In order to monitor the floating monolayer during compression and to establish when an ordered monolayer has been formed, surface tension of the air-water interface is measured. The change in surface tension is known as the surface pressure of the monolayer and is measured with a pressure sensor placed in the sub phase. A plot of surface pressure versus area per molecule at a constant temperature, an isotherm, is taken during compression. An idealised isotherm, such as that shown in figure 5.4, can be interpreted as showing the three distinct phases that the film undergoes during

compression. These phases may be understood as 'gas', 'liquid' and 'solid' and relate to the different degrees of freedom experienced by the molecules.

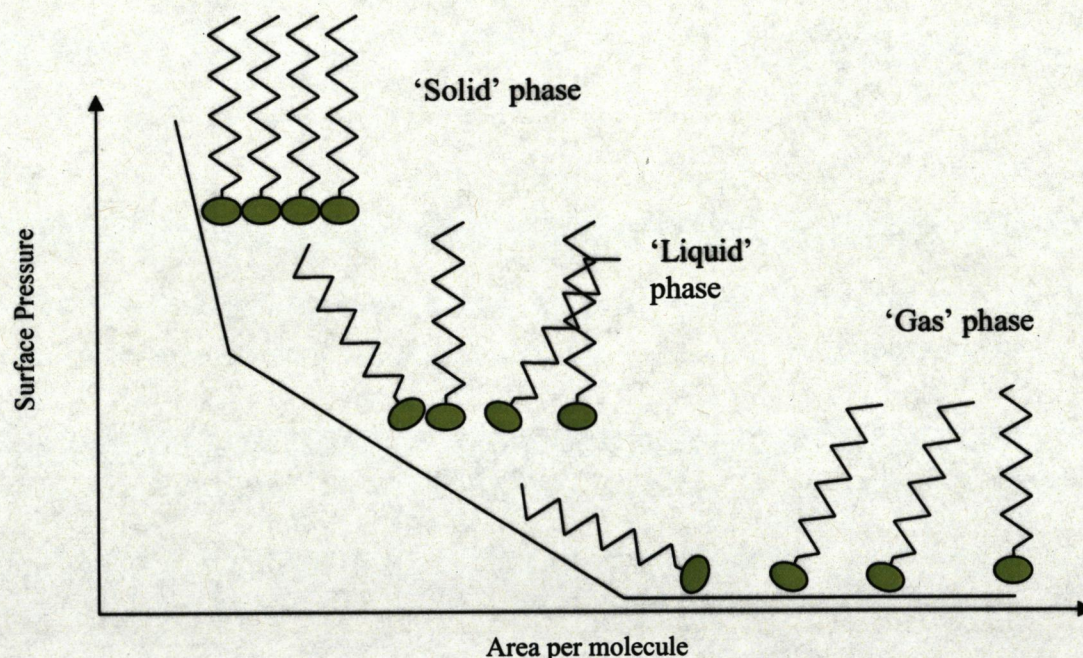


Figure 5.4: Idealised isotherm showing different phases of molecules on the water surface.

Prior to compression of barriers, the molecules have a high degree of freedom and move on the surface of the water like a two dimensional gas. As the molecules are compressed they enter a 'liquid' phase where some ordering begins to occur resulting in a rise in the surface pressure. Further compression forces the molecules to form a regular structure of singular molecular thickness and to behave as a quasi-solid, characterised by a steep and usually linear increase in surface pressure. It is possible to continue applying further pressure until eventually the film collapses and loses its mono-molecular form, identifiable by a sudden drop in surface pressure. Deposition onto substrates is achieved during the 'solid' phase and so the isotherm provides critical information about the ideal pressure for deposition.

5.4 Deposition of films

All LB deposition was carried out using a Nima technology alternate layer trough, such as that illustrated in Figure 5.5. The main features of the trough and a description of its operation will be given here. The trough was modified to facilitate deposition on optical fibres. The influence of the film upon the properties of the optical fibre was monitored on-line during deposition. This was achieved by coupling the output from a broadband source into the fibre and monitoring the transmission spectra using a spectrometer.

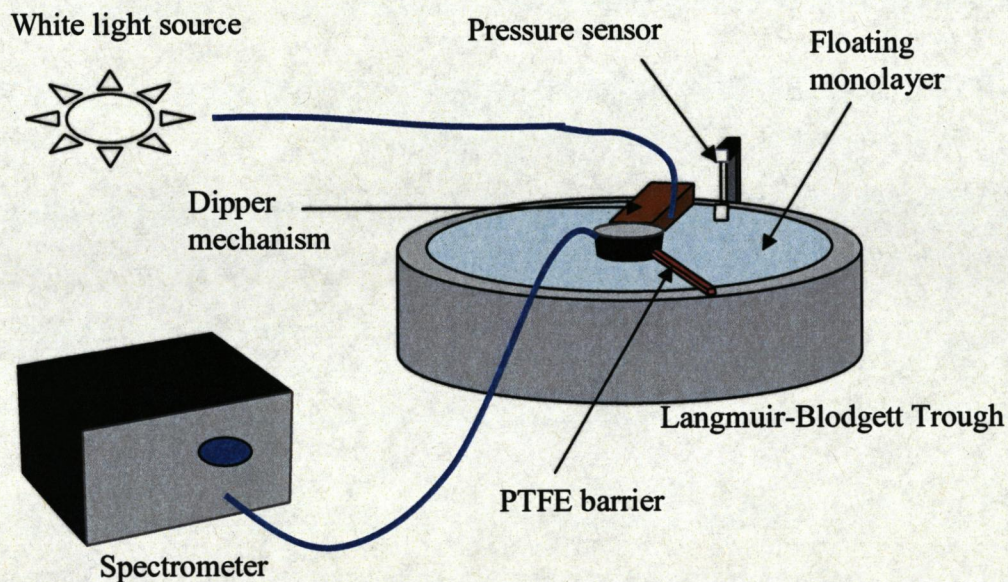


Figure 5.5: Schematic Diagram of a LB trough.

An LB trough usually has a relatively small surface area, typically 100cm^2 to 1200cm^2 , is made from PTFE and contains a centrally placed well in which the dipper mechanism is located. The dipper mechanism separates the area into two compartments whose surface pressure can be controlled in isolation by two rigid PTFE barriers enabling deposition in either compartment. All research reported here was carried out in one compartment of a circular trough of a surface area 600cm^2 , which was modified to facilitate deposition onto

an optical fibre. The dipper mechanism contained a motor driven chain to lower and raise a substrate, the motor of the dipper was controlled via a computer interface and its position monitored. The dipper was made from stainless steel and PTFE parts, whilst the chain was fabricated using nylon.

To allow deposition to an optical fibre, the glass slide holder (a) was replaced by a bracket (b), which was attached to the chains of the dipper mechanism. Figure 5.6 shows in detail the dipper mechanism used and how it was adapted for the purpose of this research.

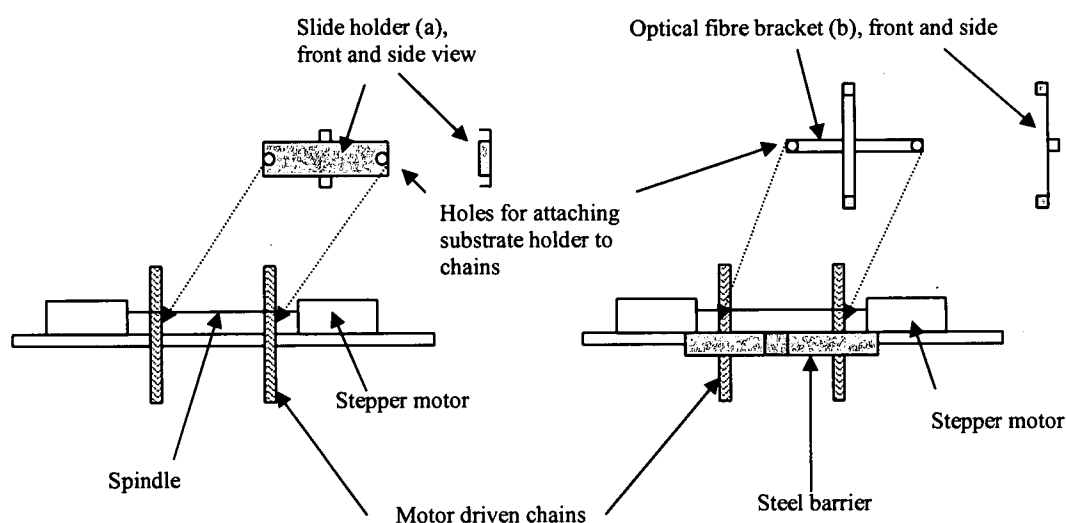


Figure 5.6: Schematic Diagram of central dipper mechanism showing the original glass slide holder and the modified bracket for optical fibre.

Optical fibres may be attached to the modified bracket by applying pressure to two PTFE washers by screwing two stainless steel screws at either end of the vertical arm of the bracket. The PTFE washers enable even distribution of the applied force and provide a surfactant-free surface when cleaned with chloroform. The bracket was then fixed to the chains of the dipper mechanism by screwing directly into both the chains.

A steel barrier was constructed to avoid wastage of the monolayer¹². It was designed to run along the whole length of the dipper mechanism and to be in contact with the floating monolayer and was fixed between the bracket and the optical fibre.

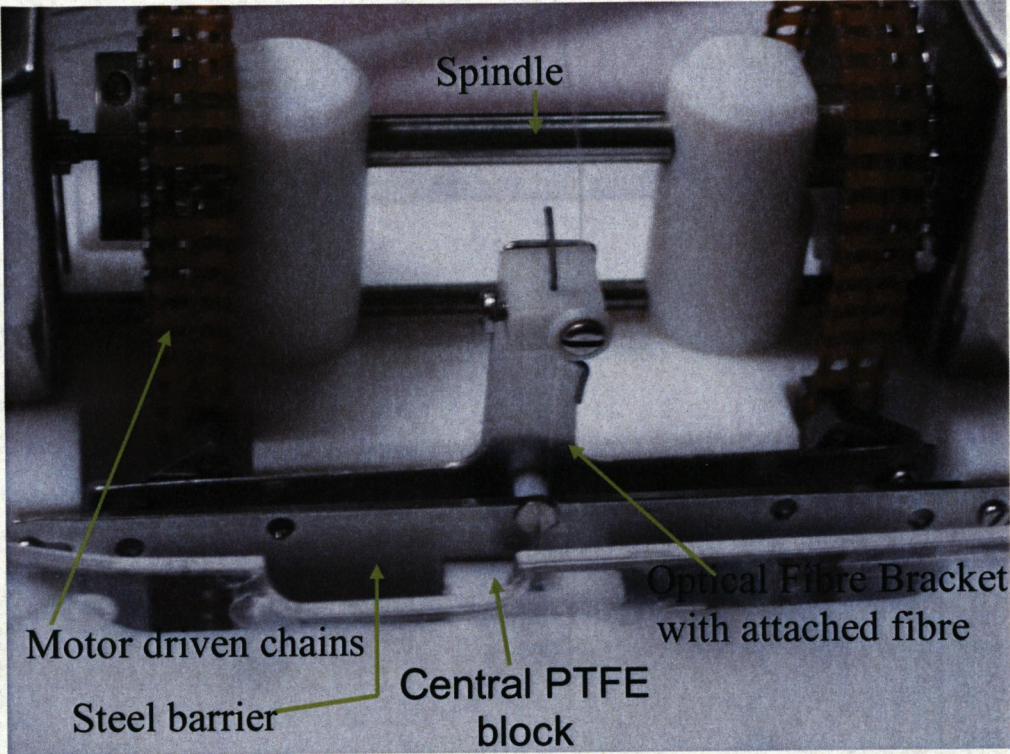


Figure 5.7: A photograph to show the dipper and modified optical fibre bracket in position, with an attached optical fibre.

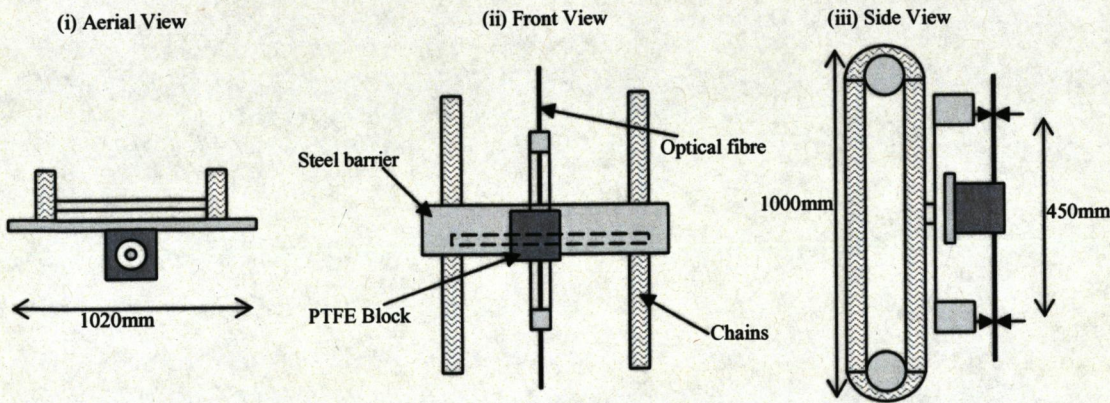


Figure 5.8: Diagram to show detailed schematic diagrams of the modified dipper mechanism with an optical fibre attached; (i) an aerial, (ii) front and (iii) side view.

The barrier ensured the deposition only occurred on the optical fibre itself and not onto peripheral surface areas. This provided a significant improvement in the technique by ensuring hundreds rather than tens of molecular layers could be deposited from a single spreading, thus reducing device fabrication time. This was advantageous since each molecular layer originated from the same source, eliminating issues of contamination in progressive depositions. The barrier restricts deposition to only one compartment of the trough, but was not a limiting factor for the research presented.

5.4.1 Types of Deposition

Deposition occurs as the substrate is passed down into the water (down-stroke) or as it is passed up out of the water (up-stroke). It is possible to deposit a multi-layer film onto a substrate in a unidirectional fashion, depositing only on the up-stroke or on the down-stroke or to deposit on both the up- and down-strokes of the fibre. These two deposition methods lead to different ordering of the LB films on the substrate. One of the biggest advantages of this technique is the ability for asymmetric deposition, when unidirectional deposition is used. There are two methods to achieve unidirectional deposition x-type and z-type structure³. For the work presented here deposition occurred during both directions, known as symmetric deposition. Repeated passes through the film gave a Y-type structure in which the amphiphilic molecules packed head-to-head and tail-to-tail.

With the barrier position such that the surface pressure is sufficient to ensure that the Langmuir film is in the solid phase, the substrate is passed through the floating monolayer in one direction. Once the required deposition length has been achieved, the direction of the dipper is reversed and the substrate passes through the subphase in the opposite direction. During deposition the surface pressure is kept constant by closing the barriers to decrease the surface area to compensate for the loss of molecules from the subphase. The deposition process is illustrated in Figure 5.9, showing the deposition of an LB film onto a hydrophilic substrate. As the substrate passes through the surface it

attracts the hydrophilic headgroup of the LB film. When the substrate is drawn back through the monolayer, the first molecular layer is deposited on the surface. The subsequent pass then attracts the hydrophobic tailgroups of the floating monolayer to those deposited onto the fibre thus depositing a second layer. This process can continue until multi layers are deposited onto the solid.

In order to ensure that the monolayers don't peel off after deposition, it is important to remove the substrate completely out of the water. Where this is not possible, as in the case of optical fibre, it is necessary to ensure a step change in the fibre at the end of the deposition stroke. This can be achieved by fabricating the LPG such that the buffer jacket of the fibre can be used to provide the step change. If even this is inappropriate, then the deposition must be carried out very slowly as this is found in practise to enable the layers to stay attached to the substrate. The slow movement of the substrate allows the Langmuir film to overcome its attraction to the sub phase in preference to the substrate.

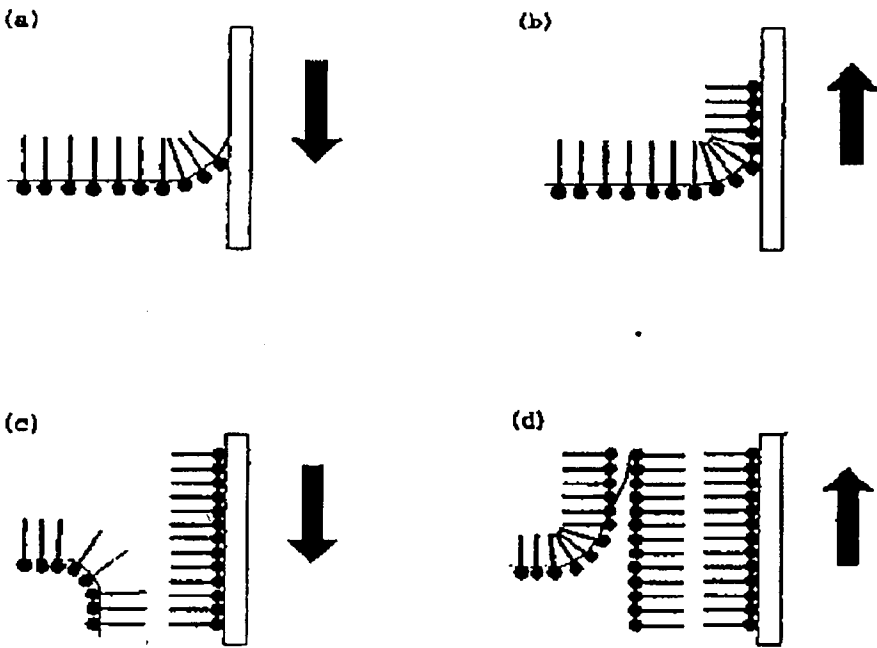


Figure 5.9: Diagram showing how multi layer films are deposited one layer at a time as the substrate is passed through the water.

5.4.2 In Situ monitoring of Langmuir-Blodgett deposition

A major advantage of the LB technique is the ability to monitor the deposition on the device during construction, and to observe in real time the response of the transmission spectra of the device to increases in the thickness of the Langmuir-Blodgett film. Figure 5.10 shows schematically the experimental apparatus used during the deposition of the LB films onto optical fibres containing the LPGs. The experimental configuration of the interrogation system is given in Chapter Four. A program in Labview controlled the movement of the dipper and monitored the transmission spectrum, logged at a rate of up to 1Hz.

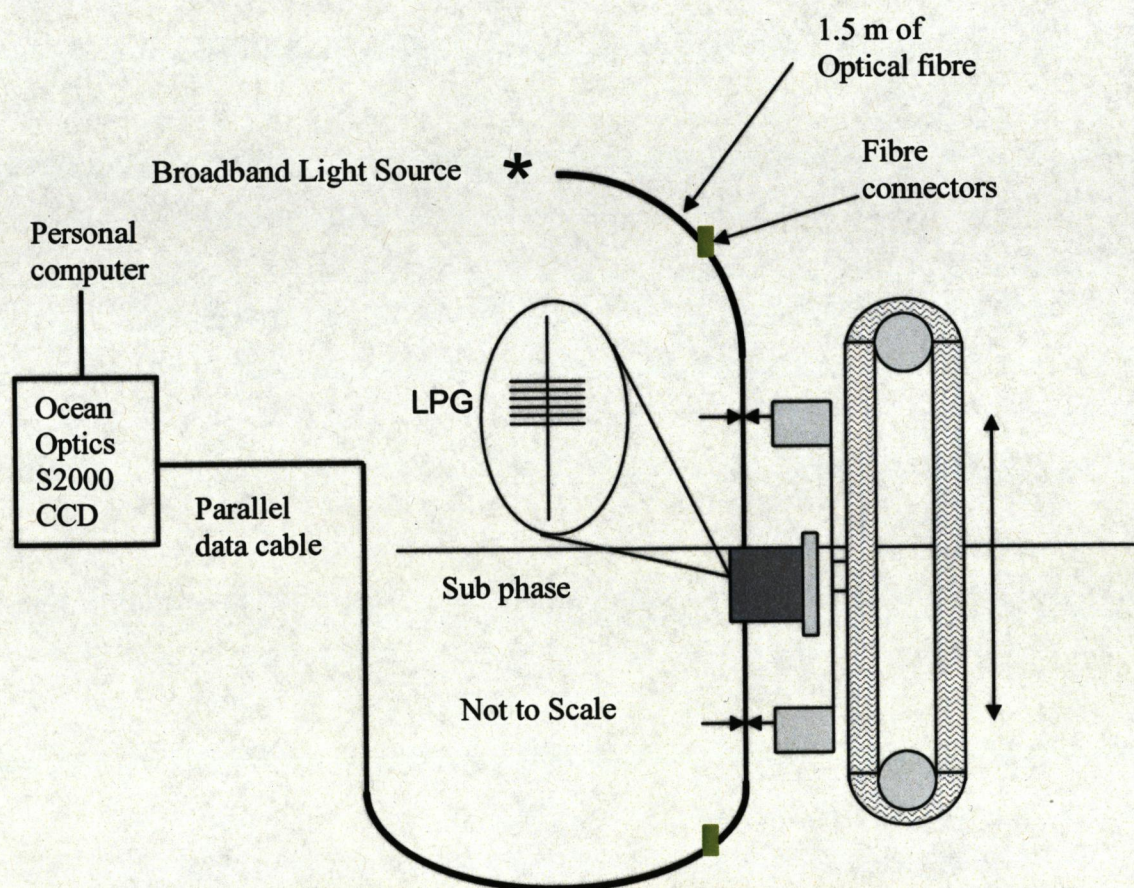


Figure 5.10: General experimental configuration of the major components of the interrogation system used to monitor the transmission spectrum of the LPG.

The optical fibre with the LPG is mounted on the dipper mechanism by tightening the PTFE washers on the modified bracket, ensuring the position of the LPG is known. The fibre ends were fusion spliced between the source and the detector in order to ensure additional spectral features were not introduced, which can occur when using fibre optic connectors.

All depositions within this thesis were carried out on LPGs fabricated in Fibrecore's Boron-Germanium co-doped photosensitive fibre (PS750) with a cut-off wavelength of approximately 650nm; the detail of the fabrication method is described in Chapter Four. The use of this fibre ensures that the spectral features of interest lie within the spectral response of silicon detectors, allowing the use of the low cost Ocean Optics CCD coupled spectrometer to monitor the response to deposition.

Using the LB technique it is possible to fabricate sensors with channel dropping response centered at any wavelength and thus taking full advantage of the thickness control offered by Langmuir-Blodgett deposition.

5.5 Chapter Summary

The LB technique was used to deposit nano-structured overlays onto optical fibres and so a brief discussion was presented of the technique. The modification to allow the deposition onto cylindrical substrates was given. The technique offers high resolution control over the overlay thickness and is therefore ideal for fabrication of devices based upon planar waveguides. The technique offers the potential to deposit non-centrosymmetric films which offers the possibility to exploit non-linear optical processes. The practical details for monitoring the deposition onto optical fibre were discussed.

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Chapter 6 Characterisation of Fibre Optic Long Period Gratings with Nano-structured Overlays

6.1 Introduction

As discussed in Chapter Two, the transmission spectrum of an LPG written in an optical fibre contains a series of attenuation bands centered at discrete wavelengths. The locations of the central wavelengths of these bands are dependant on the local environment surrounding the LPG. LPGs have been explored recently for sensing applications and in some instances they exhibit higher sensitivity than conventional sensors. They offer a number of advantages which make them an exciting and interesting class of sensor. They exhibit sensitivity to a greater number of parameters than FBGs and since each attenuation band displays a distinct sensitivity to different measurands, they offer the potential for multi-parameter sensing applications using a single LPG¹. The exact form of the response of the attenuation bands to these various measurands can be controlled by the choice of period and fibre composition.

Although LPGs are sensitive to a number of parameters, the investigation presented here is concerned with the sensitivity to the surrounding refractive index (SRI). LPGs exhibit sensitivity to the RI of the medium surrounding the cladding^{2, 3, 4, 5, 6, 7, 8, 9, 10, 11} due to the dependence of the phase matching condition on the effective refractive index of the cladding modes, as discussed in Chapter Two. The central wavelengths and the minimum transmission of the LPG attenuation bands have also been shown to exhibit a dependence on the thickness and the refractive index of overlays, even when the overlay has a refractive index higher than silica^{12, 13}.

In this Chapter, nano-structured overlays are deposited onto the optical fibre containing an LPG and the response to SRI is shown to be modified by the presence of the overlay.

The results are presented and compared with a theoretical model and are in good agreement. The overlays were deposited using the LB technique and the central wavelengths of the attenuation bands were monitored during deposition.

The characterisation of the response of an LPG to SRI was achieved by total immersion of the LPG into an aqueous solution of ethylene glycol (EG). The RI of the solution was varied in the range of 1.33 to 1.43 by altering the concentration of EG. The investigation was extended by immersing the LPG in Cargille oils of RI ranging from 1.400 to 1.500 at room temperature. Varying thicknesses of overlay were then deposited onto the LPG. These devices were investigated to determine the effect of the thickness of overlay on the response to SRI. The optimum overlay thickness that provided the maximum sensitivity to SRI was investigated and was compared to a theoretical model based on the transfer matrix method^{12, 14, 15}.

Finally, a chemical concentration sensor with enhanced sensitivity was demonstrated by the deposition of an overlay of the optimum thickness. Since the coupling wavelength of the core and cladding modes were shown to be particularly sensitive to changes in the optical thickness of the overlay, an enhanced response was expected to SRI.

The LPGs used in this investigation were fabricated in B-Ge co-doped photosensitive optical silica single mode fibre (PS750) with a cut-off wavelength of 650nm. The LPG fabrication was, as described in Chapter Four, written using UV irradiation through an amplitude mask. The LPGs had a length of 40mm and periodicity of 400 μ m.

6.2 LPG's with Langmuir-Blodgett Overlays

6.2.1 Introduction

The majority of studies of the refractive index sensitivity of LPGs have concentrated on the bulk immersion of the LPG into a solution. However, more recently the Langmuir-Blodgett (LB) and Electrostatic Self-Assembly (ESA) nano-deposition techniques have been used to facilitate the investigation of the presence of a nano-structured overlay upon the response. The use of active and passive overlays can contribute to the sensitivity exhibited by the attenuation bands since the cladding modes can interact with the overlay. Therefore the optical properties of the overlays can influence the cladding mode properties and thus the exhibited sensitivity of the LPG. The LB¹⁶ and ESA techniques allow high resolution control over the thickness of the overlay. This is typically of the order of a few nanometers per layer, and thus the optimisation of the interaction can be achieved.

ESA is a technique for the deposition of multilayered overlays of magnetic¹⁷, electrically conductive¹⁸, and nonlinear optical materials¹⁹ onto a substrate. The growth of the overlay is achieved by electrostatic attraction between opposite charges of alternate monolayers²⁰. The monolayers deposited can consist of different molecules at each deposition, thus providing greater versatility than the LB technique.

However the LB technique was exploited here as a result of the continuing interaction with the Nanomaterials Research Group (Cranfield University) which specializes in LB overlay synthesis and provided expertise in this technique. In addition, the LB overlay material can be engineered to be optically non-linear, with a large non-linear coefficient, and also to be chemically sensitive. The technique was readily adapted to deposit onto optical fibres, as described in Chapter Five.

During LB deposition, the thickness of the overlay is governed by the length of the molecule used and by the number of layers deposited. Typically, the molecules suitable for deposition by this technique are long chain fatty acids and alcohols. Using the experimental configuration detailed in Section 6.2.2, the LPG's transmission spectrum was monitored following the deposition of each bi-molecular layer, and thus the effect of the increase of the thickness of the overlay by a few nanometers was readily observed. Presented here are the results observed when the transmission spectrum of an LPG was monitored during the construction of a multilayer device of thickness 1200nm.

6.2.2 Experiment

The material deposited onto the LPG was tricosenoic acid $[\text{CH}_2=\text{CH}(\text{CH}_2)_{20}\text{CO}_2\text{H}]$ ²¹ which has a molecular length of 2.8 nm and refractive index of 1.57 measured at 633 nm^{16,33}. It was spread from dilute chloroform (HPLC) solutions (0.1 mg ml⁻¹) onto the ultra pure water (conductivity 18MΩ.cm) subphase. The material was spread in one compartment of a Nima Technology LB trough (model 2410A) and left for 20 minutes at *ca.* 20°C. This gave sufficient time for the chloroform to evaporate and leave a monolayer of tricosenoic acid on the surface of the pure water. The layer was then compressed at 0.5 cm² s⁻¹ (*ca.* 0.1% s⁻¹ of total surface area). Deposition was achieved at a surface pressure of 30mN m⁻¹ and a rate of 10 mm min⁻¹. The fibre containing the LPG was orientated such that dipping direction was aligned with the long axis of the fibre and was alternately raised and lowered through the floating monolayer at the air-water interface. A modified attachment to secure an optical fibre rather than a glass slide was used, as detailed in Section 5.4. Repeated passes through the overlay gave a Y-type structure in which the amphiphilic molecules packed head-to-head and tail-to-tail. The product of the number of passes and the thickness of each molecular layer, approximately 3 nm, allowed the calculation of the thickness of the overlay deposited onto the LPG. Figure 6.1 shows a schematic of an LPG with an overlay deposited on the surface of the optical fibre..

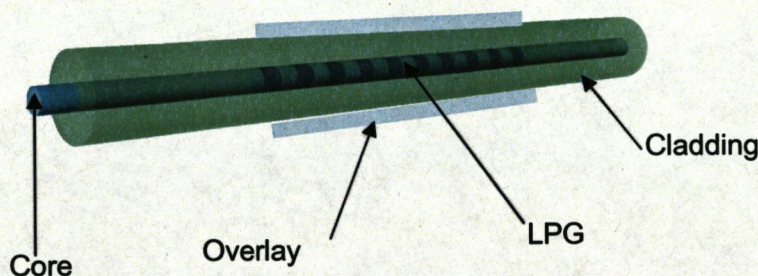


Figure 6.1: Schematic showing the LB overlay deposited on a section of optical fibre containing an LPG.

The interrogation system used for real time monitoring of the transmission spectrum was as described in Chapter Four. It consisted of an Ocean Optics fibre coupled CCD spectrometer (S2000), and a fan cooled Tungsten halogen broadband light source (HL-2000) with a spectral range of approximately 220-1100nm and a power stability of $\pm 0.05\%$. The spectrometer was connected to the optical fibre containing the LPG using fibre connectors (FC 301) and index matching gel to avoid extra spectral features being introduced due to cavity formation between the ferrules in the uniter housing. The resolution of the spectrometer was 0.7nm but the effective resolution was improved further to 0.3nm by using an algorithm to fit a polynomial function to the spectrum. The demodulation of the LPG signal was achieved using a zero-crossing detection algorithm written in-house as a Labview subroutine. This algorithm detected where the derivative of the spectrum changed sign and thus determined the local minimum in the attenuation band signal. The Labview program allowed the transmission spectrum to be logged at a rate of up to 1Hz.

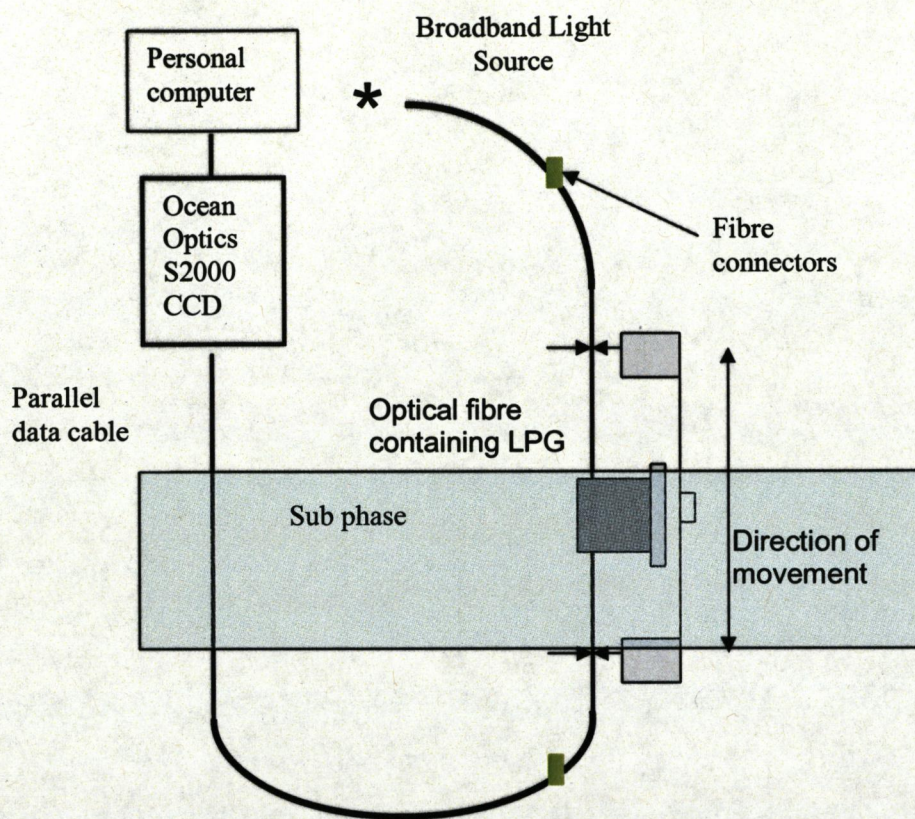


Figure 6.2: General experimental configuration of the major components of the interrogation system used to monitor the transmission spectrum of the LPG during deposition.

6.2.3 Results

Figure 6.3 shows the shift in wavelength of the central wavelengths of all observable attenuation bands as a function of increasing overlay thickness. All Attenuation bands within the transmission spectrum exhibited the same form of response. However the magnitude of the wavelength shift and the reduction in the amplitude of the attenuation bands were seen to be dependant on the order of the corresponding cladding mode. The response of the second and third band was observed to be less than the bands' at longer wavelengths.

The sixth attenuation band was the most sensitive to changes in the thickness of the overlay. This difference in sensitivity exhibited by the different attenuation bands can be used to isolate the response to different measurands and facilitate multi-parameter sensing with a single LPG device¹.

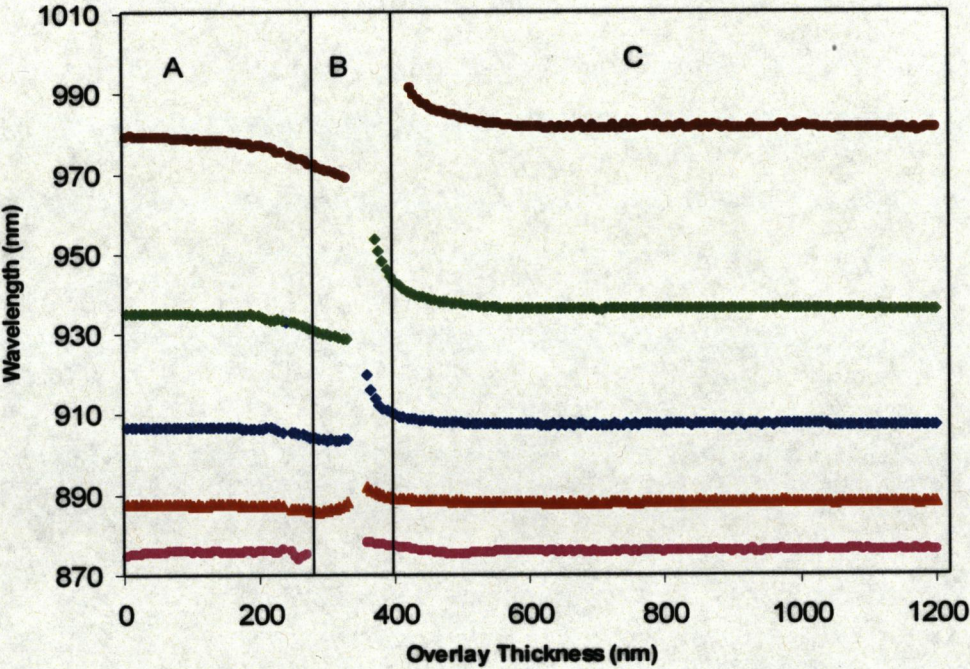


Figure 6.3: Experimentally observed shift in central wavelengths plotted as a function of the thickness of the overlay. Shown are the results for the 2nd (■), 3rd (▲), 4th (◆), 5th (◆) and the 6th (■) attenuation bands observed within the transmission spectrum.

Figure 6.4 illustrates the response of the transmission at the central wavelength of the attenuation bands as a function of increasing overlay thickness. In the most sensitive region, corresponding to an overlay thickness in the range 300nm to 350nm, there is a rapid reduction in the extinction of the attenuation bands.

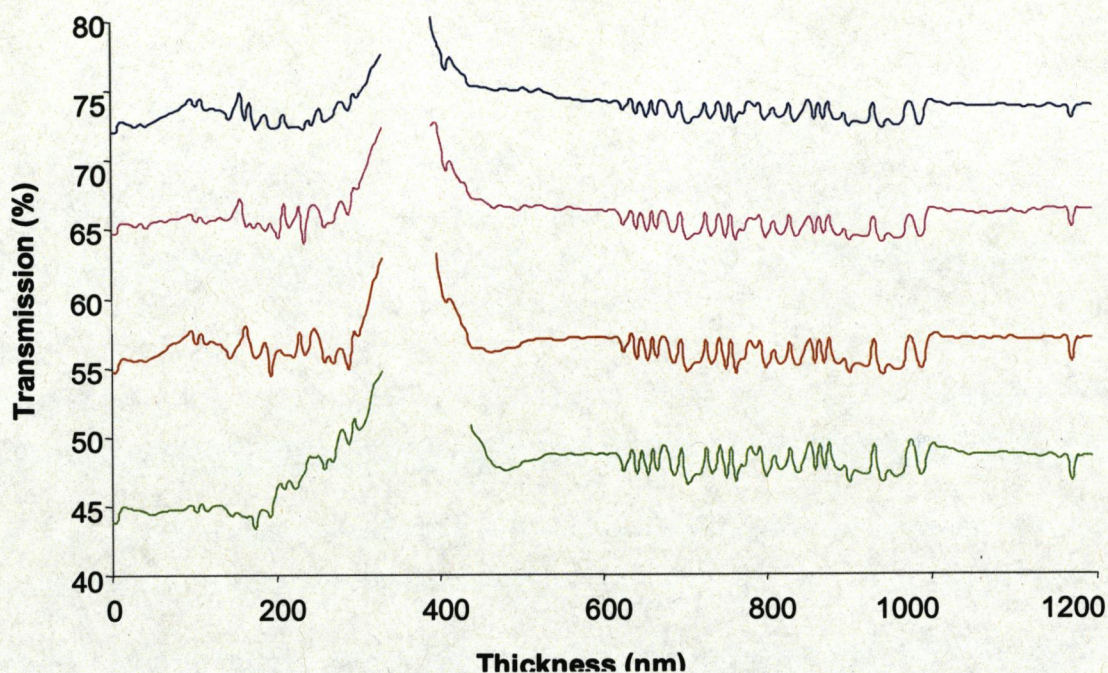


Figure 6.4: A plot of the depth of the attenuation bands as a function of overlay thickness. Shown are the results observed the, 3rd (-), 4th (-), 5th (-) and the 6th (-) attenuation bands observed within the transmission spectrum.

Figure 6.5 shows the shift in central wavelengths of the attenuation bands corresponding to the fifth and sixth attenuation bands plotted as a function of the overlay thickness. To aid discussion the graph can be observed as having three distinct regions, characterised by the different response of the transmission spectrum. As the overlay thickness was increased to approximately 300nm, both the attenuation bands exhibit blue shifts of up to 6nm, shown in Region A.

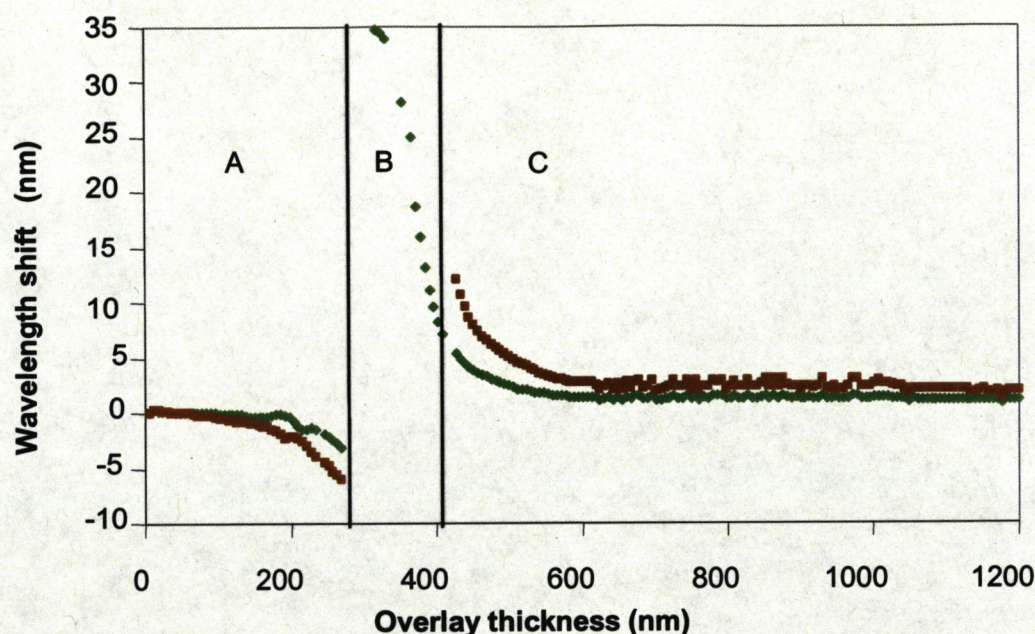


Figure 6.5: Experimentally observed shift in central wavelengths plotted as a function of the thickness of the overlay. Shown are the results observed for the 5th (♦) and the 6th (♦) attenuation bands within the transmission spectrum.

As the overlay thickness was increased further the response shown in region B was observed, where the amplitude of the attenuation bands reduced to zero. Thus no coupling wavelengths could be recorded. For an overlay thickness of greater than 350nm for the sixth band and 400nm for the fifth band, the bands reappeared at wavelengths higher than originally observed in the absence of any overlays. Subsequent increase in overlay thickness caused the central wavelengths of the attenuation bands to return towards their original value. Similar effects at longer wavelengths have been observed for overlays deposited using the ESA technique, where larger wavelength shifts, up to 40nm were observed for overlay thickness 25nm^{22, 23}.

Figure 6.6 shows the movement of the central wavelength of the attenuation band corresponding to coupling to the fifth cladding mode at the most sensitive region. A small increase in overlay thickness was observed to cause a large wavelength shift. The central

wavelength reappeared at longer wavelengths after only a few nanometers of increase in overlay thickness, indicated by the vertical arrow. The Labview subroutine was used to identify the central wavelengths of the cladding modes during their transition and when they reappeared.

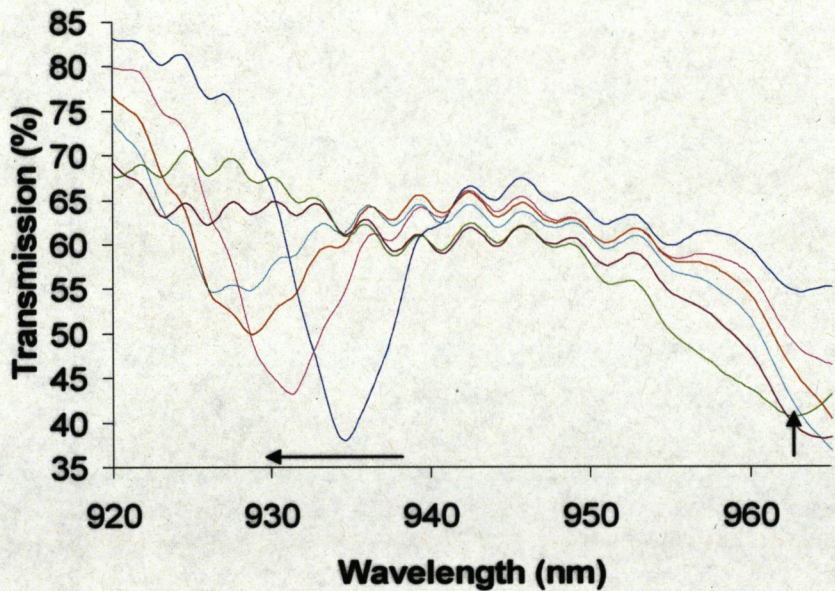


Figure 6.6: The central wavelength of the attenuation band corresponding to coupling to the 5th attenuation band as a function of the thickness of the overlay at 0nm (-), 270nm (-), 310nm (-), 330nm (-), 340nm (-) and 350nm (-). The central wavelength of the band reappeared at higher wavelength.

6.2.4 Discussion

The central wavelengths of the attenuation bands in the transmission spectrum of an LPG exhibit a response to changes in thickness of an overlay material, as has been shown by previous studies¹². A numerical model has been developed at Cranfield University by

Dr. S. W. James, to determine the dependence of the central wavelengths of the attenuation bands on the thickness and the refractive index of the overlay material^{12, 24}. The effective refractive indices of the cladding modes were calculated as a function of wavelength and overlay thickness by considering the cladding and overlay system as a stack of thin films and using the transfer matrix method¹⁴. The effective refractive index of the propagating core mode was calculated by the weakly guiding approximation of Gloge¹⁵. The dispersion of the core and cladding modes calculated in this way was used in the phase matching equation to determine the central wavelengths of the LPG attenuation bands as a function of overlay thickness. The model is broadly in agreement with the experimental results and qualitatively predicts the experimentally observed behaviour.

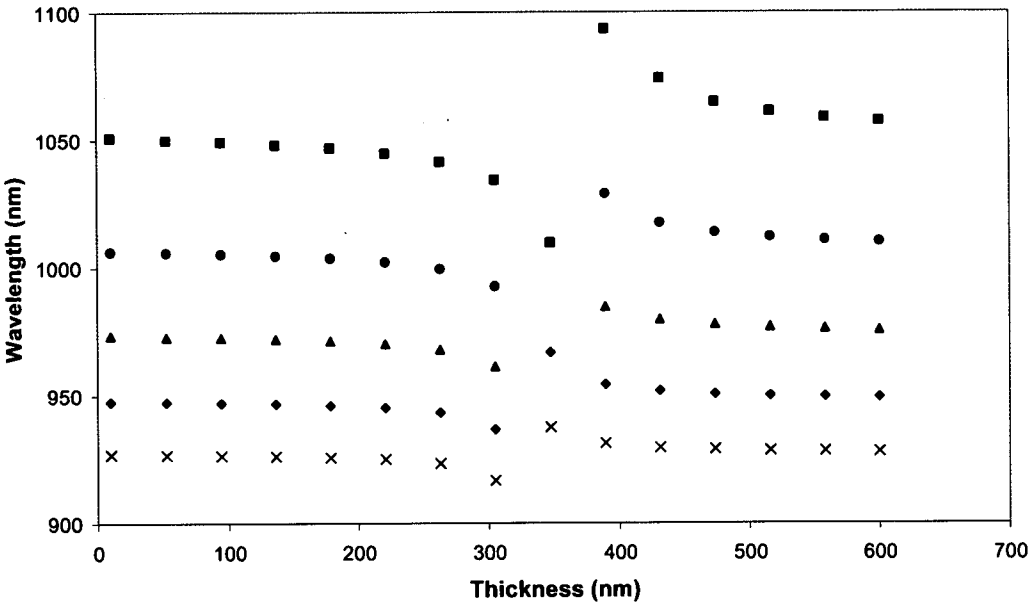


Figure 6.7: Theoretically predicted central wavelengths of the attenuation bands plotted as a function of the thickness of an overlay of RI 1.57. Diagram reproduced from²⁴.

The model approximates that the system can be modeled as a multilayer planar waveguide and ignores the coupling efficiency between core and cladding modes. The coupling efficiency influences the coupling wavelength and depth of the attenuation bands. This therefore introduces a limitation in the model, namely that the maximum wavelength shifts predicted by the model may in fact be higher. Any discrepancies in the

predicted values arise from these approximations and the accuracy of the knowledge of the fibre and overlay material parameters²⁴.

A theoretical prediction of the variation of the wavelength and wavelength shift as a function of overlay thickness is shown in Figure 6.7 and Figure 6.8. The model does not predict the disappearance of the attenuation bands and, if the order of the attenuation bands are ignored, the wavelengths shifts appear to be continuous, as experimentally shown in Figure 6.3. This behaviour has also been analysed using coupled mode theory^{22, 25} and by modeling step index waveguide geometry²⁶. The behaviour is attributed to a reorganization of the cladding modes in region B of Figure 6.5. In this region, the overlay is sufficiently thick to guide the cladding mode with the highest effective index i.e the lowest order cladding mode. This causes a shift in the effective index of each cladding mode towards that of its immediate lower order cladding mode in the absence of the overlay.

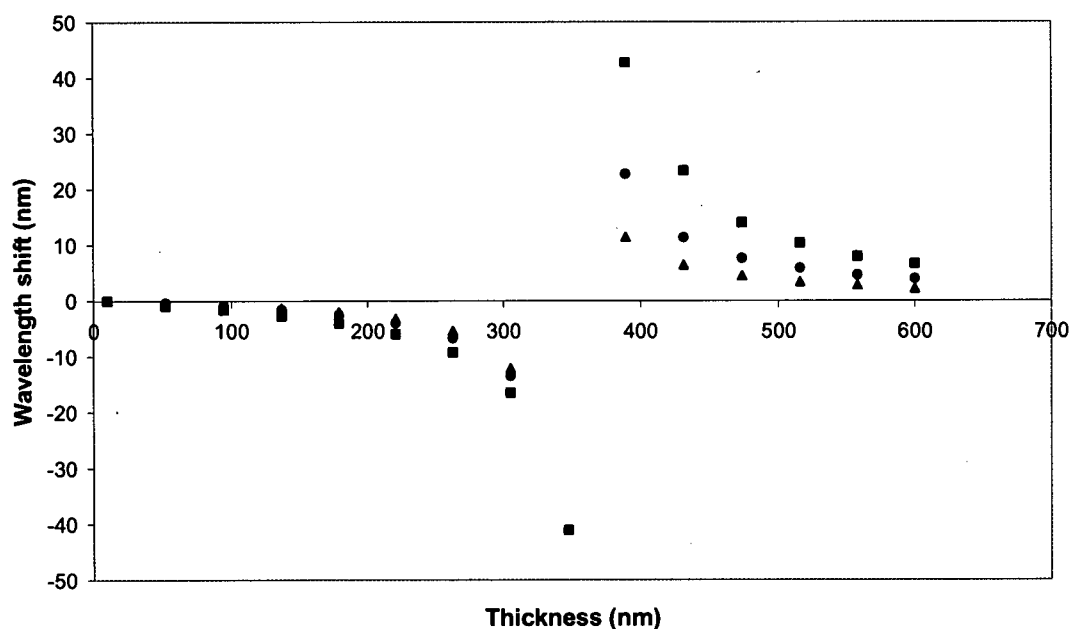


Figure 6.8: Theoretically predicted shift in central wavelengths plotted as a function of the thickness of the overlay, the RI of which was 1.57. Shown are the the results observed for the 4th (▲), the 5th (●) and to the 6th (■)attenuation bands within the transmission spectrum. Diagram reproduced from²⁴.

During this transition to guidance by the overlay, the effective RI of the cladding modes varies rapidly. This is apparent from the rapid reduction of the amplitude to zero and from the blue wavelength shift in central wavelengths of the attenuation bands. Figure 6.9 illustrates the reorganization of the cladding modes during this transition, and it is apparent that further increases in the overlay thickness cause the process to repeat²².

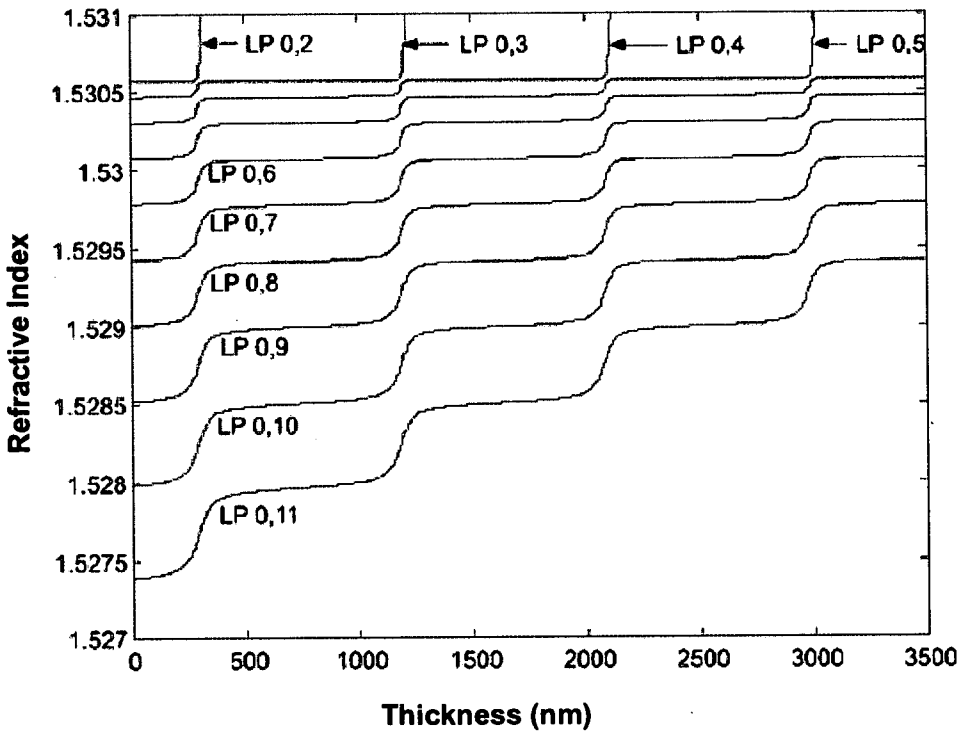


Figure 6.9: Theoretically calculated effective refractive indices of cladding modes as a function of overlay thickness. Shown is the reorganization of the cladding modes of order 1-11 during the transition to guidance of the overlay. Diagram reproduced from²².

The disappearance of the attenuation bands is not predicted by this model unless the overlay displays a complex refractive index where the guiding can take place to higher order cladding modes during the transition. In this case, the attenuation of the cladding modes is predicted to increase, thus reducing the efficiency of the coupling between the

core and the cladding modes. This results in the reduction in the extinction of the attenuation bands as observed experimentally¹³.

The model can be used to predict the response of the central wavelengths' of attenuation bands to the deposition of overlays of differing refractive index, as shown in Figure 6.10.

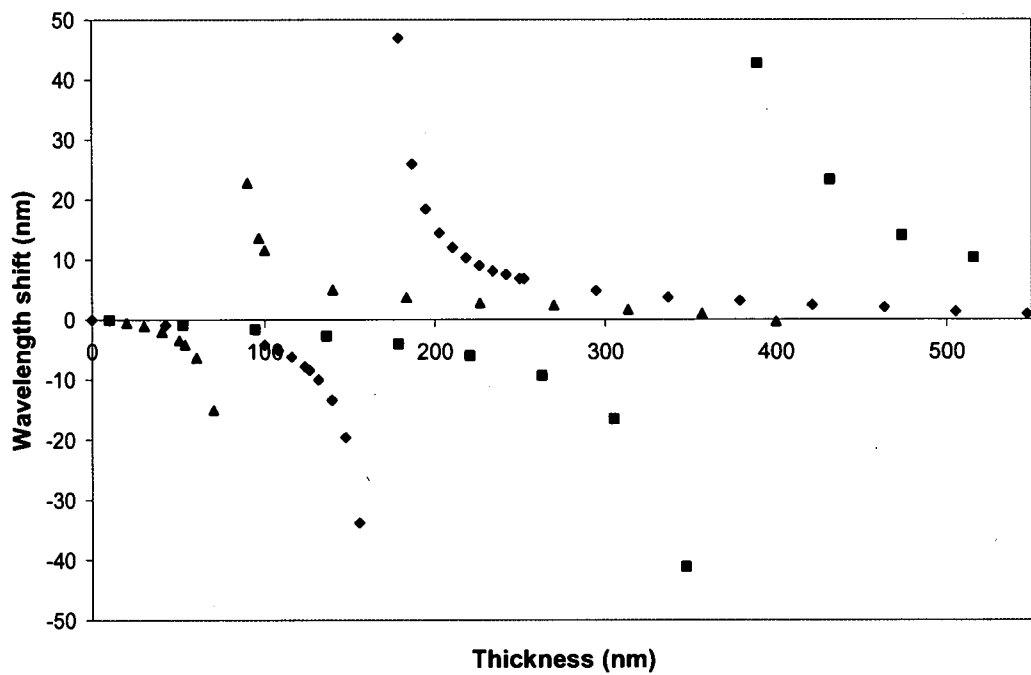


Figure 6.10: Theoretical prediction of the influence on the refractive index of the overlay on the sensitivity of the LPG transmission spectrum to overlay thickness: $\blacktriangle$ refractive index 2, $\blacklozenge$ refractive index 1.7, $\blacksquare$ refractive index 1.57. Diagram reproduced from²⁴.

Figure 6.10 illustrates that, as the index of the overlay material increases, the region of high sensitivity moves to lower overlay thicknesses. This indicates the potential for developing sensors based upon LPGs with overlays whose refractive index modifies in response to an external stimulus.

An area of particular interest is the transition from region A to region C in Figure 6.5 as it offers a large wavelength shift for a small change in the optical thickness of the overlay. If an overlay of the appropriate thickness is deposited, then a sensor could be configured that exhibits a large wavelength shift for a small change in refractive index offering an ultra sensitive sensor, or an optical modulator. If, additionally, the overlay was an active overlay, such that it may undergo refractive index changes on exposure to a particular chemical species, then a highly sensitive chemical sensor could be developed.

A practical consideration when employing sensors based on this technology is the stability of the overlay to exposure to the atmosphere. Hermetically packaged devices that offer resistance to the atmosphere may provide a solution but cause limitations to exposure of the LPG to measurands under investigation. Cross sensitivity to other measurands such as temperature, strain and bending must be minimized by fabricating devices which have suppressed sensitivity to these or are packaged such that they are not exposed to them. The RI of the overlay may also exhibit a thermal dependence and this also must be determined prior to use in any application.

Presented here were the experimental results to show the wavelength shift and the reduction in the amplitude of the attenuation bands in the transmission spectra of an LPG as a function of increasing overlay thickness. This was compared to a theoretical model that predicts the response observed and showed good qualitative agreement.

6.3 Characterisation of the sensitivity of an LPG with Nano-structured overlays to changes in the surrounding refractive index.

6.3.1 Introduction

The dependence of the central wavelengths of the attenuation bands upon the thickness and RI of the overlay has recently been demonstrated experimentally, using overlays deposited by the LB and the ESA techniques^{27, 28}. An LPG is sensitive to SRI due to dependence of the phase matching condition upon the effective RI of the cladding modes, as discussed in Chapter Two. In this section the response of an LPG to SRI with no overlay is compared to the response exhibited by an LPG *with* an overlay. The optimum overlay thickness was investigated by characterising the response to SRI with different thickness of overlays deposited onto the LPG.

6.3.2 Experiment

To characterise the SRI response, an LPG without an overlay was immersed in a series of liquids of differing refractive index. Solutions of ethylene glycol (EG), diluted incrementally with water to reduce the EG concentration at a rate of 10% per addition, were prepared. The RI of an aqueous ethylene glycol solution is known to depend on the concentration^{2,33}. The required concentrations were achieved by using two separate $0.5\mu\text{l} \pm 0.02\mu\text{l}$ syringes to add either distilled water or pure ethylene glycol to several $5\text{ml} \pm 0.001\text{ml}$ volumetric flasks. The liquids chosen to surround the LPG ranged in RI from 1.33 for pure distilled water to 1.43 ± 0.0002 for pure ethylene glycol. The liquids were stored and used in a thermal enclosure, which took the form of a polystyrene box.

Once mixed, the solution of a particular concentration was poured into an aluminium receptacle, designed such that the section of optical fibre containing the LPG could be fully immersed in the liquids. The buffer jacket of the optical fibre had been removed along the entire section prior to the fabrication of the LPG. The receptacle took the form of a well, 5mm deep, 50mm long and 20mm wide, formed in an aluminium block. V-grooves ran longitudinally into the well from either end of the block and were used to support the fibre either side of the LPG. The configuration of the receptacle is shown in Figure 6.11.

The LPG was positioned in the centre section of the well. Leakage of the solution was prevented by sealing the ends of the V grooves using a wax, supplied by Radio Spares. The wax served to secure the fibre under tension, ensuring that the strain experienced by the LPG was constant throughout the experiment. It also prevented any bend induced distortion of the transmission spectrum²⁹. The characterisation was carried out at room temperature and to avoid temperature variations around the LPG, the liquid receptacle was insulated in a thermal enclosure. The temperature was actively controlled using a thermo-electric cooler.

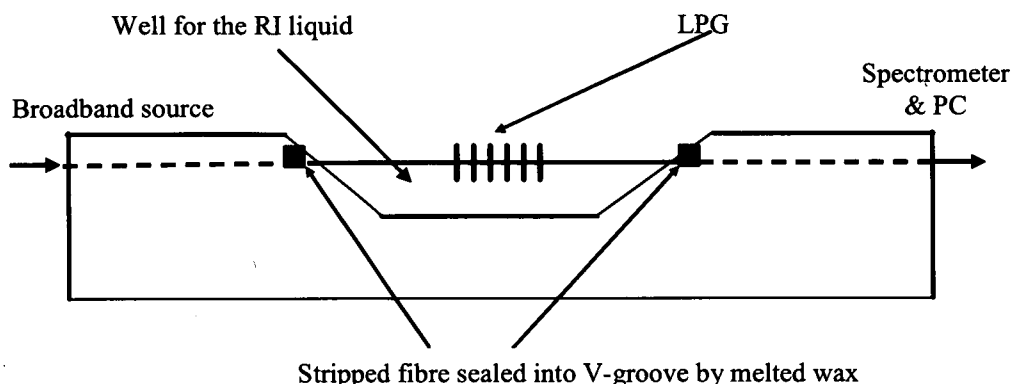


Figure 6.11: *The configuration of the receptacle used (side profile) to hold the LPG immersed under liquids of varying RI.*

The LPG and receptacle were cleaned thoroughly between exposure to each liquid concentration using Isopropyl alcohol (IPA) and lens tissue to remove remaining traces of the liquid in which the fibre was previously immersed. The LPG was then dried with a clean lens tissue and also allowed to air dry for fifteen minutes. The LPG and the receptacle were cleaned and dried repeatedly until the attenuation bands returned to their original central wavelengths, as this indicated no impurities were present. The response exhibited by the LPGs were monitored using the system described in Section 6.2.2

The transmission spectrum of the LPG was recorded when positioned in the receptacle surrounded by air at 25°C. Subsequent spectra were recorded after the application of each solution to monitor the effect of the different SRI on the central wavelength of the attenuation bands.

6.3.3 Results

The response of an LPG to change in the refractive index of medium surrounding it manifests itself as a change in the central wavelength and in the minimum transmission

value of the attenuation bands, as shown in Figure 6.12. The central wavelength of the attenuation bands within the transmission spectrum experienced a blue shift and a reduction in amplitude as the refractive index of the solution increased from 1.33 to 1.43. The attenuation bands corresponding to coupling to higher order cladding modes displayed higher sensitivity, apparent by the magnification of the response of the sixth and the third attenuation bands, in Figure 6.12.

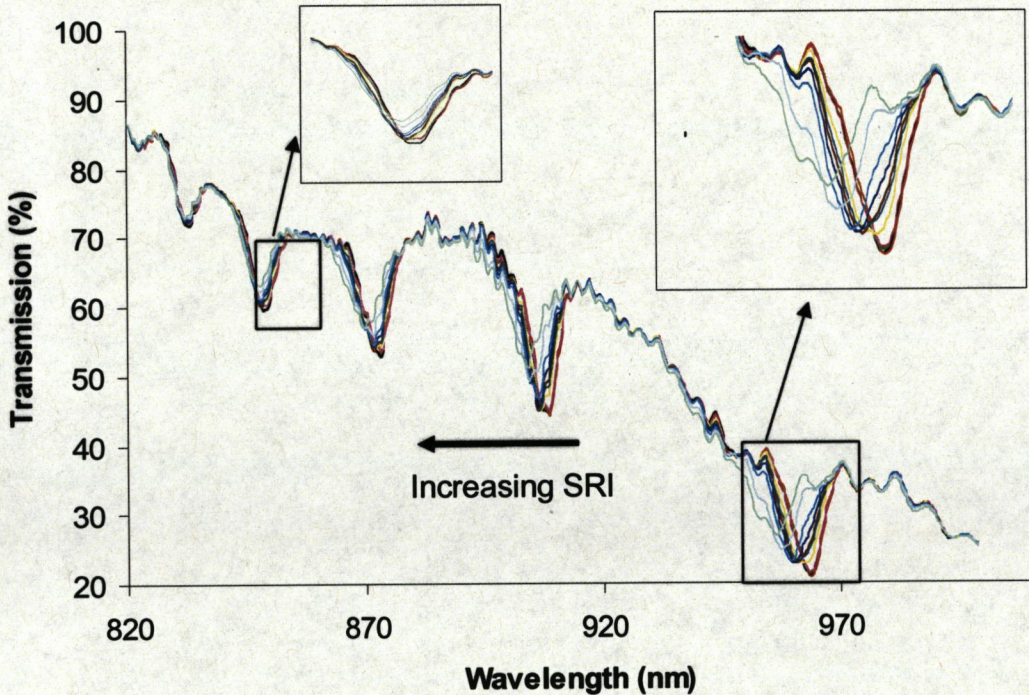


Figure 6.12: A plot to show the movement of the central wavelength of all observed attenuation bands in response to immersion in solution of varying refractive indices ranging from 1.33 to 1.43. The bands were observed to experience a blue shift and reduction in amplitude in response to an increase in RI. Also shown is the transmission spectrum in air, at the longest wavelength for each attenuation band.

Figure 6.13 displays the response of the central wavelength of the sixth attenuation band as a function of SRI. Immersion in ethylene glycol caused a greater blue shift in the attenuation band central wavelength than immersion in distilled water. This is consistent

with previous studies where the attenuation band sensitivity to SRI was observed to be largest when the external RI was approximately equal to the cladding^{12,2}.

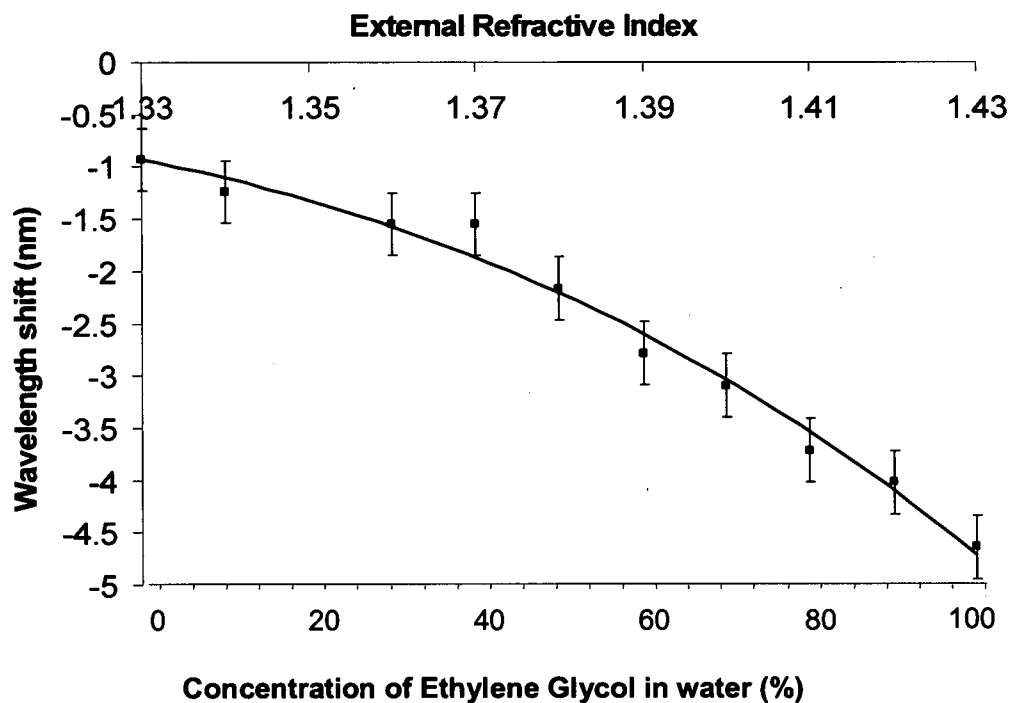


Figure 6.13: Change in central wavelength of the sixth attenuation band in the transmission spectrum of an LPG of period $400\mu\text{m}$ in response to immersion in solutions of differing concentrations of ethylene glycol.

A wavelength shift was observed for each attenuation band in response to a change in refractive index. The sensitivity exhibited by the attenuation band resulting from coupling to the 6th order cladding mode was greater ($4.5\text{nm} \pm 0.3$), than that shown by the coupling to the 3rd order cladding mode ($0.4\text{nm} \pm 0.3$) when immersed in 100% EG. The different sensitivities shown by the bands have been used previously to separate the temperature^{2,30} and RI response³¹. The maximum shift of central wavelengths exhibited by the attenuation bands corresponding to coupling to the 6th, 5th, 4th and the 3rd cladding modes were determined and are presented in the Table 6.1.

Table 6.1: *The maximum measured wavelength shift of attenuation bands induced by immersion in ethylene glycol.*

Attenuation Band λ (nm)	Maximum Wavelength Shift (nm)
963 (6 th)	4.64± 0.3
912 (5 th)	1.8± 0.3
875 (4 th)	0.9± 0.3
851 (3 rd)	0.4± 0.3

6.3.4 Discussion

The characterisation reported here has shown that an increase in the RI of the medium surrounding the LPG brought about a distinct blue shift in the central wavelength of the attenuation bands. The maximum wavelength shift was observed for a solution of 100% ethylene glycol, corresponding to an SRI of 1.43. The sensitivity exhibited by the central wavelength of the attenuation band corresponding to coupling to the sixth cladding was the largest. Good agreement was obtained between previous studies where this configuration was used as a sensor capable of determining the concentration of a solution of ethylene glycol ^{2, 11}.

The phase matching equation can be rearranged to produce Equation 6.1 to determine the wavelength shift an attenuation band would exhibit as the SRI is changed,

$$\frac{d\lambda}{dn_{Ext}} = \frac{d\lambda}{dn_{cl}} \frac{dn_{cl}}{dn_{Ext}}$$

Equation 6.1

where $d\lambda$ is the change in the central wavelength, dn_{Ext} and dn_{cl} are the change in the RI of the external environment and the cladding respectively. A change in n_{Ext} will change the wavelength and, since $dn_{\text{cl}}/dn_{\text{Ext}}$ is unique for each cladding mode, the response that is exhibited by each attenuation band is different¹.

The RI response is appreciable for RI between 1.4 and 1.45 but almost insignificant beyond this, as shown in Figure 3.6 in Chapter Three; thus limiting the range of chemical concentrations that can be measured. As the SRI approaches the RI of the cladding, the effective index of the cladding mode becomes very sensitive to changes in the SRI. Thus the central wavelengths of the attenuation bands also become sensitive. For SRI greater than the RI of the cladding, the cladding does not support guided modes, but leaky modes propagate due to Fresnel reflections at the interface. The effective indices of the leaky modes are insensitive to the SRI. Therefore fibres with higher cladding RI would need to be used to offer an extension to the sensing range, if required

The LPG may be used to provide sensitive measurements of chemical concentrations for liquids in the 1.33-1.43 index range. The response is non linear and for concentrations near 50% the minimum detectable concentration change is $< 1\%$, comparable to conventional Abbe refractometer measurements^{2,11}.

6.3.5 Modification of the observed response of the LPG by overlay deposition

The response of an LPG to the SRI may be modified by the deposition of overlays onto the fibre's cladding. A recent theoretical analysis¹² has shown that the deposition of an overlay, thickness of a few hundred nm, of a material of RI greater than that of the cladding, can change the range of RI's to which the LPG is sensitive. The purpose of the

investigation detailed in the following section is to validate the theoretical model. The technique used to deposit overlays was the Langmuir-Blodgett (LB) technique, reviewed in Chapter Five. The influence of overlays of different thicknesses was assessed.

6.3.6 Experiment

The overlay was deposited and the transmission spectra monitored using the techniques described in Section 6.2.2. The response of LPGs to SRI was investigated using the technique described in Section 6.3.2. The range of SRI was extended by immersing in Cargill series 'A' oils, a series of pre-prepared refractive index liquids in the range 1.400-1.500 (± 0.0002), when measured at 25°C at a wavelength of 589.3³².

6.3.7 Results

The wavelength shift of the attenuation band resulting from coupling to the sixth order cladding mode is shown in Figure 6.14, where it is plotted as a function of increasing SRI. As the RI of the liquids increased from 1.4 to 1.456, the principal effect was a blue shift of the central wavelengths of the attenuation bands, accompanied by a reduction in the minimum transmission value. For indices higher than that of the silica cladding, a red shift was observed when overlays of a few hundred nm were deposited. However, no response was observed in this region for an LPG without an overlay.

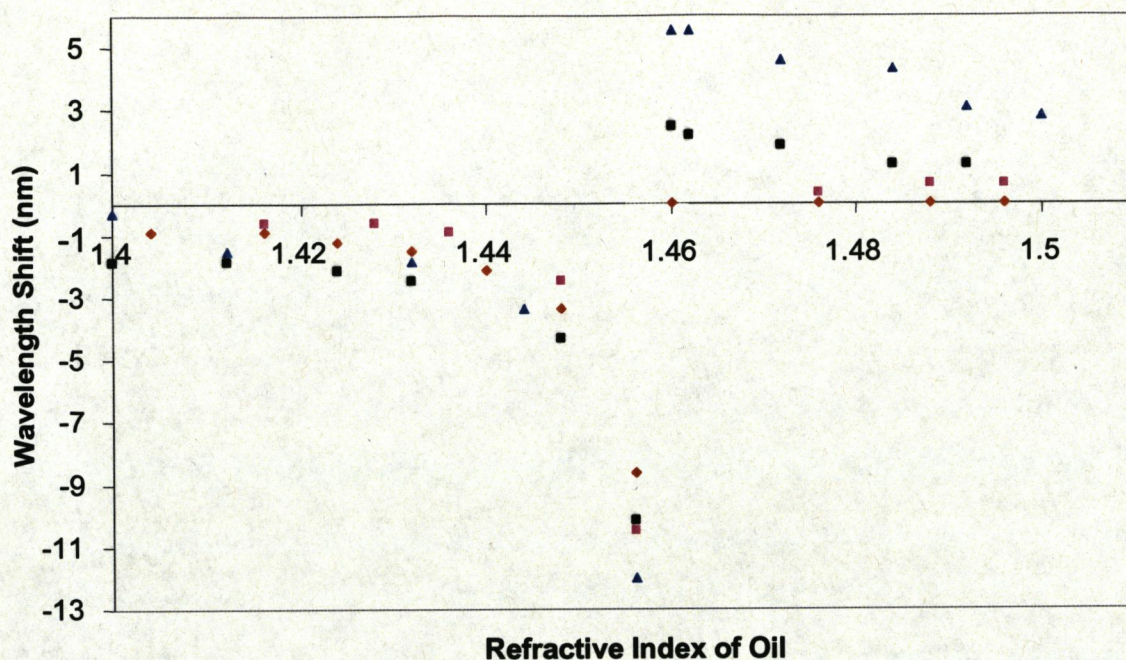


Figure 6.14: Response of the sixth attenuation band to immersion in Cargille oils of differing refractive indices with no overlay(♦), and with overlays of tricosenoic acid, of RI 1.57 and thickness 90nm(■) , 120nm(▲) and 150nm(▣).

The sensitivity to SRI greater than that of the cladding is a new observation and offers advantages such as the potential development of LPG sensors operating over a larger RI range. The sensitivity exhibited by the LPGs was generally greater with increasing thickness of overlay, with the optimum thickness achieved at 120nm.

To illustrate the influence of the overlay upon the performance of the LPG as a chemical concentration sensor, the response of the LPG to immersion in solutions of varying concentrations of ethylene glycol was measured. For the comparison, an overlay of thickness 90nm was deposited onto the LPG. Figure 6.15 compares the sensitivity of the LPG with and without an overlay, by showing the response of the attenuation band resulting from coupling to the sixth order cladding mode. A second order polynomial was fitted to the data obtained from the LPG with no overlay and a fourth order polynomial

was fitted to the data from the LPG with a 90 nm thick overlay of tricosenoic acid, and are shown in the figure as an aid to the eye. The presence of the 90nm thick overlay can be seen to enhance the sensitivity of the LPG. A larger wavelength shift with increasing concentration of EG and concomitant increase in SRI was observed³³. The sensitivity increases at higher concentrations and is approximately double at 100% EG concentration.

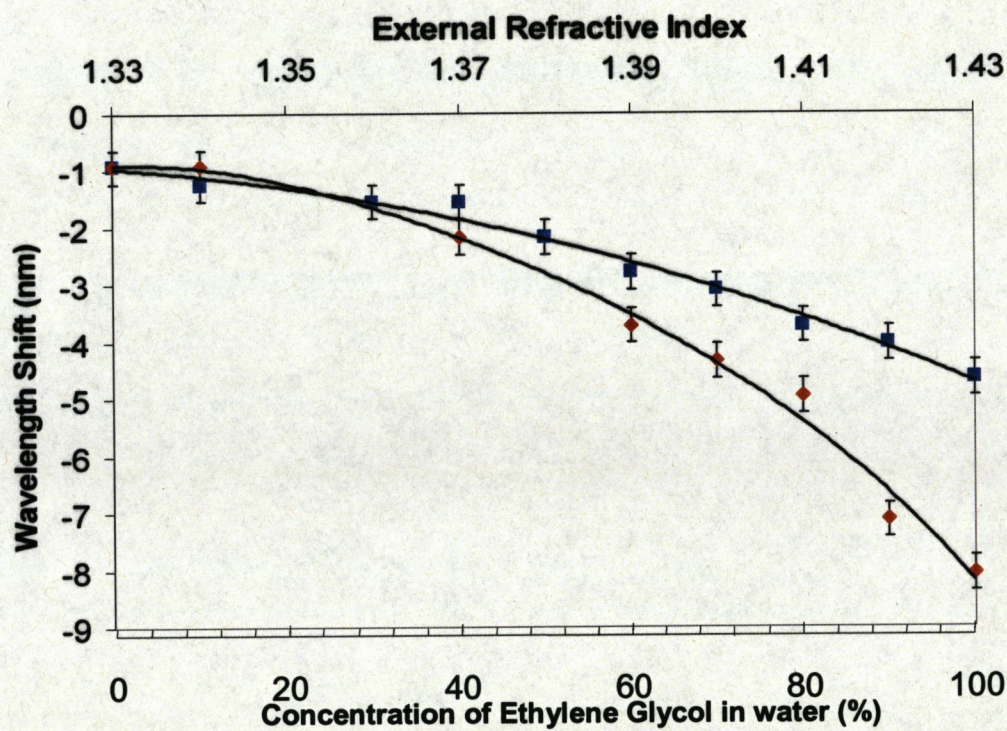


Figure 6.15: Response of the sixth attenuation band corresponding with the transmission spectrum to the change of concentration of ethylene glycol (EG) and refractive index with no overlay (■) and with a 90nm overlay (◆).

6.3.8 Discussion

The experiments detailed here show that the deposition of an overlay of organic material, of thickness of a few 100nm and of RI greater than that of the fibre's cladding, can modify the response of the LPG to external index of refraction. In the absence of an overlay, the LPG was shown to exhibit SRI sensitivity over the range 1.4-1.46, which manifests itself as a blue shift in the central wavelengths of the attenuation bands. For refractive index greater than 1.46 the central wavelengths returned to their original value and the LPG showed no response to further increase in RI. However, in the presence of the overlay, the LPG response was enhanced in the RI range 1.4-1.46 by about 25% for an LPG with 120nm thick overlay, as apparent by Figure 6.14. At refractive indices *higher* than 1.46 the LPG exhibited significant red shift in wavelength of approximately 5nm for an LPG with 120nm of overlay compared to no response, without the overlay.

An EG chemical concentration sensor, with almost twice the sensitivity at high concentrations than an LPG without the overlay was demonstrated by the deposition of 90nm of overlay. LPGs have already been shown to offer higher resolution, almost three times than that for an Abbe refractometer at high concentrations of EG¹¹. The proposed sensor could offer an alternative method for concentration measurements than the Abbe refractometer..

This increased sensitivity can be attributed to the effect of the increasing external refractive index of the cladding mode as the overlay is deposited and to the existence of more complex waveguide structure observed when the refractive index surrounding the fibre is higher than the cladding ¹². A model has been developed at Cranfield University by Dr. S. W. James to predict the response of the central wavelengths of the attenuation bands to the external refractive index, both with and without an overlay and has been discussed in Section 6.2.4. There is good correlation with the theoretical prediction and the experimental results, notably an enhancement of sensitivity at refractive indices lower than silica and the development of a response at SRI higher than the cladding refractive index, as shown in Figure 6.16.

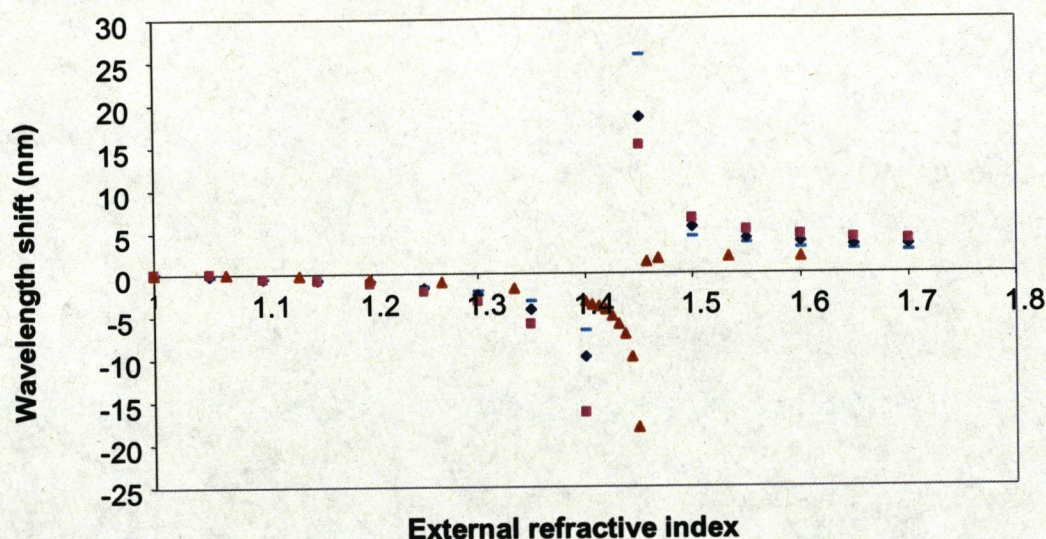


Figure 6.16: Theoretical prediction of the response of a LPG to external refractive index at varying overlay thickness; ($\blacktriangle$) 0nm, (-) 75nm, ($\blacklozenge$) 100nm and ($\blacksquare$) 125nm. The overlay RI is assumed to be 1.57. Diagram reproduced from²⁴.

6.4 Chapter Summary

The deposition of overlays of RI higher than silica onto optical fibres containing LPGs was investigated. The attenuation bands within the transmission spectra of LPGs were observed to be influenced by the thickness and RI of the overlays. The response of an LPG to SRI was characterised, with and without a nanostructured overlay. The optimum overlay thickness was determined to be 120nm and an overlay of thickness 90nm was used to demonstrate an enhanced chemical concentration sensor. In the experiments discussed here, the optical properties of the material used to form the overlay were insensitive to the Cargille oils. However it is possible to engineer LB overlays that change their refractive index in response to, for example exposure to a particular chemical species³⁴, offering the prospect for developing a new chemical sensing capability. The use of non-centrosymmetric overlay deposition may be used for optical modulation, as will be discussed in Chapter Eight.

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Chapter 7 Characterisation of Concatenated Fibre Optic Long Period Gratings with Nano-structured Overlays

7.1 Introduction

A concatenated LPG consists of multiple, identical LPGs written in series in the core of an optical fibre, a full discussion of which was given in Section 3.6. The presence of two LPGs, fabricated in-series in the fibre results in the formation of a Mach-Zehnder interferometer. Light coupled into the cladding by the first LPG is coupled back into the core by the second LPG. The different optical path lengths experienced by the light propagating in the core of the fibre and light propagating in the cladding produces an interference pattern within the LPGs' attenuation bands. This provides fine scale detail within the relatively broad bandwidth of the attenuation bands, potentially allowing the development of sensors with greater resolution.

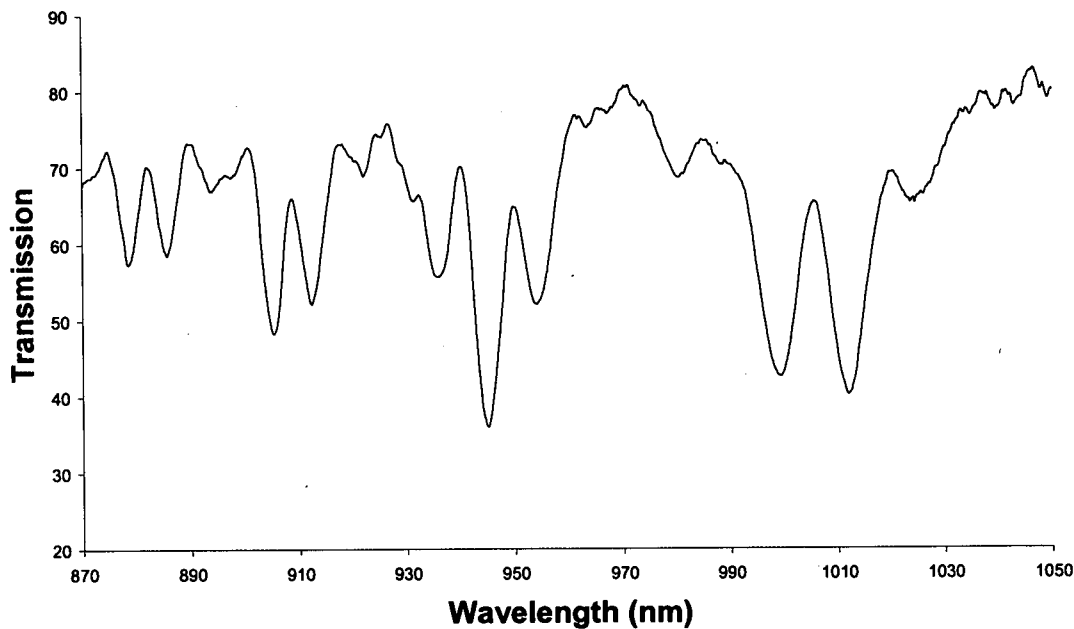


Figure 7.1: Transmission spectrum of two concatenated LPGs each of length 10mm and of period $400\mu\text{m}$, separated by 10mm, written through UV irradiation at 266nm using an amplitude mask. The fibre was photosensitive with a cut-off wavelength of 650 nm recorded in air.

In this Chapter the response of concatenated LPGs to the deposition of nano-structured overlays on the fibre cladding is investigated. The overlays are deposited using the Langmuir-Blodgett (LB) technique^{1,2} rather than the electro-static self assembly (ESA) techniques recently reported^{3,4,5} or the Covalent self-assembly method, where monolayers are formed by reaction at the active surface site to form covalent bonds⁶. The LB technique facilitates the deposition of overlays with high degree of order at room temperature and pressure. The material used to build the overlays in all the configurations investigated is tricosenoic acid in a Y-type or centrosymmetric form, as described in Chapter Five⁷.

Several configurations are considered. Concatenated LPGs of various lengths and differing separations are investigated, with the overlays deposited along the entire length of the device or along only the length of fibre separating the LPGs as shown in Figure 7.2.

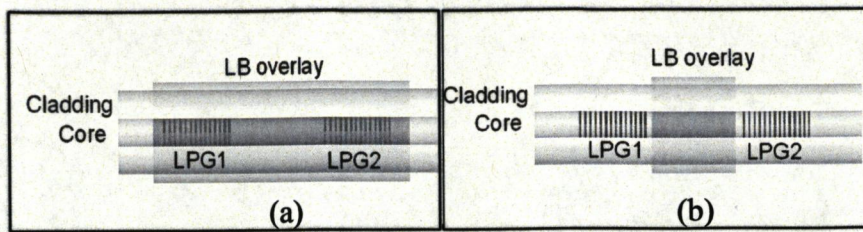


Figure 7.2: *LB deposition on concatenated LPG, (a) over entire section of fibre containing the gratings and (b) over the separation length only. Diagram reproduced from [8].*

Deposition on the entire device results in the refractive index and the thickness of overlay influencing both the fringes and the attenuation band envelope. In the second configuration, shown in Figure 7.2 (b), the deposited overlay only affects the interference fringes. The dependence of the central wavelengths of attenuation bands upon overlay properties has been modelled^{3, 9, 10, 11, 12, 13} and has also been experimentally investigated^{11, 14, 13}. The interference fringes within the attenuation bands are expected to display similar behaviour, with the fringes exhibiting a change in phase in response to variation in overlay thickness, and a reduction in visibility for thicknesses where the extinction of the attenuation bands has been previously shown to reduce.

All devices investigated consisted of two LPGs of near identical characteristics fabricated in-series within a single optical fibre, since the coupling strengths and lengths of the individual LPGs influence the fringe pattern¹⁵. The physical separation of the two LPGs was varied to control the properties of the interference pattern produced since the channel spacing between the fringes is dependent on this¹⁶. The overall length of the device was dictated by practical limitations imposed on the length of optical fibre upon which the LB overlay could be deposited, using the current experimental arrangement. All LPGs were fabricated in a photosensitive fibre of cut off wavelength 670 nm, with period 400 μm using the method described in Chapter Four.

7.2 Concatenated LPGs with Langmuir-Blodgett overlays

7.2.1 Introduction

A series of concatenated LPG devices were fabricated and overlays deposited using the LB technique. The introduction of interference fringes within the transmission spectrum due to the presence of the two in-series LPGs was observed and the evolution of the fringes was monitored whilst increasing overlay thickness. All experimental techniques were as described in Chapter Six, with the exception of a number of enhancements to the deposition technique, which improved the control of the deposition length, described in Section 7.2.2.

7.2.2 Experiment

Previous reports have discussed deposition of LB overlays onto the entire length of a single LPG^{11,14,17}. For the investigation discussed here, where the LB overlay was to be deposited only on the length of fibre separating the two LPGs, depicted in Figure 7.2 (b), greater control over deposition was necessary.

Since the LPGs are not visible to the naked eye, to aid the definition of the length of fibre over which the LB overlay was to be deposited, a second optical fibre was secured parallel to the optical fibre containing the concatenated LPGs with visible markings to show the location of the start and end of each LPG in the device. The markers corresponded since the start of the first LPG was aligned at the point where the buffer jacket was removed during fabrication and was therefore known. This scalar guide to the different regions within the device enabled accurate control over the movement of the dipper via the Labview program.

During set-up, the dipper mechanism and attached optical fibres were lowered into the subphase, the vertical height of the dipper was observed when the visible markings to indicate the end of the first LPG reached the meniscus of the water. The height was noted again when the meniscus was at the markings indicating the start of the second

LPG. Since the deposition of subsequent layers could not occur until the meniscus had reversed, the scaler markings included an additional 2mm at either end of deposition length to allow for this^{18,19}. This was especially critical where the deposition length was as low as 5mm, as inaccuracies could be introduced. These heights were entered into the Labview program that controlled the movement of the dipper.

The reference optical fibre was cleaned thoroughly and was sufficiently far away from the optical fibre under investigation to ensure the meniscus during deposition was unaffected.

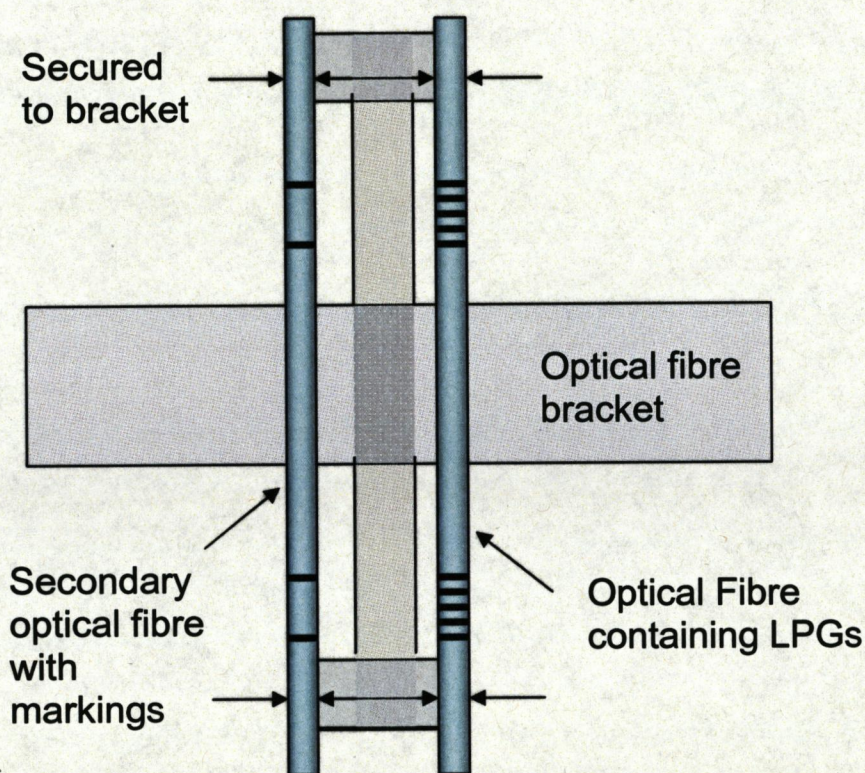


Figure 7.3: Schematic to show how a second optical fibre with visible markings was placed parallel to fibre containing concatenated LPGs to aid deposition accuracy.

The evolution of the transmission spectrum during deposition was monitored using a broadband optical source and CCD spectrometer, as described in Chapter Four. The optical fibre containing the two LPGs was fusion spliced between the source and the detector using a Fujikara40S fusion splicer with an average loss of 0.03dB. This was to avoid extra spectral features being introduced that might occur when connectors are

used. The use of connectors can result in the formation of a cavity between the polished fibre ends in the unit housing

7.2.3 Results

A concatenated LPG device was fabricated with LPGs of length 10 mm, separated by 10 mm. The transmission spectrum obtained prior to deposition is shown in Figure 7.4, where the interference fringes within the attenuation band corresponding to coupling to the sixth order cladding mode are shown. Three interference fringes were observed and these were monitored during deposition of the overlays. The number of interference fringes observed was directly dependent on the parameters chosen for the device, in particular on the optical path difference experienced by the modes propagating within the cladding and within the core. This is a consequence of the difference in their effective refractive indices. The sensitivity to external refractive index has been shown to be greater for attenuation bands at longer wavelengths²⁰ and thus it was the fringes within these attenuation bands that were monitored.

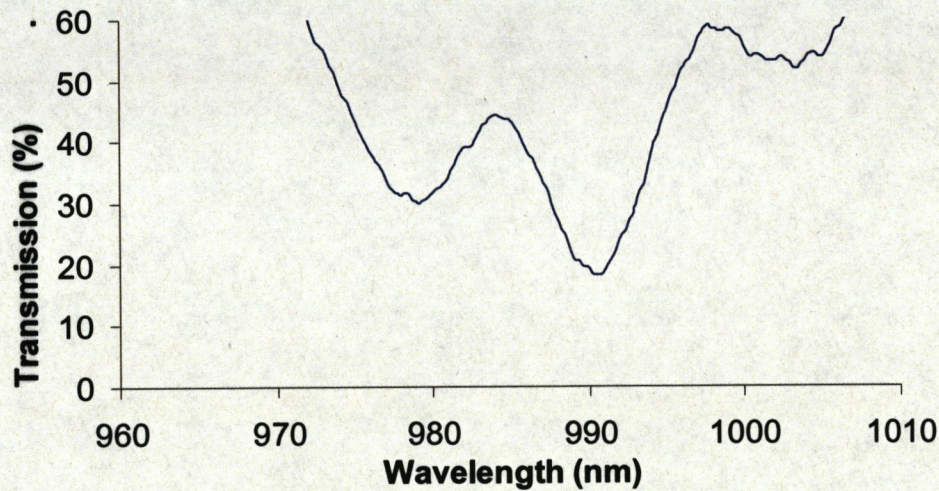


Figure 7.4: Transmission spectrum of two concatenated LPGs each of length 10mm and of period 400 μ m, separated by 10mm, recorded in air. Shown are interference fringes within the sixth attenuation band which were monitored during deposition.

For the configuration shown in Figure 7.2 (a), in which the overlay was deposited on the entire length of the device, the evolution of the interference fringes as a function of the thickness of the overlay is illustrated in Figures 7.5 (a) and 7.5 (b). Figure 7.5 (a) shows a phase change experienced by the fringes, apparent by the blue shift in central wavelengths followed by their visibility reducing to zero. The fringes exhibit the highest phase shift, and therefore highest sensitivity, for an overlay thickness of approximately 350 nm, just prior to the fringe visibility reducing to zero. A change in the central wavelength and amplitude of the attenuation band envelope was noted, in accordance with the results reported¹¹.

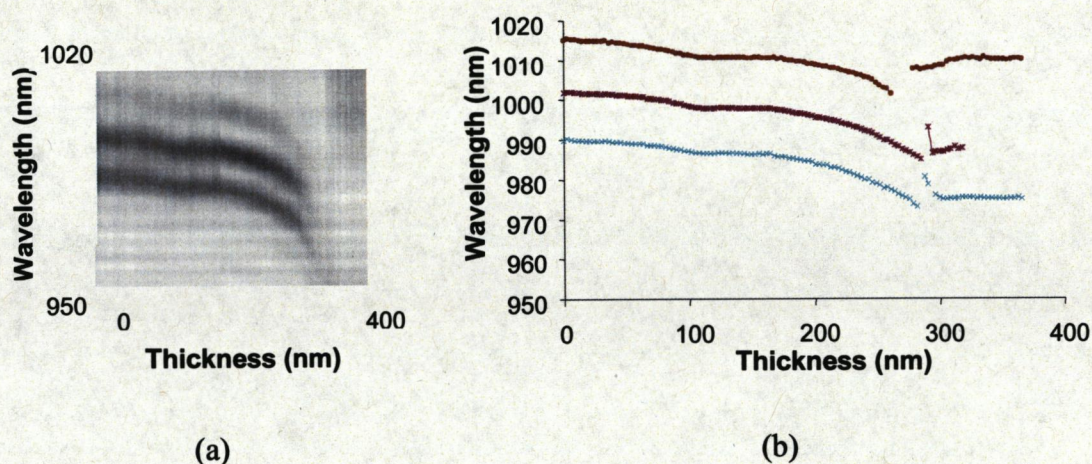


Figure 7.5: (a) Response of the Mach-Zehnder interference fringes to an increase in overlay thickness when deposited over the entire length of the concatenated LPGs. The greyscale represents the transmission, where white corresponds to 100% transmission with no interference fringe. (b), a plot to trace the minima of fringes, (●), (*) and (x), as a function of increasing overlay thickness.

The device was cleaned thoroughly using Isopropanol and lens tissue prior to subsequent deposition, carried out on the length of fibre separating the two concatenated LPGs. Figure 7.6 (a) shows the evolution of the interference fringes as the thickness of the overlay was increased. The changes in the wavelengths of the minima of the interference fringes are shown in Figure 7.6 (b). The fringes exhibit a blue wavelength shift as expected and disappear at the overlay thickness of the order

shown in previous studies to cause the amplitudes of attenuation bands to decrease to zero¹¹. As discussed in Chapter Six, in this region, the attenuation of the cladding modes increases significantly and thus they cannot propagate to the second LPG resulting in the concomitant disappearance of interference fringes. However, the attenuation bands corresponding to coupling to the cladding modes remain fixed, as the overlay was not deposited on the LPGs.

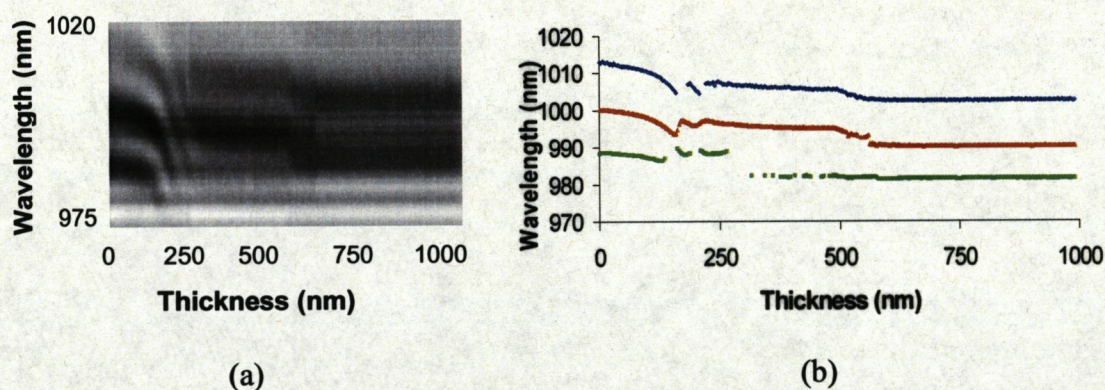


Figure 7.6: (a) Response of Mach-Zehnder interference fringes to increase in overlay thickness when deposited on section of fibre separating the concatenated LPGs. The greyscale represents the transmission, where white corresponds to 100% transmission with no interference fringes. (b) a plot to trace the minima of the fringes, (♦), (▲) and (■), as a function of increasing overlay thickness.

These observations can be qualitatively described as follows. The modification of the effective index of the cladding modes by the deposition of the overlay on the separation length only had the effect of modifying the optical path difference between the core and cladding modes. This resulted in a phase change in the interference pattern, while the envelope provided by the attenuation band remained fixed. The fringes could not be observed to reappear with increasing deposition as would be expected. This may have been due to subsequent deposition being unsuccessful due to impurities causing the overlay thickness not to increase with deposition.

A second device was fabricated with an increased separation and thus an increased number of fringes. The greater interaction between the cladding mode and the overlay

would be expected to have a larger effect on the sensitivity of the phase of the fringes to the properties of the overlay. The two configurations described in Figure 7.2 (a) and (b) were considered again. The device contained two LPGs, each of length 10mm, but separated by 20mm. The transmission spectrum obtained prior to deposition is shown in Figure 7.7, where the interference fringes within the sixth cladding mode are shown. The spectrum was observed to contain an increased number of interference fringes as apparent by the greater path length between the LPGs.

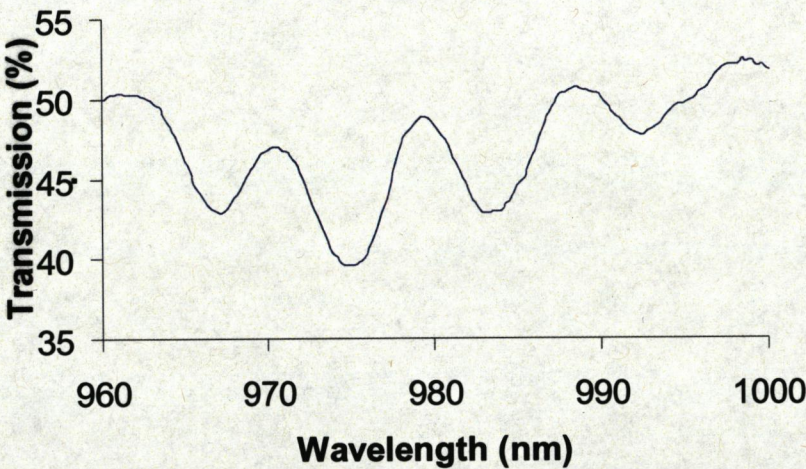


Figure 7.7: Transmission spectrum of concatenated LPGs, of length 10mm and period $400\mu\text{m}$, separated by 20mm, recorded in air. Shown are interference fringes within the sixth attenuation band which were monitored during deposition, greater in number than those observed in Figure 7.4.

For the deposition of the overlay on the entire length of the device, the evolution of the interference fringes is shown in Figure 7.8 (a). The fringes initially exhibit a blue shift with little change in visibility. As the phase of the fringes becomes more sensitive to overlay thickness, the visibility decreases rapidly. Further increase in overlay thickness allows the fringes to reappear at longer wavelengths, and subsequently blue shift to their original wavelengths. The fringes exhibit the highest wavelength shifts, and therefore sensitivity, at an overlay thickness of 300 nm, just prior to the fringe visibility reducing to zero. The movements of fringes were observed to be faster than a device with a separation length of 10mm. This is evident by the larger wavelength shift of the minima of the fringes and the overlay thickness

which causes the highest sensitivity, occurring at a thickness approximately 50nm earlier. This can be understood by reference to Equation 7.1 which describes the behaviour of a Mach-Zehnder interferometer²¹.

$$\theta = \Phi + (\beta_{\text{core}} - \beta_{\text{cladd.}}) L$$

Equation 7.1

Where θ is the phase of the interference fringes, Φ is the phase change imposed on the light coupled to the cladding mode by the LPG and β_{core} and β_{cladd} are the propagation constants of core and cladding modes respectively and L is the length of optical fibre separating the LPGs. The phase of the interference pattern is dependent upon a phase difference imposed by the LPG, and upon the optical path difference between the core and cladding modes. A longer path difference allows greater interaction of the overlay

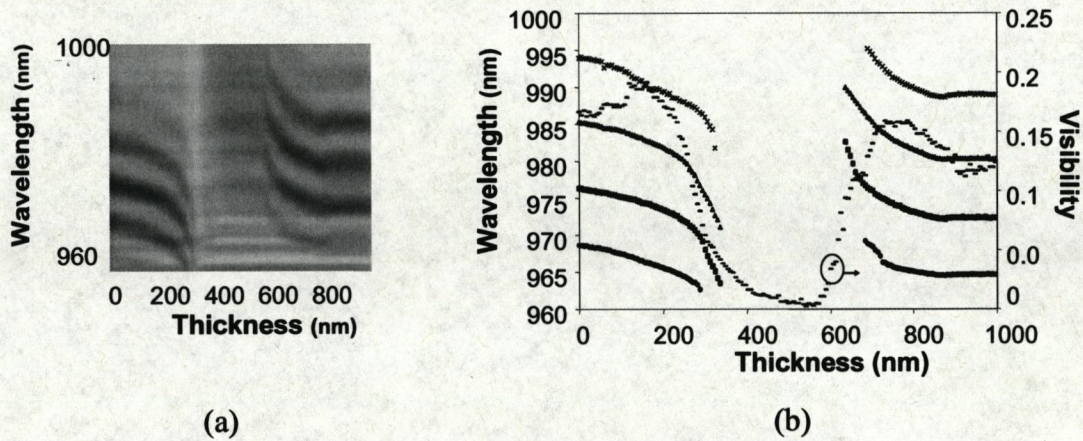


Figure 7.8: (a), Response of Mach-Zehnder interference fringes to increase in overlay thickness when deposited over entire length of the cascaded LPGs. The greyscale represents the transmission, where white corresponds to 100% transmission with no interference fringes. (b), a plot to trace the minima of the fringes, (x), (▲), (■), and (●), while (–) shows the visibility of the fringes as a function of increasing overlay thickness.

The changes in the wavelengths of the minima of the interference fringes are shown in Figure 7.8 (a), along with the corresponding changes in fringe visibility in Figure 7.8 (b). In addition to the change in the phase of the fringes, a change in the central

wavelength and amplitude of the attenuation band was noted, in accordance with the results reported previously¹¹.

After thorough cleaning of the optical fibre containing the device with Isopropanol and lens tissue, the deposition was carried out on the length of fibre separating the LPGs. Figure 7.9 (a) shows the evolution of the interference fringes as the thickness of the overlay was increased. The fringes exhibit a blue shift as expected but as the overlay thickness increases sufficiently to support guided modes, the increased attenuation causes the fringes to disappear. As observed with the device investigated in configuration 7.2 (b) with a separation length of 10mm, (results presented in Figure 7.6), the interference fringes disappeared and reappeared. The modification of the optical path difference caused by the deposition of the overlay resulted in a phase change in the interference pattern, while the envelope provided by the attenuation band remained fixed.

The increase in the interaction length between the cladding mode and the overlay increased the sensitivity of the phase of the fringes to the properties of the overlay. The response of the transmission at the central wavelength of the sixth attenuation band as a function of overlay thickness was determined. The gradient was calculated at the most sensitive region, prior to the extinction of the sixth attenuation band reducing to zero for the results of configuration shown in Figure 7.2 (b). The device with a separation of 20mm exhibited a reduction in the extinction twice as fast than the device with a 10mm separation.

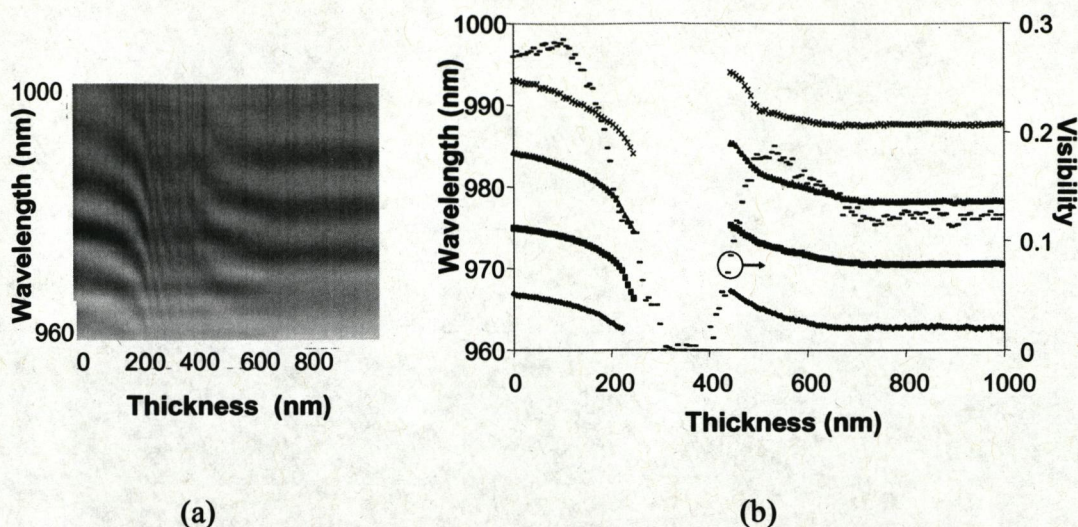


Figure 7.9: (a) Response of Mach-Zehnder interference fringes to increase in overlay thickness when deposited on section of fibre separating the cascaded LPGs. The greyscale represents the transmission, where white corresponds to 100% transmission. (b), a plot to trace the minima of the fringes, (x), (▲), (■), and (◆), while (–) shows the visibility of the fringes as a function of increasing overlay thickness.

Figure 7.9 (b) shows the changes in the wavelengths of the minima of the interference fringes along with the variation in visibility as the overlay is deposited in the central region. The parameters chosen for this device resulted in four interference fringes of high spectral extinction in the sixth order attenuation band, as can be observed by Figure 7.7.

In order to increase the number of fringes and introduce greater resolution still, a third device was, fabricated in the same fibre type, with LPGs of length 5 mm, separated by 30 mm. The two configurations represented in Figure 7.2 (a) and (b) were investigated with this device. In addition a third configuration in which the overlay was deposited on only one LPG was also investigated.

Figure 7.10 shows the spectrum of the device, where the number of interference fringes have increased to six although the fringe visibility was lower, attributed to the

shorter LPG lengths. The increased separation resulted in finer fringes and therefore the effect of deposition would be detectable to a greater degree of accuracy.

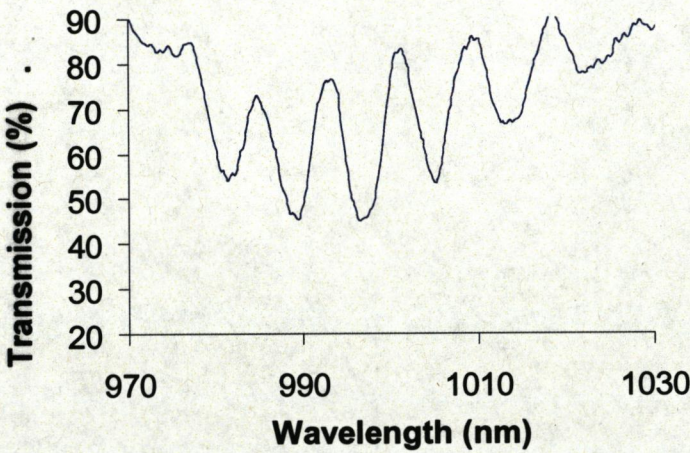


Figure 7.10: Transmission spectrum of concatenated LPGs, each of length 5mm, and of period 400 μ m, separated by 30mm recorded in air. Shown are interference fringes within the sixth attenuation band which were monitored during deposition, greater in number than those observed in Figure 7.4 and 7.7.

As before, the overlays were deposited on the entire length of this third device and the results shown in Figure 7.11 (a) and (b). The interference fringes were observed to exhibit a phase change more sensitive to the overlay thickness. The fringes were not observed to reappear at higher wavelengths. This may be attributed to the low extinction of the attenuation bands making the bands less visible after deposition.

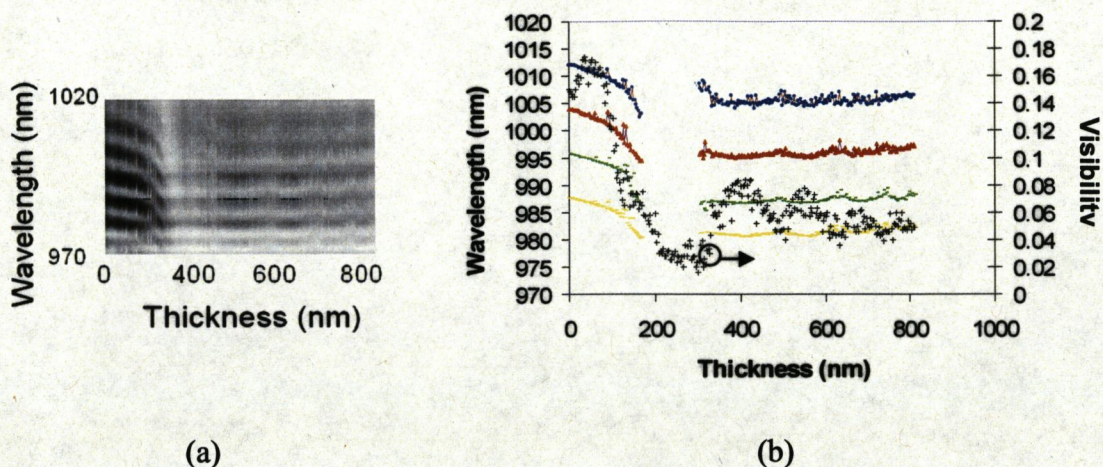


Figure 7.11 (a) Response of Mach-Zehnder interference fringes to increase in overlay thickness when deposited over entire length of the cascaded LPGs. The greyscale represents the transmission, where white corresponds to 100% transmission. (b) a plot to trace the minima of the fringes, (●), (▲), (–) and (– –), while (+) shows the visibility of the fringes as a function of increasing overlay thickness.

The changes in the wavelengths of the minima of some of the interference fringes are shown in Figure 7.11 (b), along with the corresponding changes in fringe visibility. They were observed to exhibit the highest wavelength shifts, and therefore sensitivity, at an overlay thickness of 200 nm, just prior to the fringe visibility reducing to zero. The rate of change was faster as apparent by the reduction in the extinction to zero for a smaller overlay thickness.

The deposition was subsequently carried out on the length of fibre separating the two concatenated LPGs, producing the results shown in Figure 7.12, where the evolution of the interference fringes can be observed. The fringes experienced a blue shift and a reduction in the amplitude of the fringes before reducing to zero. They then reappeared with further deposition of the overlay. The attenuation band envelope would be expected to remain stable as no deposition occurs on the LPGs but due to

the small LPG length compared to other devices investigated, the attenuation band could not be observed.

The gradient was calculated at the most sensitive region, prior to the extinction of the sixth attenuation band reducing to zero for the results of configuration shown in Figure 7.2 (b). The device with a separation of 30mm exhibited a reduction in the extinction faster than the device with a 20mm separation.

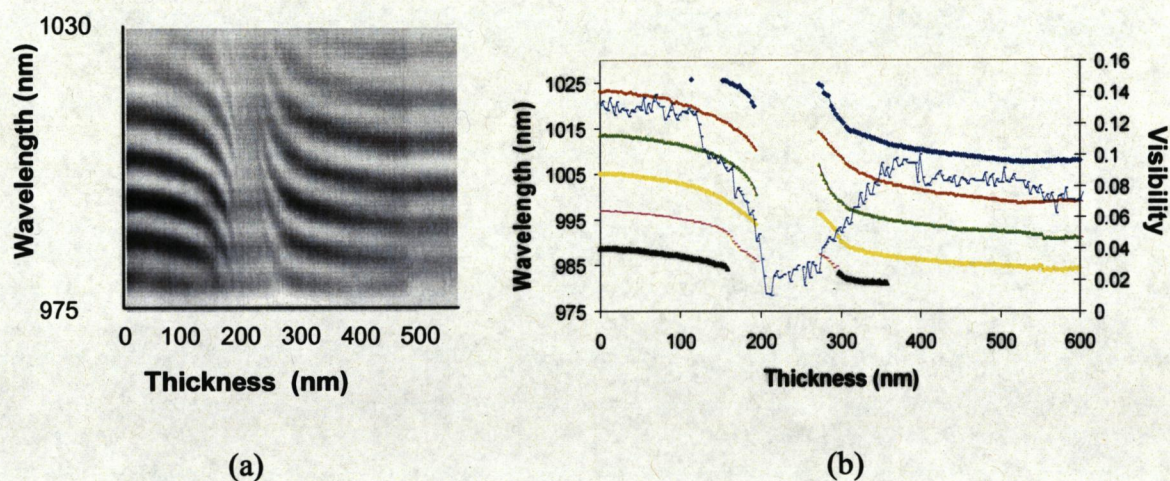


Figure 7.12 (a) Response of Mach-Zehnder interference fringes to increase in overlay thickness when deposited on section of fibre separating the cascaded LPGs. The greyscale represents the transmission, where white corresponds to 100% transmission. (b) a plot to trace the minima of the fringes, (■) (■) (■), (▲), (—) and (▲), while (—) shows the visibility of the fringes as a function of increasing overlay thickness.

A third configuration was also investigated where the overlay was deposited on only one of the two LPGs. Since the overlay was not deposited on the length of fibre separating the LPGs, the fringes would not be expected to experience any phase shift. The deposition on the single LPG was expected to cause the fringes to disappear briefly when the overlay was thick enough to support modes; thus further increasing the attenuation within the cladding modes. The fringes should then reappear once the

thickness of the overlay is increased further. Figures 7.13 (a) and (b) show the behaviour exhibited.

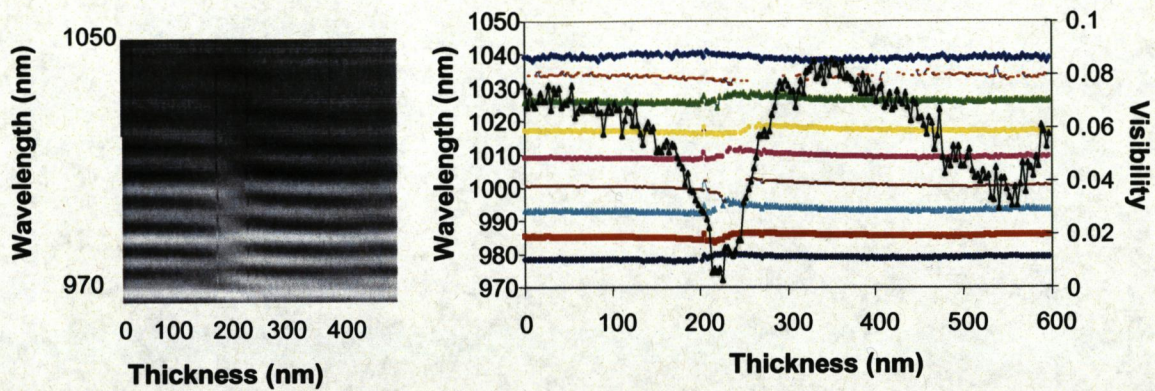


Figure 7.13: (a) Response of Mach-Zehnder interference fringes to increase in overlay thickness when deposited on one of the cascaded LPG only. The greyscale represents the transmission, where white corresponds to 100% transmission. (b) Traces the minima of multiple interference fringes, while (▲) shows the visibility of the fringes as a function of film thickness.

The fringes did not display any sensitivity to the deposition, but were observed to disappear briefly due to the overlay providing increased attenuation in the cladding modes prior to reappearing.

7.2.4 Discussion

The fine structure introduced into the attenuation bands of an LPG by the formation of a Mach-Zehnder interferometer between two LPGs in-series indicates the potential for development of high resolution fibre sensors. The variation in wavelength of the interference fringes as a function of overlay thickness was demonstrated.

The transmission spectrum of concatenated LPGs is described by the following expressions 7.1 to 7.4 inclusive²¹.

$$I = I_{core} + \alpha I_{cladd^i} - 2\sqrt{\alpha I_{core} I_{cladd^i}} \cos \theta \quad \text{Equation 7.2}$$

$$\theta = \Phi + (\beta_{core} - \beta_{cladd^i})L \quad \text{Equation 7.1}$$

$$\Phi = 2 \tan^{-1} \left(\frac{\Delta\beta}{2s} \tan sd \right) - \Delta\beta d \quad \text{Equation 7.3}$$

$$\Delta\beta = \beta_{core} - \beta_{cladd^i} - \frac{2\pi}{\lambda} \quad \text{Equation 7.4}$$

Where I_{core} and I_{cladd^i} , β_{core} and β_{cladd^i} are the intensities and propagation constants respectively of the core and cladding modes, α is the attenuation of the cladding mode, d is the grating length, L is the separation of the LPGs, and s depends upon the coupling coefficient, κ and the detuning, $\Delta\beta$, $s^2 = \kappa \kappa^* + \Delta\beta/2$. The phase of the interference pattern is dependent upon a phase difference imposed by the LPG, and upon the optical path difference between the core and cladding modes, the first and second terms on the RHS of Equation 7.1, respectively.

This was demonstrated in Section 7.2.2 by varying the physical separation of the LPGs, L , and investigating two configurations shown in Figure 7.2 (a) and (b). It was demonstrated that increasing the path length resulted in a greater number of fringes and therefore a greater interaction of the overlay with the cladding modes. This caused the fringes to display greater sensitivity at the same thickness of overlay for devices with longer separation lengths. The fringes reduced to zero at just 200nm thick overlay for a 30mm path length compared to approximately 350nm overlay thickness required for a device with 10mm path length when deposition was carried on the entire device.

It was also shown, by investigating the two configurations described in Figure 7.2 (a) and 7.2 (b), that the magnitude of phase change was larger for the first configuration. This was due to the properties of the overlay influencing both the LPGs and the optical path difference between the cladding and core modes.

The results obtained for configuration 7.2 (a) show the phase of the fringes changed as a function of thickness and in addition to this a change in the central wavelength and amplitude of the attenuation band envelope was recorded, in agreement with the results reported in [11]. The fringes reappeared with further deposition, before experiencing a blue shift and returning to their original wavelengths. The magnitude of the phase change was greater, as evidenced by the larger wavelength shift of the minima of the interference pattern. Depositing on the entire length of the devices resulted in contribution to the phase change from both terms on the RHS of Equation 7.1.

The investigation into configuration 7.2 (b) showed a smaller phase change with increasing overlay thickness which was accompanied by a decrease in visibility. As expected, the phase of the fringes changed as a function of film thickness, whilst the envelope of the broad attenuation band remained unchanged. The reduction of the amplitude of the fringes to zero with additional overlay deposition was followed by the fringes reappearing, the phase showing high sensitivity at first before becoming constant.

The smaller change in the phase of the fringes in this configuration can be attributed to contribution of only one term on the RHS of Equation 7.1. In both configurations, the larger the value of L , the greater the phase change that results, due to greater contribution by this term in Equation 7.1.

Theoretical predictions of the variation of wavelength as a function of overlay thickness have been discussed in Chapter Six. These models use coupled mode theory^{3,9} and step index waveguide geometry²² to demonstrate the behaviour of LPGs. The models can be used to predict the response of attenuation bands' central wavelength to the deposition of overlays. The behaviour of concatenated LPGs response to overlays has not yet been modeled but a theoretical study is in progress. Sensors based on concatenated LPGs have been reported¹⁶ and have displayed an increased sensitivity and higher resolution than those based on single LPGs. The sensitivity to surrounding refractive index has previously been shown to depend on

the phase shift ($\Delta\phi$), of the interferometer and the optimum sensitivity was demonstrated²³.

7.3 Chapter Summary

The fine structure introduced into the attenuation bands of an LPG by the formation of an intrinsic Mach-Zehnder interferometer between concatenated LPGs, offers the prospect for development of high resolution fibre sensors. The influence of nano-structured overlays on the transmission spectrum of concatenated LPGs has been investigated. The phase of the fringes has shown a high sensitivity to overlay thickness. Increased resolution in the transmission spectra was achieved by fabricating devices with longer path length between the two LPGs. Devices with a longer separation length between the LPGs exhibited greater sensitivity as the RI and thickness of the overlay could interact and influence the longer path length.

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Chapter 8 Conclusions and Further Research

8.1 Conclusions

LPGs have been studied extensively over the last ten years and have been proposed for a number of applications. The potential for multi-parameter sensing using a single optical element offers an exciting alternative to conventional sensors. The sensitivity of LPGs to the surrounding refractive index is being exploited to develop chemical sensors, and this area has been invigorated by the recent observation that nano-scale overlays of materials of refractive index higher than that of silica can have a significant influence on the properties of the LPG. Research into the effect of a nano-structured overlay deposited on an optical fibre containing LPG based devices using the LB technique has been presented in this thesis.

In Chapter Six, the effect of a nano-structured overlay on the central wavelength of attenuation bands within the transmission spectrum of an LPG was presented. Increasing the thickness of the overlay caused the central wavelengths of the attenuation bands to initially experience a blue shift, followed by a rapid reduction in the extinction of the band to zero at an overlay thickness of approximately 350nm. This was attributed to the reduced efficiency of the coupling to the cladding modes when the overlay is sufficiently thick to guide the lowest order cladding mode¹. The central wavelengths reappear at a red shifted wavelength and subsequently blue-shift to their original wavelength².

The characterisation of the sensitivity of an LPG to SRI was discussed in Chapter 6. The sensitivity of a chemical concentration sensor was shown to be enhanced by the presence of an overlay of a material of refractive index 1.57 and thickness 90nm. At maximum concentrations of the chemical under investigation, ethylene glycol, the response was observed to increase two-fold, comparable to Abbe refractometer measurements³. The

response of devices with overlays of thickness 90nm, 120nm and 150nm were compared to the response of an LPG with no overlay to immersion in oils of RI in the range 1.400-1.500. It was shown, in addition to the enhancement of their sensitivity to refractive indices lower than that of silica, the LPGs with overlays exhibited a previously unobserved sensitivity to refractive indices greater than that of the silica cladding. The optimum overlay thickness was observed to be of the order 100nm.

A method for further enhancing the response of an LPG based device to SRI was presented in Chapter Seven. The use of concatenated LPGs to introduce fine structure within the attenuation band was exploited. Nano-structured overlays were deposited upon the concatenated LPGs in two separate configurations. In the first, the overlay was deposited along the entire device, while in the second the overlay was deposited only on the length of fibre separating the two LPGs. The evolution of the fringes within the attenuation bands was monitored as the overlay was deposited on the entire length of the device and two effects were observed. Firstly, changes in the central wavelength and extinction of attenuation band's envelope, as described in Chapter Six and secondly, a change in the phase of the interference fringes. This phase change was caused by the overlay induced modification in the effective index of the cladding mode. In the second configuration, in which the overlay was deposited only on the length of fibre separating the two LPGs, only the phase of the fringes and their visibility were effected.

The effect of varying the optical path length between the two LPGs was also investigated. It was shown that the greater the interaction length between the cladding mode and the overlay, the greater the sensitivity exhibited by the device. Devices with optical path lengths of 30mm, exhibited a reduction in the visibility of the fringes to zero with an overlay of thickness 200nm, compared to 350nm thick overlay needed for a 10mm path length. The region in which the visibility of the fringes changes rapidly corresponds to the highest sensitivity for the device.

The experimental results were in good agreement with a theoretical prediction of the behaviour of the transmission spectrum of concatenated LPGs⁴. The enhanced sensitivity

can be attributed to the effect of the increasing external refractive index of the cladding mode as the overlay is deposited and to the existence of more complex waveguide structure observed when the refractive index surrounding the fibre is higher than the cladding². Limitations were imposed on the length of the device by the current experimental arrangement, which limits the length of fibre on which the overlay could be deposited.

LPGs can offer alternatives to conventional sensors for niche sensing applications but further research is still necessary to overcome a number of limitations. A sensing system would need to reduce the cross sensitivity by careful design, since an LPG exhibits a sensory response to many measurands. Several methods to achieve this have been proposed and discussed in Chapter Three. The large number of attenuation bands in the transmission spectrum also limits the number of LPG based devices that can be multiplexed using a broadband source, but this should not be a limiting factor in most applications, however, means for overcoming this are currently under investigation⁵. LPGs are an exciting class of sensor which may be used in conjunction with other sensors such as fibre Bragg gratings to compliment them and achieve the desired responses⁶. Their fabrication via the amplitude mask technique allows the possibility for large scale economical production. Additionally, demodulation using low cost spectrometers is an attractive feature.

The LB technique used for the deposition of the nano-structured overlays proved to be a favourable technique for fabricating the LPG devices presented in this thesis. It allowed the deposition of the overlays with extremely accurate control over film thickness. The technique was readily adapted to facilitate uniform deposition onto cylindrical structures. The LB technique also offers the advantage of facilitating the deposition of non-centrosymmetric films which will be discussed in Section 8.2. There is however a limit in the types of compounds that can be deposited and the stability of the deposited overlays over time is not yet completely understood.

Achievements

8.2 Further work

The deposition of LB overlays on LPGs was shown to modify the response of the central wavelengths of the attenuation bands in the transmission spectrum. The model predicts that higher refractive index overlays cause the region of high sensitivity in the response of the attenuation bands central wavelengths, to move to lower thicknesses⁷. If an overlay of thickness is deposited such that an RI change causes the transition from region A to region C of Figure 6.5, this could offer the potential to develop highly sensitive sensors; since a small RI change would result in a large wavelength shift in the central wavelength of the attenuation bands.

Research can be extended to deposit other types of overlays with intrinsically interesting properties, such as those which exhibit changes in their RI in response to local environment. This offers the possibility to form a sensitive tunable loss filter, an advanced species-specific chemical concentration sensor or a pH sensor. If the overlay material is electro-optic, then a voltage controlled optical modulator could be formed. The Z-type LB deposition onto an optical fibre of a non-centrosymmetric compound which is electro-optic could be investigated to achieve this. This type of deposition is ideally suited for non-linear optics and is formed by aligning the dipoles of the molecules during deposition. The monolayers are transferred onto the substrate on the *upstroke* only and a mechanism to achieve this on an optical fibre has been developed⁸.

Concatenated LPGs can offer greater sensitivity than a single LPG and the response to SRI could also be characterised. The current practical limitation on the deposition length can be overcome in order to fabricate devices with longer path lengths. The response of LPGs with different period to the deposition of nanoscale overlays could also be investigated. The work presented within this study has focused on the response to SRI of LPG devices. This can be extended to include other measurands to develop sensors with