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Experimental observations on the response of 1st and 2nd order fibre optic long period grating coupling bands to the deposition of nanostructured coatings.

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Abstract: The sensitivity of attenuation bands corresponding to the 2nd order coupling to cladding modes by a fibre optic long period grating (LPG) to the deposition of nanostructured coatings is investigated and compared with that of the 1st order coupling. The experimental observations support previously reported theoretical descriptions of LPGs with nanoscale coatings.

OCIS codes: (060.2370) Fiber optics sensors, (050.2770) Gratings, (310.1860) Deposition and fabrication

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1. Introduction

The deposition of nano-structured coatings onto optical fibre long period gratings (LPGs) has received considerable attention [1, 2, 3, 4]. Coatings of thickness of order 100 nm, deposited using the Langmuir Blodgett [1], electrostatic self-assembly [2] and dip coating [3] techniques have been shown to have a significant influence on the transmission spectrum of an LPG. The effect has been analyzed theoretically [5, 6, 7] and has been exploited to modify the response of LPGs to surrounding refractive index [7, 8] and to demonstrate pH [9], copper [10], humidity [11], chloroform [12] and DNA damaging materials [13].

To date, experimental studies of the effect of nanostructured coatings on an LPG's transmission spectrum have been limited to the monitoring of the attenuation bands corresponding to 1st order coupling from the core to cladding modes. This paper reports on the evolution with increasing coating thickness of the attenuation bands corresponding to the 2^{nd} order coupling between core and cladding modes, which may offer new sensing capabilities. The 2^{nd} order coupling attenuation bands are shown to exhibit a high sensitivity to the thickness of the coatings, and onset of the highest sensitivity was observed to occur for thinner coatings than were required for the 1^{st} order coupling attenuation bands. The observation of dual attenuation bands that exhibit sensitivities of different sign to increasing film thickness is also discussed. Further observations on the 1^{st} order coupling attenuation bands are also reported within this paper, which support and extend the observations made by Del Villar *et al* in [14] and in the theoretical treatment of [5].

2. Background

LPGs are a relatively new class of fibre optic device that offer exciting properties for optical sensing applications [15]. An LPG is an UV induced periodic modulation of the refractive index of the core of an optical fibre, with a period in the range 100 μm - 1000 μm. The small grating wave-vector of an LPG allows the phase matching of forward propagating modes of the core and the cladding. Efficient coupling between the core and cladding modes occurs only where there is significant overlap between the electric field profiles of the modes. Thus features corresponding to coupling a from the guided core mode to a discrete set of the $HE_{1,x}$ cladding modes (x=2,4,6..) appear in the transmission spectrum of the fibre. These result from the large attenuation of the cladding modes, which generates a series attenuation bands in the transmission spectrum of a fibre containing an LPG, centered at discrete wavelengths that are governed by the phase matching expression [16].

$$\lambda_{(x)} = \frac{\left(n_{core} - n_{clad(x)}\right)\Lambda}{N} \;;\; x = 2,4,6.. \tag{1}$$

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Where $\lambda_{(x)}$ represents the wavelength at which coupling occurs to the $HE_{1,x}$ modes, n_{core} is the effective refractive index of the mode propagating in the core of the fibre, $n_{cladd(x)}$ is the effective index of the $HE_{1,x}$ cladding mode, Λ is the period of the LPG and N is an integer representing the order of diffraction. The efficiency of coupling to the EH modes is small, and thus attenuation bands corresponding to coupling to EH modes are in general not visible in the transmission spectrum [5]. The interaction between core and cladding modes causes the exact form of the spectrum and the centre wavelengths of the attenuation bands to be sensitive to the local environment: temperature, strain, bend radius and refractive index of the medium surrounding the fibre [17], with sensitivities governed by the differential sensitivities of the core and cladding modes.

The transmission spectrum of an LPG is shown in Fig. 1, exhibiting attenuation bands corresponding to 1st order and 2nd order coupling to the cladding modes. The attenuation bands have been labeled according to their associated cladding modes. The modes have been identified by using the LP approximation to calculate the core and cladding mode dispersion. The indices were then used in equation 1 to determine the coupling wavelengths and, noting that the LP_{0x} modes approximate to the $HE_{1,x}$ modes, the $HE_{1,x}$ modes were labeled in order.

2nd order coupling of LPGs had received little attention to date. There has been a report of the use of the differential sensitivities of the 1st and 2nd order-coupling attenuation bands to discriminate between the temperature and strain responses of the LPG [16]. Here the response of the 2nd order coupling attenuation bands to the deposition of a nanostructured coating is investigated

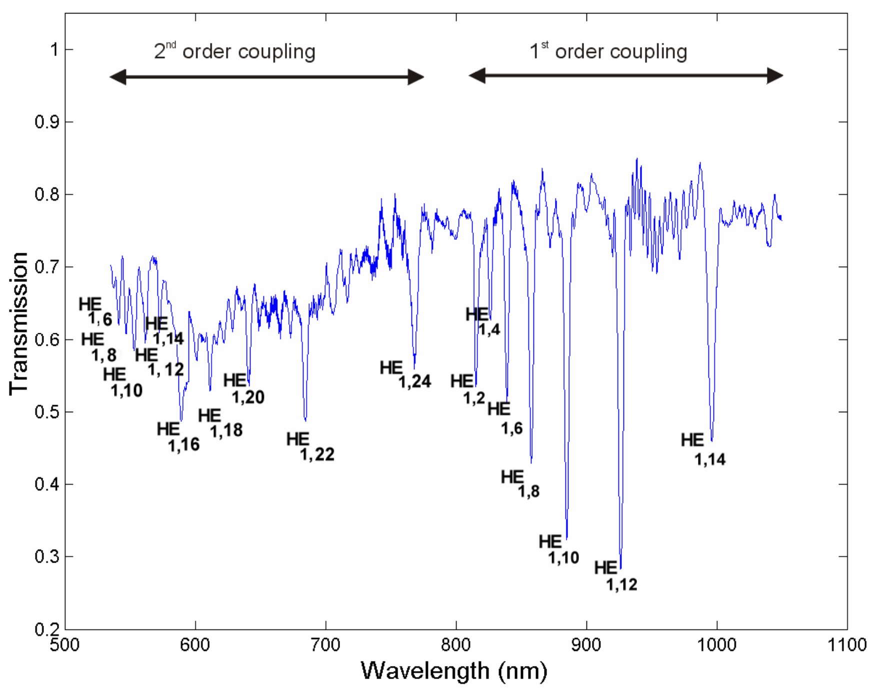

Fig. 1. The transmission spectrum of a long period grating, of length 40 mm and of period 400 μm fabricated in Fibrecore SM750, a single mode optical fibre with cut-off wavelength 700 nm. The labels indicate the cladding modes to which coupling occur.

The sensitivity of the cladding modes' effective refractive indices to the refractive index of the surrounding material [18] is particularly attractive for chemical sensing applications, as

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LPGs offer a method for facilitating the interaction of modes of the fibre with a chemically sensitive coating, without the requirement for removal of the cladding.

A number of studies have shown that the optimum thickness of the coating lies in the 100 – 200 nm range for coatings of refractive index 1.5 – 1.7. At these thicknesses the attenuation bands show a significant response to the optical properties of the coating [1, 2, 3]. The large sensitivity is attributed to a reorganization of the cladding modes which occurs when the thickness of the coating is sufficient such that it acts as a waveguide that is phase matched to one of the cladding modes. In this region, the effective indices of the cladding modes change rapidly [5, 6, 7] resulting in the observed phenomenon.

In a recent publication the evolution of the spectrum of a selection of 1st order coupling attenuation bands to increasing overlay thickness was presented by Del Villar *et al* [14]. The data originally presented in [1], where the attenuation bands corresponding to the 1st order coupling of an LPG of period 400μm fabricated in a boron-germanium co-doped photosensitive optical fibre were monitored as the LPG was coated with ω–tricosenoic acid using the LB technique, is re-plotted in Fig. 2 in the style of [14]. The results illustrate clearly the now well-established form of the response, where the attenuation bands show a blue-shift in wavelength, the rate of change of wavelength accelerating as the thickness approaches a few hundred nm, before the extinction ratio of the bands reduces. Thereafter the bands reappear, taking on the characteristics of the neighboring lower wavelength attenuation band.

2.1 LP mode description

An analysis that approximates the cladding modes to LP modes [6], applicable when the index contrast between the cladding and the coating is small, agrees well with the form of the behaviour shown in Fig. 2. Within the transition region where the attenuation bands' extinction ratios reduce, the analysis predicts that the cladding modes undergo a reorganization as the coating becomes thick enough to guide one of the LP modes. The effective indices of the modes change rapidly, with the effective index of the modes of order higher than that guided by the coating taking on that of the immediate lower order LP mode, i.e. the effective index of the $LP_{0,i}$ (i=1,2,3..) cladding mode becoming that of the $LP_{0,i-1}$ cladding mode.

For coatings with large losses, the treatment in [6] predicts that a significant increase in the imaginary part of the effective index of the cladding modes accompanies the transition to guidance by the coating of one the cladding modes. This acts to reduce the efficiency of the coupling between the core and cladding modes, resulting in a reduction of the extinction of the attenuation bands. As the thickness of the coating is increased further, the mode reorganization becomes complete, and the efficiency of coupling to the reorganized modes, and thus the extinction ratio of the attenuation bands, increases. The form of the curves representing the wavelength shifts of the attenuation bands supports the assertion that this takes place to compensate for the fact that one of the cladding modes becomes guided by the overlay, and that the mode reorganization allows the original mode distribution within the cladding to the recovered.

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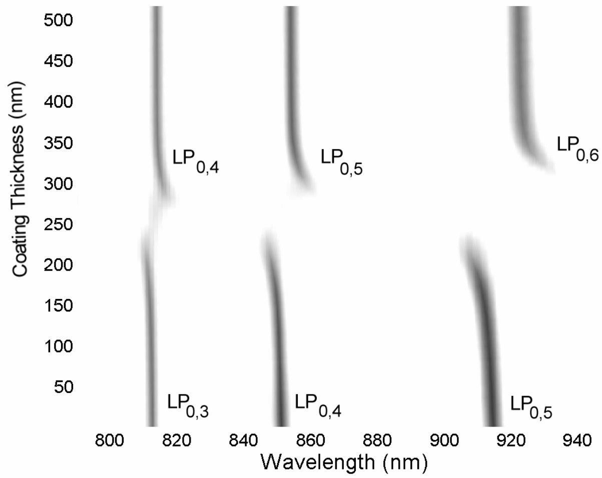

Fig. 2. Evolution of the optical spectrum of an LPG in response to an increase in the overlay thickness, for a coating of refractive index 1.57. The LPG was fabricated in a photosensitive optical fibre with cut off wavelength 650nm. The gray scale represents the measured transmission, with white corresponding to 100%, and black to 0%. The attenuation bands are labeled according to the attenuation bands to which coupling occurs, and the LP notation has been adopted to aid the discussion of the process provided in section 2.1

2.2 Hybrid mode description

When the index contrast between the cladding and the coating is large, a hybrid-mode approach is adopted [5]. The hybrid-mode treatment predicts that, at the beginning of the transition region, the electric field profiles and the mode effective indices undergo significant changes. As the coating becomes sufficiently thick, the lowest order HE mode becomes guided by the coating. Associated with the onset of the guidance of the HE mode by the coating is a reorganization of the cladding modes, such that the $HE_{1,x}$ mode profile and its effective refractive index evolve into that of the $EH_{1,x-1}$ mode, and the $EH_{1,x-1}$ takes on the characteristics of the $HE_{1,x-2}$ mode. At the end of the transition region the lowest order EH mode becomes guided by the coating, and a further reorganization of the EH and HE modes takes place, with the net effect that the $HE_{1,x}$ takes on the properties of the $HE_{1,x-2}$ after transition region. Thus the transition is a two step process.

In terms of the extinction of the attenuation bands, on the first step of the transition region, as the HE modes take on the symmetry of the EH modes, the coupling efficiency and thus extinction ratio of the attenuation bands corresponding to core to HE cladding mode coupling decrease. However, the EH modes that evolve into the HE mode symmetry increase their coupling strength and associated attenuation bands appear within the spectrum. At the 2nd step of the transition region, this process reverses, with the EH cladding mode-associated attenuation bands disappearing, and the HE cladding mode-associated bands reappearing [5]. An experimental observation of this effect is reported in this paper.

3. Experiment

An LPG of length 40 mm and of period 400 μm was fabricated in Fibrecore SM750, a single mode optical fibre with cut-off wavelength 700 nm. The photosensitivity of the fibre was enhanced by pressurizing it in hydrogen for a period of 2 weeks at a pressure of 150 bar at room temperature. The LPG was fabricated by illuminating the fibres by the output from a frequency-quadrupled Nd:YAG laser, operating at 266nm, through an amplitude mask. The transmission spectrum of the optical fibre was recorded by coupling the output from a

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tungsten-halogen lamp to into the fibre, and analyzing the transmitted light using a fibre coupled CCD spectrometer. The transmission spectrum is shown in Fig. 1.

The transmission spectrum contains two sets of attenuation bands. The bands in the range 800 nm to 1100 nm correspond to the 1st order coupling. The attenuation bands are labeled corresponding the $HE_{1,x}$ cladding mode to which coupling takes place. The HE notation is used as it facilitates direct comparison with the theoretical analysis reported in [6]. Given the characteristics of the LPG and of the optical fibre, the core mode is phase matched to the first 8 ($HE_{1,2}$ - $HE_{1,16}$) cladding modes, with seven of the associated attenuation bands appearing within the spectral range of the CCD spectrometer. The bands in the lower wavelength range, 530 nm to 800 nm, correspond to 2nd order coupling to cladding modes. The 2nd order coupling process allows phase matching of the core mode to a larger numer of cladding modes. This can be predicted, as shown in Fig. 3 (a) and Fig. 3 (b), where equation (1) has been used to plot the relationship between the coupling wavelengths to each of the bands and the period of the LPG. The data plotted in Fig. 3 was calculated using the LP approximation to calculate the dispersion of the core and cladding modes. The approximations made in the model and lack of information of the exact fibre parameters means that this approach provides a qualitative model of the effects observed.

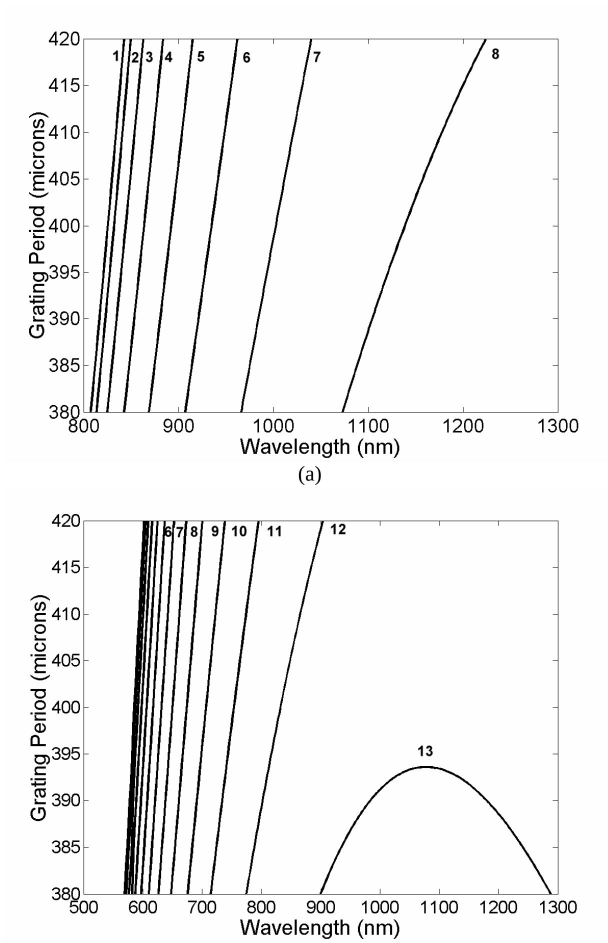

Fig. 3. The relationship between the grating period and the wavelength at which coupling occurs to a set of cladding modes, assuming that the LPG was fabricated in an optical fibre of cut off wavelength 670 nm. (a) 1st order coupling and (b) 2nd order coupling. The numbers refer to the order of the cladding mode, LP_{0x}.

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The evolution of the attenuation bands in response to increasing coating thickness was investigated by depositing a film of ω-tricosenoic acid, which has a refractive index of 1.57 and a molecular length of 2.6 nm [19], using the Langmuir-Blodgett (LB) technique. The LB technique facilitates deposition of the material one molecular layer at a time onto a substrate. The ω-tricosenoic acid was spread from dilute chloroform solutions (0.1 mg mL^{-1}) onto the pure water subphase (conductivity 18MΩcm) of one compartment of a Nima Technology Model 2410A LB trough, left for 20 min at 20 °C, and compressed at 0.5 cm^2 s^{-1} (0.1% s^{-1} of total surface area). Deposition was achieved at a surface pressure of 30 m Nm^{-1} and a transfer rate of 8 mm min^{-1}. The fibre containing the LPG was positioned vertically so its long axis was aligned with the dipping direction and was alternately raised and lowered through the floating monolayer at the air–water interface to deposit the coating. Transmission spectra were recorded after the deposition of each monolayer, with the LPG below and above the water subphase for alternate layers.

4. Results and discussion

The results are shown in Fig. 4(a) and Fig. 4(b), where the gray scale image allows the evolution of the transmission spectrum with increasing coating thickness, with the LPG under and above the water subphase, respectively, to be seen clearly. The results show a number of interesting features.

4.1 1st order coupling attenuation bands

The transition from the $HE_{1,x}$ mode to the $HE_{1,x-2}$ mode is illustrated by the continuous nature of the wavelength shift of the bands across the transition region, which corresponds to a coating thickness range of approximately 250 nm - 390 nm with the LPG below the water subphase and 325 nm to 455 nm above the water subphase for the 1st order coupling. The transition for the 2nd order coupling takes place for smaller values of coating thickness, 150 nm – 200 nm with the LPG below the water subphase and 200 nm - 325 nm with the LPG above the water subphase. Note that the attenuation band depth is reduced considerably within the transition region for the reasons discussed previously [5]. It is apparent that the transition region onset occurs for smaller coating thickness when the LPG is below the water subphase, that the transition onset occurs for a larger coating thickness with increase in cladding mode order, and that the transition occurs over a wider range of coating thicknesses for higher order cladding modes, as noted in [14].

Figure 4 shows that, for the 1st order coupling attenuation bands, a new spectral feature is generated within the transition region, which is believed to correspond to the transformed $EH_{1,x-1}$ modes, predicted in [5]. This was noted in [14], but was not shown as distinctly as it is here. The spectra recorded during the transition region are shown in more detail in Fig. 5. It can be seen that the attenuation band corresponding to coupling to the $HE_{1,8}$ mode disappears from the spectrum implying that this is the mode that is guided by the coating layer.

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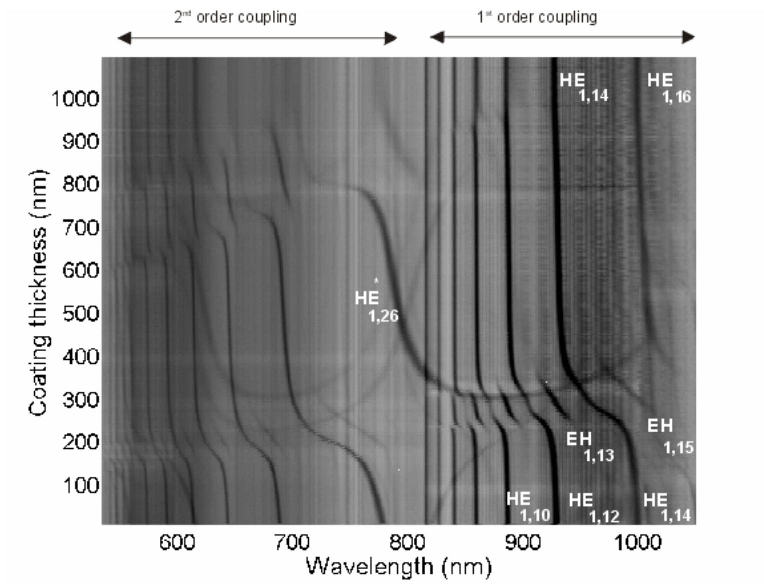
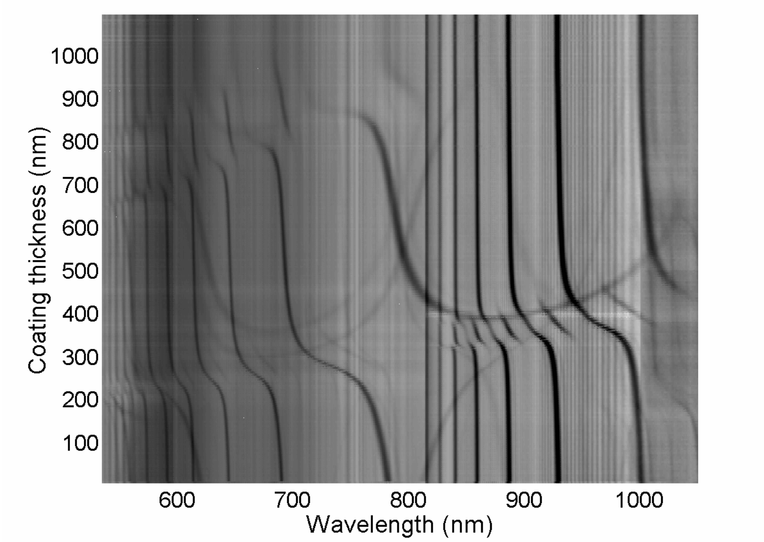


(a)



(b)

Fig. 4. Transmission spectrum response to the deposition of a coating of tricosenoic acid, (a) recorded with the LPG below the water subphase and (b) recorded with the LPG above the water subphase. In Fig. 4(a), a selection of the attenuation bands are labeled according to the corresponding cladding modes to aid the discussion in section 4.

These experimental observations are significant for sensing applications, as there is now a spectral feature that appears when the LPG is most sensitive to the optical properties of the coating, and when, in most previous reports, the attenuation bands disappear. It can be seen from the graph that the attenuation bands corresponding to the three cladding modes ($HE_{1,2}$, $HE_{1,4}$, and $HE_{1,6}$) of order less than that guided by the coating ($HE_{1,8}$) respond differently to the presence of the coating. A similar effect was noted in [14], and is attributed to the theoretical prediction that, for a coating with large losses, it is not necessarily the lowest order mode that is guided, and that the attenuation bands corresponding to coupling to cladding

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modes of order lower than the mode that is guided by the overlay behave differently to those of higher order.

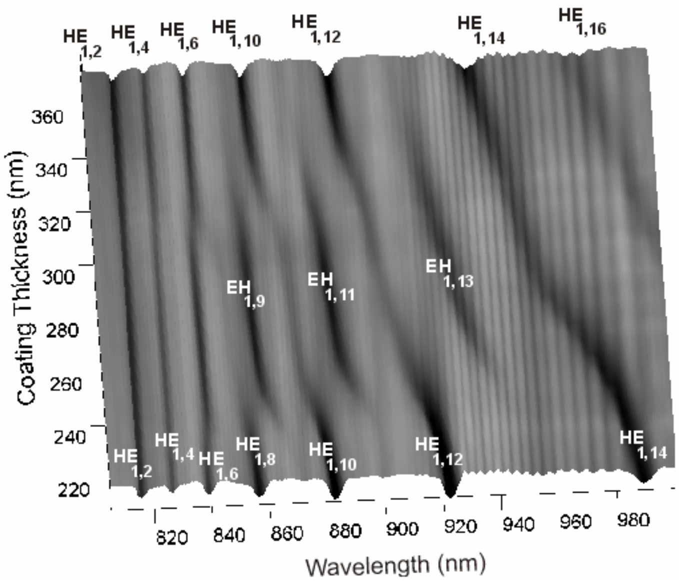

Fig. 5. Evolution of the transmission spectra of the 1st order coupling attenuation bands recorded with the LPG under the water subphase, recorded for coating thicknesses that correspond to the 1st mode- transition region.

The two-step change in the attenuation band wavelength can also be seen within the transition region shown in Fig. 4(a) and Fig. 5, for example for the $HE_{1,10}$ mode. This corresponds to a two-step change in the effective indices of the cladding modes predicted in [5], with the first step corresponding the $HE_{1,8}$ mode becoming guided by the coating, and the second to the $EH_{1,9}$ cladding mode becoming guided by the coating.

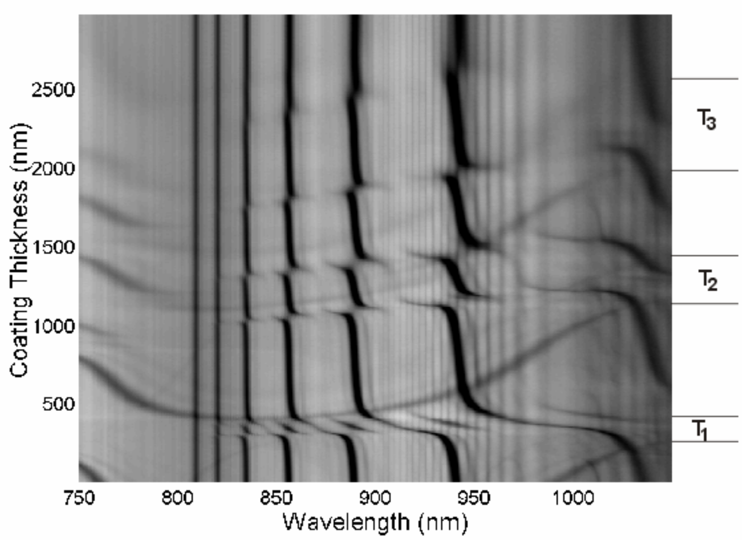

Fig. 6. The response of the 1st order coupling features of LPG transmission spectrum to the deposition of a coating of ω– tricosenoic acid. T_1, T_2 and T_3 indicate the first three transition regions. The spectra were recorded with the LPG above the water subphase

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Figure 6 illustrates the cyclical behaviour of the mode transition process, showing three transition regions. An interesting feature of the response reported here is that the width of the transition region increases with increasing coating thickness and that it appears that the separations of the transition regions are not uniform. The extinction of the EH-mode-associated attenuation bands is also observed to increase for the 2nd and 3rd transition regions, as compared with the 1st.

4.2 2nd order coupling attenuation bands

The 2nd order coupling attenuation bands show a similar response to that of the 1st order bands, but the reduction in their extinction ratio within the first transition region is not so pronounced. The existence of transformed EH modes in the transition region is indicated, but with significantly reduced extinction compared with the 1st order coupling. The onset of the transition region for the second order coupling bands occurs for thinner films. This may be attributed to the waveguide dispersion of the fibre, and also to the dispersion of the film, as ω-tricosenoic acid exhibits absorption at around 400nm [19], which indicates an increasing refractive index at the lower wavelengths. The 2nd transition region for the 2nd order attenuation bands begins sooner that that of the 1st order bands.

Figure 4 also reveals the generation of two new attenuation bands, which occurs at the end of the transition region at a wavelength of approximately 900nm. While these bands appear within the spectral region that has been associated with the 1st order coupling, they are in fact related to the 2nd order coupling. Initially degenerate, the new bands move in opposite directions along the optical spectrum with subsequent increase in coating thickness. We propose that these dual attenuation bands both correspond to 2nd order coupling to the $HE_{1,26}$ cladding mode.

Dual attenuation bands corresponding to coupling to higher order cladding modes have been observed previously [20], and are explained by considering the differential propagation constant, Δβ,

$$\Delta\beta = \frac{2\pi}{\lambda}\left(n_{core} - n_{clad(x)}\right) \tag{2}$$

and its influence on equation 1. As the wavelength increases, the cladding mode effective index decreases rapidly, an effect more pronounced for higher order cladding modes. When the rate of change of the numerator of equation (2) exceeds that of the denominator, the relationship between the resonant wavelength and the grating period (equation (1)) exhibits a turning point [16, 21]. This is illustrated in Fig. 3 where the plots show the relationship between period and coupling wavelengths for the 1st and 2nd order coupling to the cladding modes.

Figure 3 (b) predicts that, for a period of 380 nm, 2nd order coupling to the 13th cladding mode (equivalent to $HE_{1,26}$) would occur at two wavelengths within the spectral region shown, giving rise to dual attenuation bands. Such bands have been shown previously to exhibit sensitivities to parameters such as bending, transverse load [21] and refractive index [20] that have opposite sign.

Figure 7 illustrates the predicted response of these curves to increasing cladding mode effective index. Figure 7 shows that increasing the refractive index of the material surrounding the cladding, and thus increasing the effective indices of the cladding modes, causes the curve corresponding to coupling to the 13th cladding mode to move in the direction indicated by the arrow.

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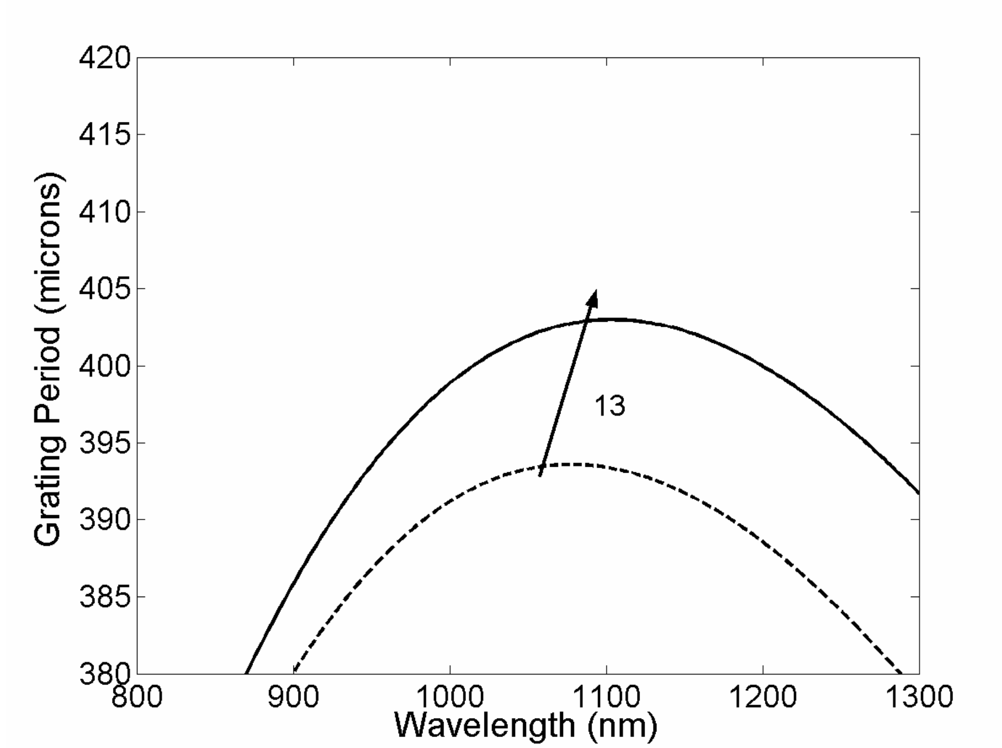

Fig. 7. The relationship between the grating period and the wavelength at which 2nd order coupling occurs to the 13th cladding mode (equivalent to $HE_{1,26}$) when the fibre is assumed to be immersed in air (dashed lines) and in a material of refractive index 1.43 (solid lines).

The turning point for the 2nd order coupling to the 13th mode (equivalent to $HE_{1,26}$) is shown in Fig. 7, illustrating that, for the period of the LPG used in these experiments (400 μm), when the LPG is uncoated there is no coupling to this mode. However, as the effective index of the cladding mode increases, as in response to the deposition of the coating, the emergence of dual attenuation bands is predicted to occur. The two bands move in opposite directions along the optical spectrum as the external index increases, matching the experimental observations. The asymmetry of the experimentally observed responses of the dual bands to increasing coating thickness may be a result of the asymmetry of the curve in Fig. 7, and of the dispersion introduced by the coating within the transition region. It is anticipated that, could the appropriate spectral range be observed from the full range of 1st order coupling attenuation bands, a similar effect would be observed. Figure 6 shows that the appearance of the dual attenuation bands repeats for each transition cycle.

The two faint bands, which lie within the 600nm to 800 nm wavelength range in Fig. 4, appear to have a similar form to the dual bands discussed above, and may correspond to third order coupling to cladding modes with such turning points.

5. Summary

An experimental investigation of the behaviour of the attenuation bands corresponding to the LPG induced first and second order coupling between core and cladding modes in response to increasing coating thickness has been reported. The results confirm many of the theoretical predictions for the response of the LPG transmission spectrum, including the two step transition, and the transformation of the mode profile from HE to EH and back to HE that occurs as part of the mode reorganization process that takes place in the transition region.

The 2nd order coupling attenuation bands show a similar behaviour to the first order bands but respond to thinner films, a result of a combination of the effects of waveguide dispersion

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and the increased refractive index of the ω–tricosenoic acid coating at lower wavelengths. The evolution of a dual attenuation band that appeared within the optical spectrum during the transition region has been observed and explained.

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