

## **Response of fibre optic long period gratings operating near the phase matching turning point to the deposition of nanostructured coatings.**

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The response of the dual resonant bands of a fibre optic long period grating operating near the phase matching turning point to the deposition of a nanostructured coating is investigated. The dual resonant bands are observed to show a high sensitivity to the thickness of the coating, but with opposite signs. Appropriate design of the device, based on the grating period, the refractive index and thickness of the coating and the fibre composition, can allow the sensitivity of the device to the optical thickness to be optimized. A sensitivity of 1.45 nm / nm is observed experimentally.

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## Introduction

Fibre optic long period gratings (LPGs) have been extensively investigated for sensing applications as a result of their sensitivities to measurands such as temperature [1], strain [1], refractive index [2] and curvature [3], and because of the ability to tune their sensitivity by virtue of grating period, fibre composition and geometry [4,5]. It has been shown that, for highest sensitivity, the LPG should be designed to couple light from the propagating core mode to a cladding mode at a wavelength near the phase matching turning point [6], which is characterised by the generation of dual resonant bands that show sensitivities of opposite sign. This has been exploited to measure temperature and refractive index with high sensitivity [6].

It has been shown that the transmission spectrum of an LPG exhibits a high sensitivity to the optical properties of coatings of materials of refractive index higher than that of the cladding when the coating is of thickness of order 200 nm [7,8,9]. This sensitivity has been exploited to demonstrate a range of chemical sensors, for example, pH [10], copper [11] and DNA damaging agents [12]. To date, the studies of the response of LPG transmission spectra to the deposition of nanostructured coatings has focussed on resonance bands corresponding to coupling to the lower order cladding modes, operating at wavelengths below their dispersion turning point. In this paper, the response of resonance features designed to appear near the cladding modes' dispersion turning points to the deposition of nanostructured coatings is investigated.

## Theory

A long period grating is a core-cladding mode coupling device, consisting of a periodic modulation of the propagation constants of the modes of a fibre. The modulation typically has a period in the range 100  $\mu\text{m}$  - 1000  $\mu\text{m}$  and may be induced using a variety of techniques, including UV laser irradiation [1], CO<sub>2</sub> laser irradiation [13] and exposure to an electric arc [14]. Efficient coupling between the core and cladding modes occurs only where there is significant overlap between the electric field profiles of the modes. Resonant bands corresponding to coupling from the guided core mode to a discrete set of symmetrical cladding modes appear in the transmission spectrum of the fibre, centred at discrete wavelengths that are governed by the phase matching expression<sup>1</sup>.

$$\lambda_{(x)} = \frac{n_{core} - n_{clad(x)} \Lambda}{N} \quad (1)$$

where  $\lambda_{(x)}$  represents the wavelength at which coupling occurs to the LP<sub>0x</sub> mode,  $n_{core}$  is the effective refractive index of the mode propagating in the core of the fibre,  $n_{clad(x)}$  is the effective index of the LP<sub>0x</sub> cladding mode,  $\Lambda$  is the period of the LPG and  $N$  is an integer representing the order of diffraction. The efficiency of coupling to the asymmetric modes is small, and thus in general no attenuation bands corresponding to this coupling are visible in the transmission spectrum.

Calculation of the core and cladding modes' dispersions allows the prediction of the coupling wavelength as a function of the period of the grating, as plotted in figure 1 (a) for periods in the range 350  $\mu\text{m}$  - 450  $\mu\text{m}$  and over a wavelength range 750 nm – 1150 nm, selected

to match the experimental constraints imposed by the use of a CCD spectrometer to monitor the transmission spectrum of an LPG fabricated in a fibre with cut off wavelength in the visible region of the spectrum. The data plotted in Figure 1 were calculated using the LP approximation to calculate the dispersion of the core and cladding modes.

In the regime depicted in figure 1 (a), the coupling is to the lower order cladding modes  $LP_{01} - LP_{08}$ , with the graphs showing a positive gradient across the wavelength range. For shorter periods, as shown in figure 1(b), coupling takes place to higher order cladding modes, The behaviour in this regime is markedly different, with the phase matching condition showing a turning point in the wavelength range of interest. The wavelength at which the turning point occurs decreases with increasing wavelength. It should be noted that all cladding modes will exhibit a turning point, however for the lower order modes this lies at longer wavelengths, outside the range of interest here.

It is thus possible to select a period where the coupling to a single cladding mode occurs at two wavelengths. As the period changes, or the difference in the core and cladding mode effective indices change in response, for example, to a change in temperature or in the surrounding refractive index, the central wavelengths of the dual resonance bands move in opposite directions. It has been demonstrated theoretically and experimentally that the LPG resonance bands may exhibit their highest sensitivity to external perturbations at their phase matching turning point [6]. Thus to design a device to operate at a specified wavelength with high sensitivity to external perturbation, a cladding mode and period that produce a turning point at that wavelength should be selected.

## Experiment

LPGs of length 30 mm and of period 80  $\mu\text{m}$  and 100  $\mu\text{m}$  were 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 150 bar at room temperature. The LPGs were fabricated in a point-by-point fashion, illuminating the fibre by the output from a frequency-quadrupled Nd:YAG laser, operating at 266nm. The transmission spectrum of the optical fibre was recorded by coupling the output from a tungsten-halogen lamp into the fibre, and analysing the transmitted light using a fibre coupled CCD spectrometer. A typical transmission spectrum, illustrating the split resonance bands, is shown in figure 2 (a).

The evolution of the resonance bands in response to increasing coating thickness was investigated by depositing a film of  $\omega$ -tricosenoic acid, which has a refractive index of 1.57 and a molecular length of 2.6 nm [15], onto the section of optical fibre containing the LPG using the Langmuir-Blodgett (LB) technique [7]. The LB technique facilitates deposition of the material one molecular layer at a time onto a substrate. The  $\omega$ -tricosenoic acid was spread from dilute chloroform solutions ( $0.1 \text{ mg mL}^{-1}$ ) onto the pure water sub-phase (conductivity  $18 \text{ M}\Omega\text{cm}$ ) of one compartment of a Nima Technology Model 2410A LB trough, left for 20 min at  $20^\circ\text{C}$ , and compressed at  $0.5 \text{ cm}^2 \text{ s}^{-1}$  ( $0.1\% \text{ s}^{-1}$  of total surface area). Deposition was achieved at a surface pressure of  $26 \text{ m Nm}^{-1}$  and a transfer rate of  $8 \text{ mm min}^{-1}$ . The fibre containing the LPG was positioned vertically so that 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. The fibre was placed under tension, to ensure no bend-induced changes to

the transmission spectrum [16,17]. Transmission spectra were recorded after the deposition of each monolayer, with the LPG below and above the water sub-phase for alternate layers. The effect of the presence of the coating material on the split resonance bands is shown in figure 2 (b).

## Results and Discussion

The response of the central wavelengths of the resonance bands to layer by layer increases in the thickness of the coating deposited onto the LPG of period 80  $\mu\text{m}$  is illustrated in the grey scale plot shown in figures 3 (a) and (b). In figure 3, a transmission of 100% is represented by white and 0% by black. The dark line originating at a wavelength of 575 nm in the uncoated LPG, represents the resonance band corresponding to first order coupling to the  $\text{LP}_{023}$  cladding mode. This resonance band exhibits the typical behaviour reported previously for nanoscale coating deposition onto LPGs, in that the resonance band undergoes a blue shift, showing a region of high sensitivity that characterises the “mode transition region”, in which one of the lower order cladding modes becomes phase matched to a mode of the waveguide formed by the coating material. In this region the effective indices of the cladding modes change rapidly with increasing coating thickness [18, 19], which, from equation 1, causes a corresponding change in the central wavelength of the resonance bands.

The LPG was designed to access the dispersion turning point of the  $\text{LP}_{024}$  mode, generating two closely spaced resonance bands, centred at 700 nm and 750 nm, in the transmission spectrum of the uncoated 80  $\mu\text{m}$  period LPG surrounded by air. As the film thickness increases the central wavelengths of the two bands shift in opposite directions, as plotted in figure 4. As the thickness

increases beyond the cladding mode transition region, the sensitivity to coating thickness reduces. The effect repeats as the coating thickness is increased further.

Comparison of figures 3(a) and 3 (b) shows the effect of monitoring the transmission spectrum with the LPG above and below the water subphase, as result of the refractive index sensitivity of the LPG. This indicates the need to consider the medium in which the LPG is immersed for sensing applications. It is interesting to note that, leading the response of each of the dominant resonance bands, there is a resonance band of considerably reduced extinction. It is suggested that these may correspond to coupling to an asymmetric cladding mode.

It can be seen from figure 4 that the sensitivity of the resonant wavelengths to the coating thickness exhibits two gradients. The initial response shows a sensitivity to coating thickness of 0.27 nm / nm for the red shifted band, and - 0.25 nm/nm for the blue shifted band, over a thickness range from 0 nm to 180 nm. The region of highest sensitivity lies within the transition region, from 180 nm to 300 nm, where the peak sensitivity is 0.47 nm / nm for the red shifted band, and - 0.62 nm / nm for the blue shifted band.

To investigate the influence of the period of the LPG on the sensitivity of the coated LPG to the thickness of the coating, the response of the transmission spectrum of the LPG of period 100  $\mu\text{m}$  was investigated. The results are shown in the multimedia file in figure 5, and in the grey scale images in figure 6. In this case, the dispersion turning point of mode  $\text{LP}_{024}$  is accessed at 800 nm for a coating thickness of order 200 nm, which is coincident with the onset of the first mode-transition-region. Initially, there is no band in the 800 nm wavelength range. As the film

thickness increases, a broad attenuation band develops and splits in two, with the central wavelengths of the two bands shifting in opposite directions, as plotted in figure 7. The peak sensitivity to coating thickness is 0.98 nm / nm for the red shifted band, and -1.45 nm / nm for the blue shifted band, over a thickness range from 220 nm to 300 nm. Clearly, the measurement of the wavelength separation of the bands will allow further increased sensitivity. To illustrate the improvement in sensitivity, the highest sensitivity exhibited by a 1<sup>st</sup> order coupling resonance band of a 400  $\mu\text{m}$  period LPG, fabricated in the same type of fibre, is 0.5 nm / nm<sup>20</sup>. It is interesting to note that the 2<sup>nd</sup> order coupling dual resonance bands shown in [20], originating at a wavelength of approximately 800 nm, equivalent to the results shown in figure 3 (a) here, also exhibit sensitivities of order 1 nm /nm. Optimisation of the sensitivities of the 2<sup>nd</sup> order resonance bands is currently being investigated.

The dual resonant bands show their highest sensitivity to external perturbation when the band is at the phase matching turning point. Thus the coincidence of the generation of the dual resonance band with the onset of the 1<sup>st</sup> transition region, where the cladding mode indices are most sensitive to the coating's optical properties [**Error! Bookmark not defined.**], ensures that maximum sensitivity is obtained. Careful choice of the grating period, taking into account the fibre properties and the refractive index of the coating material, is needed to ensure optimum sensitivity. While, in principle, the LPG sensor exhibits its highest sensitivity to all external perturbations at the phase matching turning point, it should be noted that, appropriate choice of core and cladding materials can allow the phase matching condition to specific cladding modes to be insensitive to, for example temperature, eliminating cross-sensitivity issues [6]. In addition,

cross-sensitivity effects can be minimized by appropriate packaging or by exploiting the different sensitivities of each resonance band to different measurands [1].

## **Summary**

An investigation of the response of LPG dual resonance bands near the dispersion turning point to the deposition of a nanostructured coating has been presented. Appropriate design of the device, taking into account the properties of the optical fibre, the period of the LPG and the refractive index of the coating material can ensure that mode dispersion turning point of the LPG coincides with the mode-transition-region that characterises LPGs with nanostructured coatings, ensuring optimum sensitivity to the coating's optical properties. The ability to deposit, in a layer by layer fashion, materials onto an LPG that change their optical properties in response to a particular chemical species [10, 11, 12], offers an exciting prospect for the development of chemical sensors with high sensitivity and specificity.

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## Figure Captions

Figure 1. The relationship between the grating period and the wavelength at which coupling occurs to a set of symmetric cladding modes, assuming that the LPG was fabricated in an optical fibre of cut off wavelength 670 nm. (a) for the 1st 8 modes (b) for the modes 20 – 25. The numbers refer to the order of the cladding mode,  $LP_{0x}$ .

Figure 2. The transmission spectrum of the LPG of period 80  $\mu\text{m}$  fabricated in Fibrecore SM750, a single mode optical fibre with cut-off wavelength 700 nm, with (a) no coating and (b) a coating of 110 nm of  $\omega$ -tricosenoic acid.

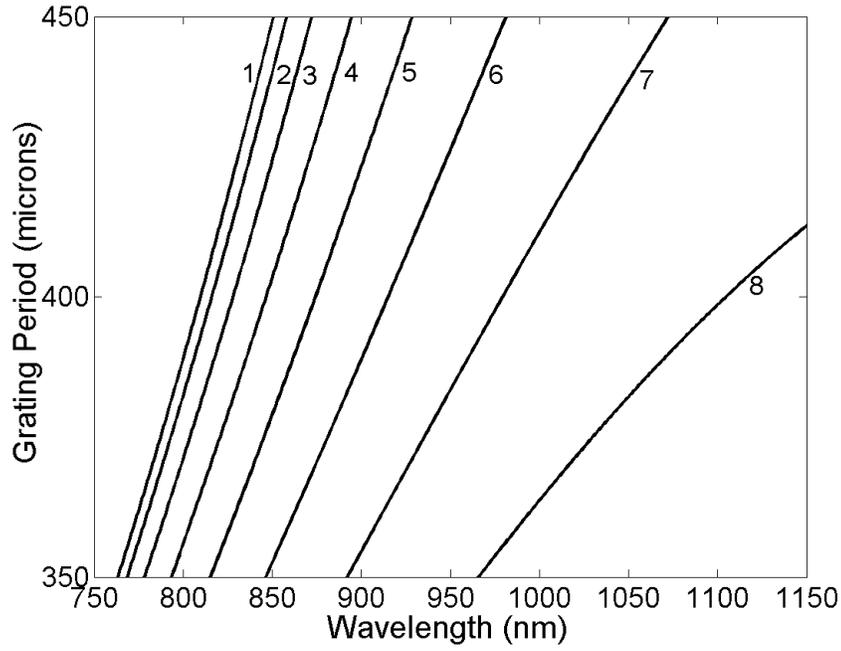
Figure 3. Evolution of the transmission spectrum of an LPG of period 80  $\mu\text{m}$ , fabricated in a single mode optical fibre of cut-off wavelength 700nm, in response to the deposition of a coating of  $\omega$  - tricosenoic acid using the Langmuir Blodgett Technique. (a) with the LPG above the water sub-phase and (b) with the LPG below the water sub-phase. The gray scale represents the measured transmission, with white corresponding to 100%, and black to 0%. The features visible in the range 800 nm to 950 nm which do not change in wavelength with increasing coating thickness are a result of a cavity formed at a mechanical splice in the system.

Figure 4. A plot of the wavelengths of the dual resonance bands as a function of the thickness of a coating of  $\omega$ -tricosenoic acid deposited using the Langmuir Blodgett technique onto an LPG of period  $80\ \mu\text{m}$  fabricated in single mode optical fibre of cut off wavelength  $700\ \text{nm}$ .

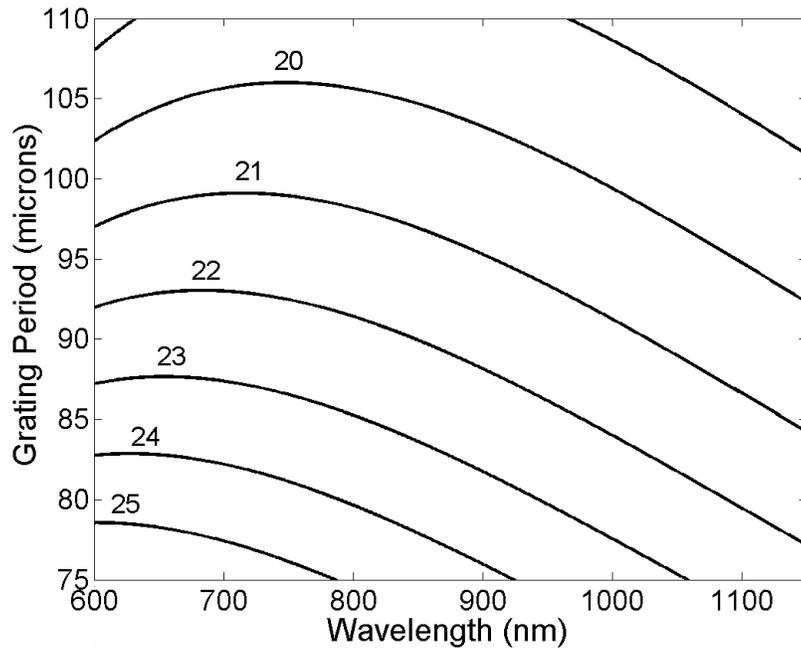
Figure 5. An animation showing the evolution of the dual resonance bands of an LPG of period  $100\ \mu\text{m}$ , fabricated in a single mode optical fibre of cut-off wavelength  $700\text{nm}$ , in response to the deposition of a coating of  $\omega$  - tricosenoic acid using the Langmuir Blodgett Technique.

Figure 6. Evolution of the transmission spectrum of an LPG of period  $100\ \mu\text{m}$ , fabricated in a single mode optical fibre of cut-off wavelength  $700\text{nm}$ , in response to the deposition of a coating of  $\omega$  - tricosenoic acid using the Langmuir Blodgett Technique. (a) with the LPG above the water sub-phase and (b) with the LPG below the water sub-phase. The gray scale represents the measured transmission, with white corresponding to  $100\%$ , and black to  $0\%$ .

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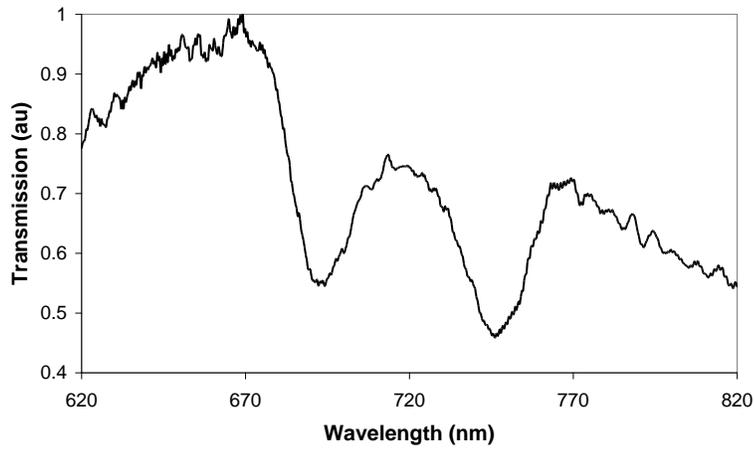


(a)

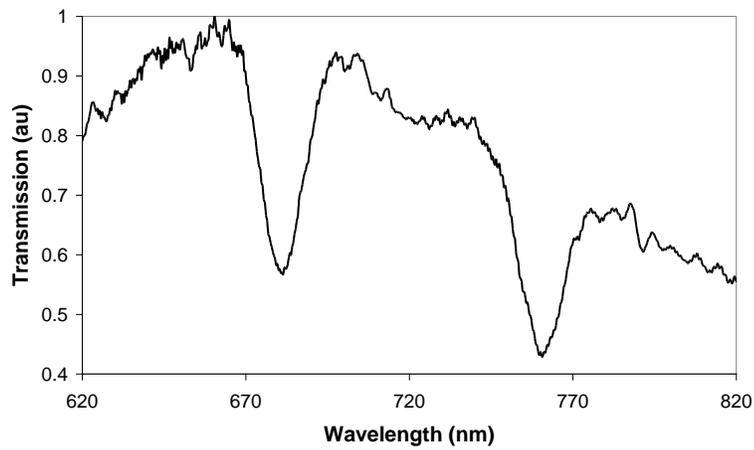


(b)

Figure 1. The relationship between the grating period and the wavelength at which coupling occurs to a set of symmetric cladding modes, assuming that the LPG was fabricated in an optical fibre of cut off wavelength 670 nm. (a) for the 1<sup>st</sup> 8 modes (b) for the modes 20 – 25. The numbers refer to the order of the cladding mode,  $LP_{0x}$ .

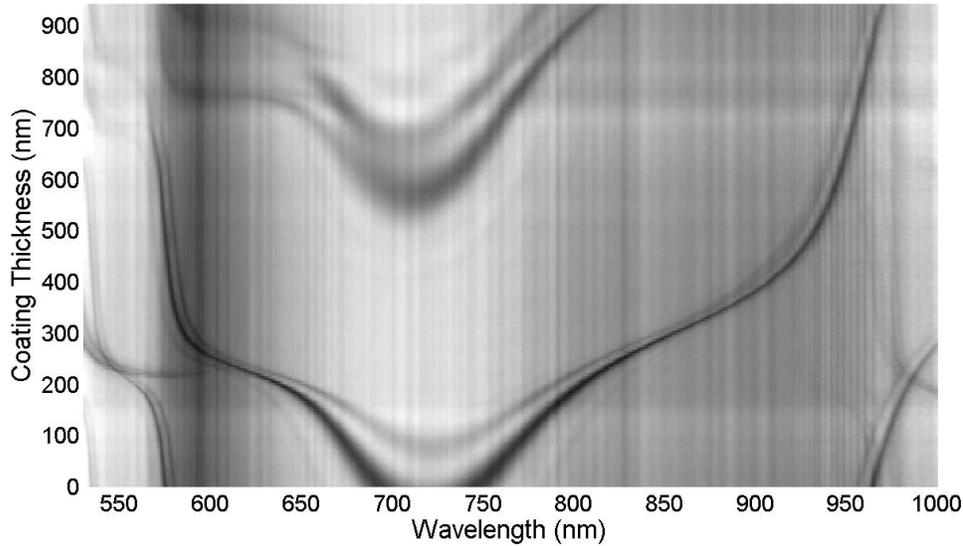


(a)

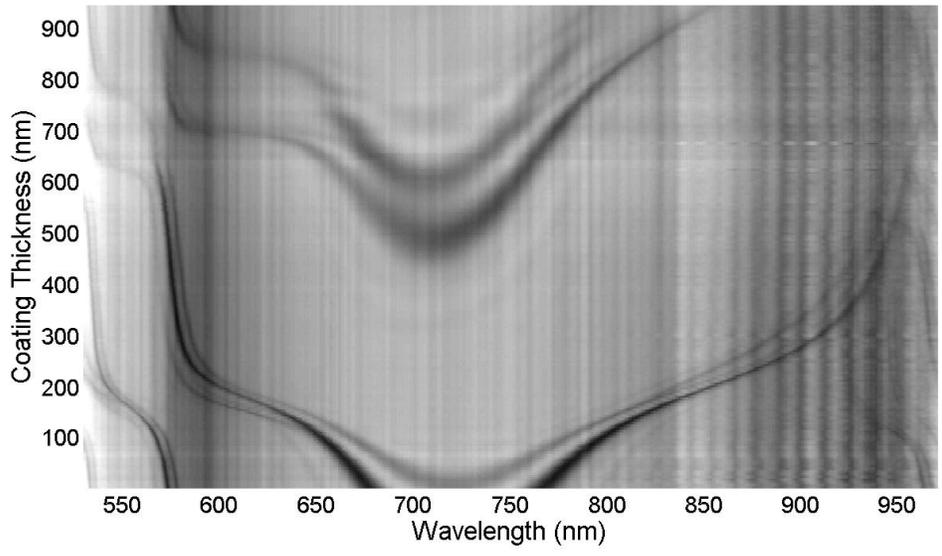


(b)

Figure 2. The transmission spectrum of the LPG of period 80  $\mu\text{m}$  fabricated in Fibrecore SM750, a single mode optical fibre with cut-off wavelength 700 nm, with (a) no coating and (b) a coating of  $\omega$ -tricosenoic acid of thickness 110 nm.



(a)



(b)

Figure 3. Evolution of the transmission spectrum of an LPG of period  $80 \mu\text{m}$ , fabricated in a single mode optical fibre of cut-off wavelength  $700\text{nm}$ , in response to the deposition of a coating of  $\omega$ -tricosenoic acid using the Langmuir Blodgett Technique. (a) with the LPG above the water sub-phase and (b) with the LPG below the water sub-phase. The gray scale represents the measured transmission, with white corresponding to 100%, and black to 0%. The features visible in the range  $800 \text{ nm}$  to  $950 \text{ nm}$  which do no change in wavelength with increasing coating thickness are a result of a cavity formed at a mechanical splice in the system.

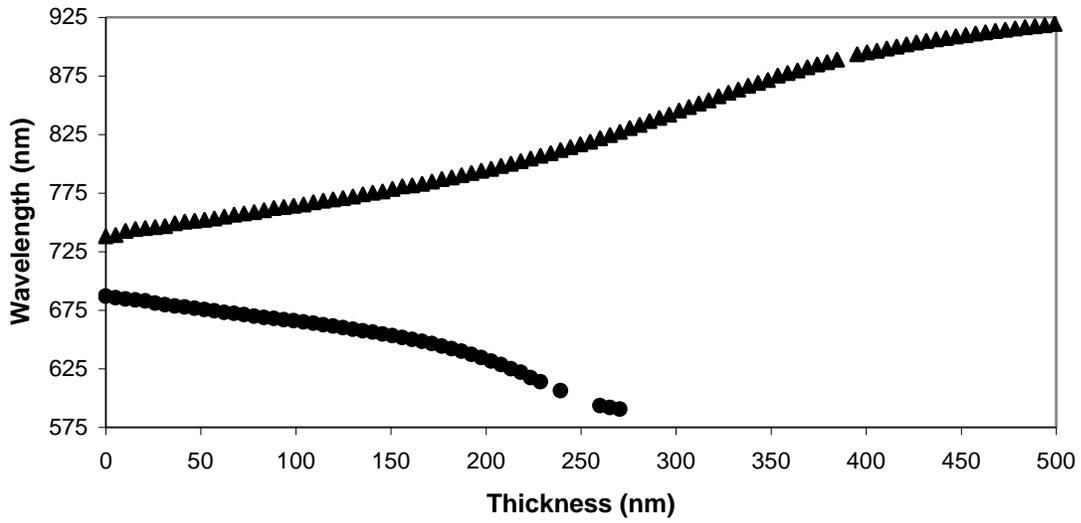


Figure 4. A plot of the wavelengths of the dual resonance bands as a function of the thickness of a coating of  $\omega$ -tricosenoic acid deposited using the Langmuir Blodgett technique onto an LPG of period  $80 \mu\text{m}$  fabricated in single mode optical fibre of cut off wavelength  $700 \text{ nm}$ .

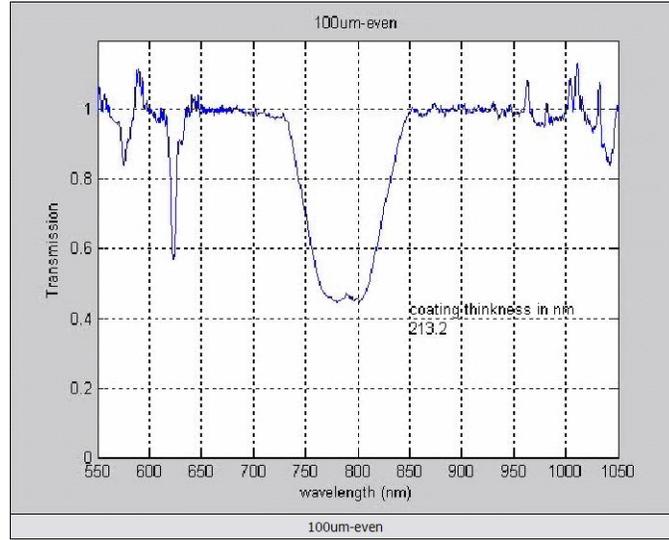
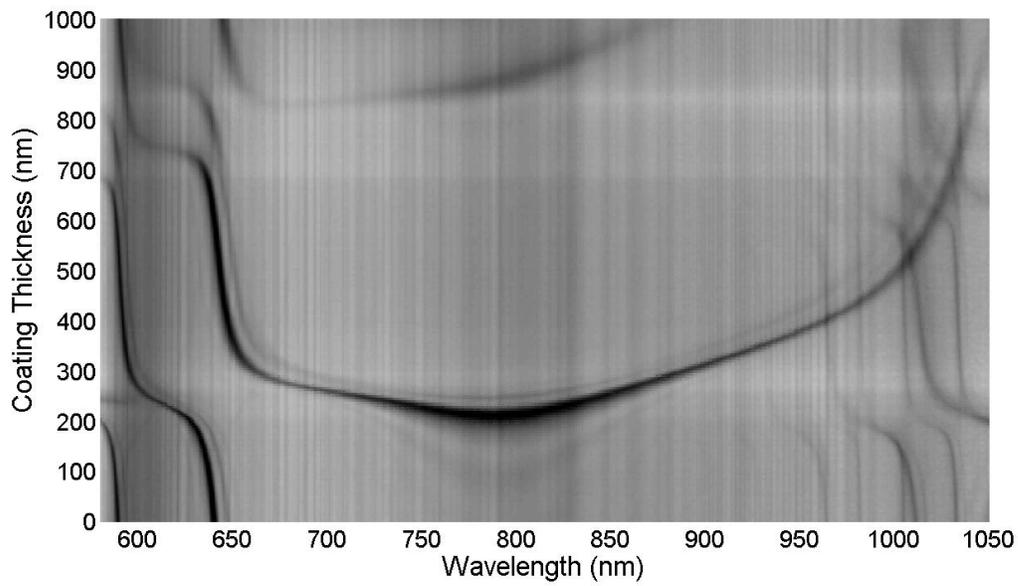
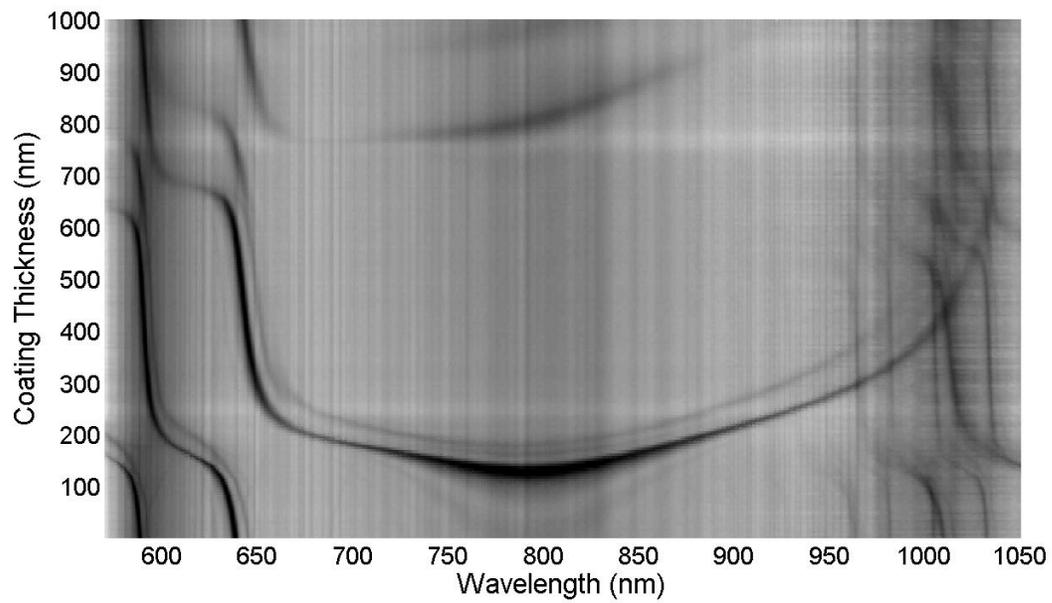


Figure 5. An animation showing the evolution of the dual resonance bands of an LPG of period 100  $\mu\text{m}$ , fabricated in a single mode optical fibre of cut-off wavelength 700nm, in response to the deposition of a coating of  $\omega$  - tricosenoic acid using the Langmuir Blodgett technique.



(a)



(b)

Figure 6. Evolution of the transmission spectrum of an LPG of period  $100\ \mu\text{m}$ , fabricated in a single mode optical fibre of cut-off wavelength  $700\text{nm}$ , in response to the deposition of a coating of  $\omega$  - tricosenoic acid using the Langmuir Blodgett Technique. (a) with the LPG above the water sub-phase and (b) with the LPG below the water sub-phase. The gray scale represents the measured transmission, with white corresponding to 100%, and black to 0%.

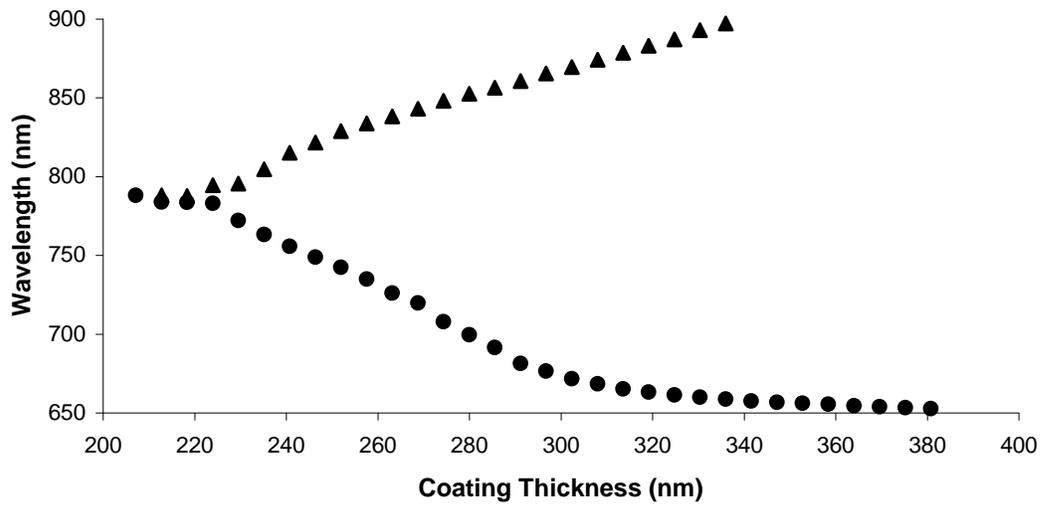


Figure 7. A plot of the wavelengths of the dual resonance bands as a function of the thickness of a coating of  $\omega$ -tricosenoic acid deposited using the Langmuir Blodgett technique onto an LPG of period 100  $\mu\text{m}$  fabricated in single mode optical fibre of cut off wavelength 700 nm.