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Refractive index sensitivity of fibre optic long period gratings with SiO_2 nanoparticle based mesoporous coatings

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ABSTRACT

A fibre optic long period grating (LPG) with a nano-assembled mesoporous coating of alternate layers of poly (allylamine hydrochloride) (PAH) and SiO_2 nanospheres was used for the development of a fibre-optic refractometer. PAH/SiO_2 films of different thickness have been deposited onto LPG in order to study the effect of the film thickness on sensor performance. The device showed a sensitivity of 1927 nm/RIU over a RI range of 1.3233–1.4906.

Keyword List: Fibre-optic sensor, Long period grating, Nanoporous film, refractive index, Refractometer

INTRODUCTION

Precise determination and control of the refractive index (RI) in real time continues to be an important task in a wide range of applications, including bio-chemical and food analysis, and environmental and industrial monitoring. The use of optical fibre gratings, both fibre Bragg gratings (FBGs)[1] and long period gratings (LPGs)[2], has been extensively investigated for refractive index measurements since they offer wavelength-encoded information which overcomes the referencing issues associated with intensity based approaches.

The refractive index sensitivity of LPGs arises from the dependence of the phase matching condition upon the effective refractive index of the cladding modes, which in turn are dependent on the RI of the medium surrounding the cladding. The central wavelengths of the attenuation bands thus show a dependence upon the refractive index of the medium surrounding the cladding, with the highest sensitivity being shown for surrounding refractive indices close to that of the cladding of the optical fibre[2]. For surrounding refractive indices higher than that of the cladding, the centre wavelengths of the attenuation bands show a considerably reduced sensitivity[2]. The RI sensitivity of an LPG is dependent on the order of the cladding mode to which it is coupled, which allows the tuning of the sensitivity by appropriate choice of grating period, with 427.72, 203.18, 53.45, and 32.10 nm/RIU being reported for LPGs of period 159, 238, 400, and 556 μm, respectively fabricated in SMF 28[3]. A further consideration is the geometry and composition of the fibre, with the sensitivity being shown to differ for step index and W profile fibres and a progressive three layered fibre[4].

In order to improve sensitivity of the LPGs written in standard optical fibre configurations to the surrounding RI, approaches such as tapering the fibre[5], or etching the cladding have been investigated[6,7]. Tapering the fibre to a diameter of 25 μm allowed the demonstration of a sensitivity of 715 nm/RIU[5]. Etching the cladding of an arc induced LPG to a diameter of 37 μm allowed the demonstration of a sensitivity of order 20000 nm/RIU[7]. Approaches that involve processing of the fibre, e.g. polishing, etching and tapering, produce significant enhancements in sensitivity, but at the cost of requiring careful packaging to compensate for the reduction in the mechanical integrity of the device.

The deposition of thin film overlays, of thickness on the order of 200 nm, of materials of refractive index higher than that of the cladding has also been investigated for the enhancement of refractive index sensitivity[8, 9]. It has been shown previously theoretically and experimentally[10] that the effective indices of the cladding modes, and thus the central wavelengths of the core-cladding mode coupling bands of LPGs, show a high sensitivity to the optical thickness of high refractive index coatings when the coating's optical thickness is such that one of the low order cladding modes is phase matched to a mode of the waveguide formed by the coating, a condition termed the *mode transition region*. To optimize the sensitivity of such devices to the coating properties, the LPG period should be chosen such that, when the coating thickness corresponds with the mode transition condition, the phase matching condition for the cladding mode of interest is satisfied at the phase matching turning point. A theoretical analysis explored the optimization of the refractive index sensitivity by selecting grating period, coating thickness and refractive index, predicting a sensitivity of 5980 nm/RIU[11].

There has recently been interest in the deposition of porous coatings onto LPGs for sensing applications. Sol-gel coatings of SiO_2 and TiO_2 have been deposited onto LPGs, revealing a gain of approximately 2 in the sensitivity to external RI, with the higher index TiO_2 coating offering the larger response (up to 1067.15 nm/RIU). The authors noted that the

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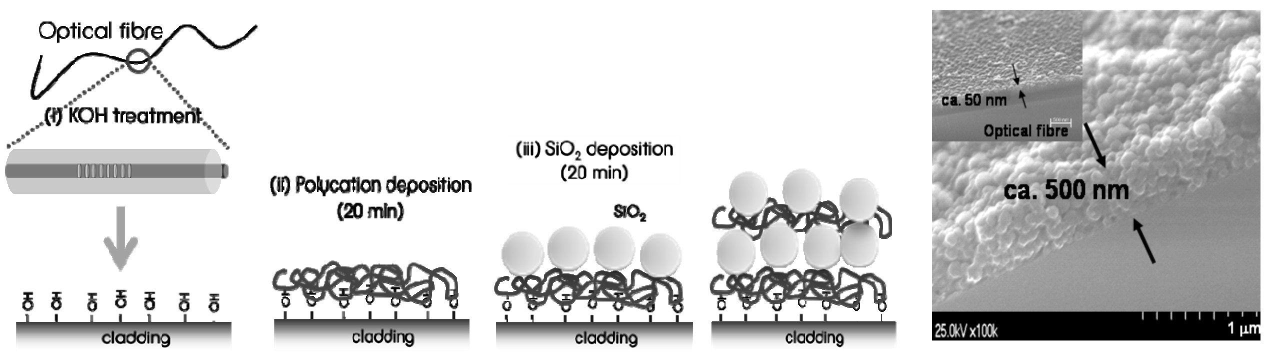
higher the index and thickness of the coating, the more pronounced the enhancement in LPG sensitivity compared to the equivalent uncoated LPG[3]. We have recently demonstrated a new approach to LPG based chemical sensing: chemical infusion of analytes into a mesoporous coating, consisting of a multilayer film of SiO_2 nanoparticles (SiO_2 NPs) deposited using the layer–by–layer (LbL) technique[12]. The initially low RI of the mesoporous coating, 1.2, was increased significantly by the chemical infusion, resulting in a large change in the LPG's transmission spectrum. Additionally, the sensing of ammonia in aqueous solution was used as an example to demonstrate the sensing principle of the LPG sensor[13]. The use of SiO_2 NPs to form the base coating allows the rapid deposition of a coating of the physical thickness required to optimize the sensitivity of the device. In this study the refractive index sensitivity of an LPG with a mesoporous coating of SiO_2 nanospheres is investigated.

EXPERIMENT

An LPG of length 30 mm with a period of 100 μm was fabricated in hydrogen loaded single mode optical fibre (Fibercore SM750) with a cut-off wavelength of 670 nm. The LPGs were fabricated in a point-by-point fashion, side-illuminating the optical fibre by the output from a frequency-quadrupled Nd:YAG laser, operating at 266nm. The grating period was selected such that, when coated and immersed in a solution, the LPG would operate at the phase matching turning point, which, for coupling to a particular cladding mode (in this case LP_{021}), ensures optimised sensitivity[14]. The transmission spectrum of the optical fibre was recorded by coupling the output from a tungsten-halogen lamp into the fibre, and by analyzing the transmitted light using a fibre coupled CCD spectrometer (Ocean



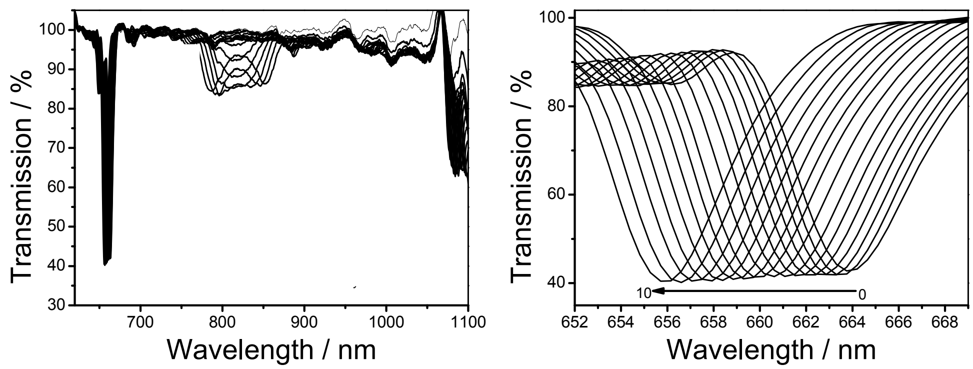
(a) (b)

Figure 1 (a) Schematic illustration of the deposition of the SiO_2 nanospheres onto the LPG, (b) SEM of a 1-layer (inset) and a 10- layer PAH/SiO_2 coated optical fibre.

Figure 1 (a) shows a schematic illustration of the layer-by-layer method employed for the PAH/SiO_2 alternate layer film deposition. Firstly, the section of optical fibre containing the LPG was rinsed in deionised water and then immersed into 1 wt% ethanolic KOH (ethanol/water = 3:2, v/v) for 20 minutes, leading to a negatively charged surface. The optical fibre was then sequentially immersed into a 0.1 wt. % solution containing positively charged polymer PAH (M_w=56 000) with the pH adjusted to ca 11 using NaOH and into a solution containing negatively charge silica nanospheres (Snowtex 20L, Nissan chemical industries) for 20 minutes, so that an alternate layer of PAH and SiO_2 were deposited onto the surface of the fibre. Steps (ii) and (iii) are repeated as appropriate to build a film of required thickness. The fibre was rinsed in distilled water, and dried by flushing with nitrogen gas after each deposition step. Figure 1(b) shows the cross-section of the PAH/SiO_2 film, demonstrating that a uniform and highly porous film with well controlled thickness was deposited onto the LPG. The thickness and refractive index of a single layer PAH/SiO_2 film deposited on a quartz substrate was measured using an ellipsometer, SopraGES5 (Tarn Electronics SARL), to be 46±3 nm and 1.2000 (measured at a wavelength of 633nm) respectively.

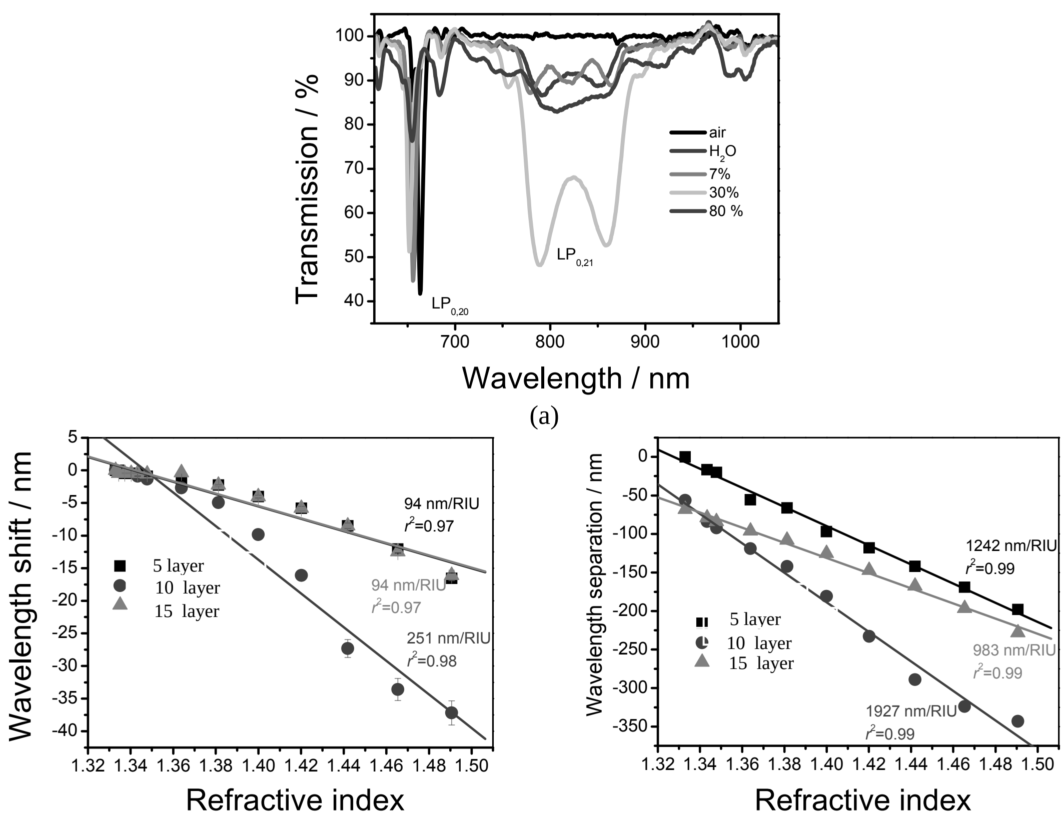
RESULTS AND DISCUSSION

After the deposition of each layer, the LPG transmission spectrum was recorded with the LPG immersed in the silica colloidal solution. The resonance band (at ca. 660 nm) corresponding to coupling to the $LP_{0,20}$ cladding mode suffered a blue shift with increasing film thickness, as shown in the insert in figure 2(a) , indicating the uniform growth of the LbL PAH/SiO_2 film. After deposition of 7 layers of PAH/SiO_2 coating, the phase matching condition for coupling energy to the $LP_{0.21}$ mode was satisfied, with the corresponding development of the resonance band at ca. 800 nm[12], and its subsequent development into dual resonant bands after the deposition of 10 PAH/SiO_2 layers, as shown in figure 2(a).



(a) (b)

Figure 2. (a) Evolution of the LPG transmission spectra in response to the deposition of the 10- layer PAH/SiO_2 film, measured with LPG immersed in the colloid SiO_2 solution. After the deposition of 4 layers, the resonance band at 800nm begins to from. The development of the resonance band saturates after the deposition of 7 layers, and thereafter the band splits into dual resonance bands as further layers are deposited. (b) Wavelength shift of the $LP_{0,20}$ resonance band observed when depositing the 10- layer PAH/SiO_2 film. The arrow indicates the direction in which the central wavelength changes with increasing layer number (0-10)



(b) (c)

Figure 3. (a), Transmission spectra of the LPG fibre coated with a 10-layer PAH/SiO_2 film measured in sucrose solutions with different concentrations; Dependence of (b), the wavelengths shift of the $LP_{0,20}$ resonance band, and (c), the wavelength separation of the $LP_{0,21}$ dual resonance bands, upon the refractive index of the sucrose solution for the three coating thicknesses: ■, 5- layer PAH/SiO_2 film; ●, 10-layer PAH/SiO_2 film; and ▲, 15-layer PAH/SiO_2 film.

When the uncoated 100 μm period LPG with a period of was immersed into water (RI=1.323), a blue shift of the $LP_{0,20}$ resonance band of 3 nm was observed. However, the sensitivity of the $LP_{0,20}$ resonance band was increased by the presence of the 10-layer PAH/SiO_2 coating, showing a blue shift of 10 nm when the LPG was immersed in water. In order to characterise the sensitivity of the LPG resonance bands to the surrounding RI, the LPG was immersed in solutions of sucrose of differing concentrations. The refractive index of the sucrose solutions was measured using an Abbe refractometer. Figure 3a shows transmission spectrum of the LPG coated with 10 layers of PAH/SiO_2, measured in solutions of different sucrose concentrations and thus of different RI.

The central wavelength of the $LP_{0,20}$ resonance band shows a linear response with a sensitivity of –251±18 nm/RIU, while the measurement of the wavelength separation of the dual resonance bands corresponding to coupling to the $LP_{0,21}$ cladding mode facilitates measurement of RI with sensitivity –1927±59 nm/RIU, as illustrated in Figure 3c. The sensitivity to RI greater than that of the cladding is a result of the presence of a low RI of the mesoporous film deposited on the LPG. Penetration of the analyte solution into film porous leads to the medium that intimately surrounds the optical fibre having a mean RI value lower than that of the original analyte solution.

CONCLUSIONS

The refractive index sensitivity of a 100 μm period LPG coated with a mesoporous coating of silica nanospheres has been studied. The sensitivity was shown to be dependent on the thickness of the deposited film, with a maximum sensitivity of 1927 nm/RIU over a wide RI range (1.3233–1.4906) when the separation of the $LP_{0,21}$ dual resonance bands were monitored. The low index mesoporous coating allowed extension of the range of refractive indices to which the LPG shows a response.

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