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Graphene oxide coated long period grating for optical sensing purposes

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Abstract. In this paper, fabrication and surrounding refractive index response of a graphene oxide (GO) coated long period grating (LPG) is presented. An improved version of the Hummer's method was followed for the synthesis of GO used in this work and GO sheets were immobilized on the LPG fibre surface by using an electrostatic self-assembly technique. In this initial performance evaluation, intensity and wavelength variations of the transmission loss bands of the GO coated LPG were recorded at room temperature and this sensor probe is introduced as a good candidate for the further development of selective biosensors.

1. Introduction

Fibre optic sensors play a major role in this technologically advanced, rapid changing era of Internet of Things and machine to machine communications. Optical fibre sensors is also a topic that has been researched extensively over the past decade. This is due to the very attractive features fibre optics contain compared to their electrical counterparts, such as immune to electromagnetic interference, small size, bio compatibility, multiplexing capability, fast response and the ability to withstand very hazardous and corrosive environments [1]. Fibre Bragg Grating (FBG), Tilted Bragg Grating, tapered fibres, interferometry are some of the major fibre optic sensing mechanisms that are being currently researched and Long Period Grating (LPG) based fibre optic sensors have been preferred and reported as a very simple and efficient way of developing refractive index based sensors due to their increased sensitivity to the refractive index of the surrounding material [2-3].

LPGs consist of relatively long period of gratings, usually in the region of several hundred micrometres that lead to a series of loss bands compared to a single loss band in FBGs. Light propagating in the core interferes with some of the co-propagating cladding modes and thus the resulting resonance bands depend on the effective refractive index of the propagating medium, which comprises of cladding refractive index, as well as the refractive index of the surrounding material around the cladding.

Graphene oxide (GO) is a two-dimensional single layered graphene sheet that contains some oxygenated groups such as hydroxyl and epoxy groups and it has attracted significant attraction after the discovery of graphene back in 2004. These oxygenated groups at the edges of the single layered graphene sheets in GO lead to a very rich surface chemistry with very unique and advantageous characteristics such as the ability to be easily dispersed in aqueous or organic solvents and the ability to perform various kinds of interactions with different kinds of bio-molecules, nano-structures and biological species [4]. This makes GO a very attractive and interesting material to be used in the field of biosensors.

In this paper, we present an external refractive index sensor with GO coated LPG as its sensing mechanism. GO has been immobilized on the LPG fibre surface by using electrostatic self-assembly with a dip coating technique. Response of the GO coated LPG for variations in the external refractive index is presented in this initial stage of experimental results. We propose this probe as a potential mechanism to develop effective biosensors by functionalizing the coated GO layer with other nanomaterials.

2. Theoretical Background

The minimum transmission of loss bands in a LPG is governed by the following equation [3].

$$T_i = 1 - \sin^2 (\kappa_i L) \tag{1}$$

The minimum transmission of the i^{th} order cladding mode is depicted by T_i. The coupling coefficient of i^{th} order cladding mode is obtained by the overlap integral of the core and cladding mode and by the amplitude of the periodic modulation of the mode propagation constants and it is depicted by K_i. L represents the length of the LPG region. Various types of changes induced by the surrounding medium of the LPG affects its evanescent field, which leads to changes in the coupling coefficient and this will eventually lead to intensity variations in LPG attenuation bands. Any noticeable intensity variations in any of the LPG attenuation loss bands can be explained by this phenomenon.

The core mode propagating in the forward direction couples with the co-propagating cladding modes, when coupling coefficients are satisfied and the resonance wavelength λ_{res} can be expressed by the following equation.

$$\lambda_{res} = (n_{core}^{eff} - n_{cladd.m}^{eff})\Lambda_{LPG} \tag{2}$$

Effective refractive index of fundamental core mode is represented by n_{core}^{eff} and the effective refractive index of m^{th} order cladding more is represented by $n_{cladd.m}^{eff}$. Λ_{LPG} is the grating period of the LPG. This equation dictates that the resonance wavelength of any of the attenuation bands depends on the effective refractive index of the core and also of the cladding. Effective refractive index of the cladding modes depends on cladding refractive index, as well as the refractive index of the surrounding medium. Variations of the wavelength peaks of resonance loss bands of a LPG can be explained by this phenomenon.

3. Fabrication of the sensor

The LPG used in this experiment was fabricated by exposing Ge/B doped photosensitive fibre (Fibercore 1250/1500) to a KrF based 248 nm excimer laser beam through an amplitude mask with a period of 400 µm. Pulse energy and the pulse frequency were fixed at 10mJ and 150 Hz, respectively. The length of the LPG was selected as 3cm. GO powder used in this research work was synthesized by following an improved version of the widely reported Hummer's method. Detailed steps of this method can be found in [5]. 0.5 mg of this prepared GO powder was sonicated in 10 ml of distilled water to make the GO dispersion that was coated on the LPG surface. Afterwards, the dispersion was centrifuged at 1500rpm for 20 minutes and the supernatant was collected to prepare a GO solution with most number of single layered GO sheets. Before coating GO, the LPG fibre surface was cleaned thoroughly by immersing in piranha solution for 15 minutes and washing with copious amount of distilled water for 10 minutes. This cleaned fibre surface was oven dried at 100 °C (for half an hour) before immersing in an APTES solution (5% V/V in ethanol) for half an hour to functionalize the fibre surface with silane groups. This creates a positively charged fibre surface (since APTES is positively charged). Afterwards, this functionalized LPG was dipped in the above prepared GO aqueous dispersion for three hours to coat GO sheets on the

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fibre surface by electrostatic self-assembly process. Figure 1 shows the microscopic image of the coated LPG surface compared to an uncoated LPG surface. It is evident that a clear coating of GO is visible under an optical microscope. Figure 2 depicts the LPG transmission spectrum before and after coating of GO. By these results it was concluded that GO was successfully coated on the LPG fibre surface.

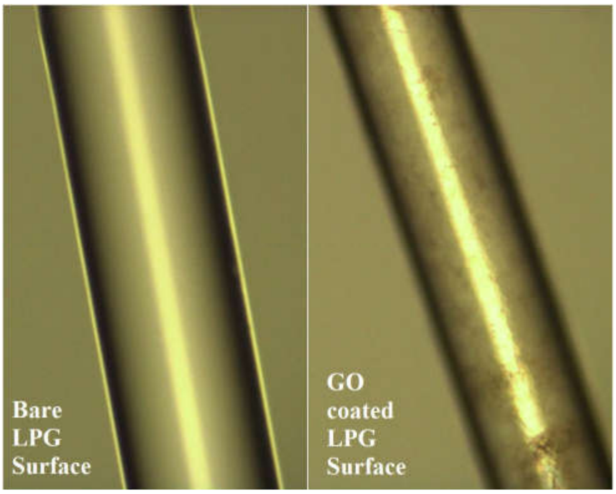

Figure 1. Microscopic image of GO coated and uncoated LPG fibre surface

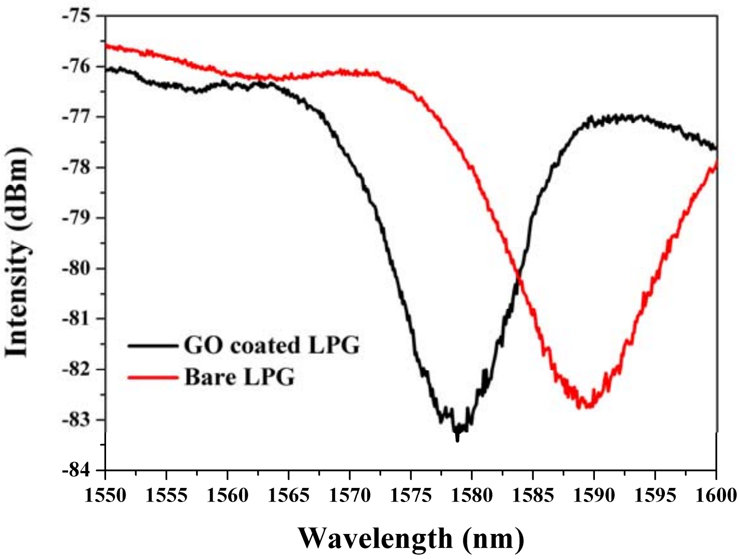

Figure 2. Transmission spectra of the LPG before and after coating

4. Experimental Setup

Figure 3 illustrates the experiment setup used to investigate the external refractive index response of the GO coated LPG. One end of the GO coated LPG was connected to a broadband light source and the other end was connected to an optical spectrum analyser to record the transmission spectrum. GO coated LPG was then mounted on two fibre holders. A microscopic glass slide was positioned on a height adjustable lab jack, which was placed under the GO coated LPG. Different refractive index solutions (water and IPA) were added on to the microscopic slide in a dropwise manner using a disposable pipette.

Then the microscopic slide with the liquid was pushed up using the lab jack so that only the solution was covering the GO coated LPG. At this point, the transmission spectrum of the GO coated LPG was recorded using the spectrum analyser. After each solvent, the microscopic slide and the GO coated LPG was cleaned and dried thoroughly with a KimWipe paper so that the transmission spectrum returned to its original position. All these experiments were done at room temperature.

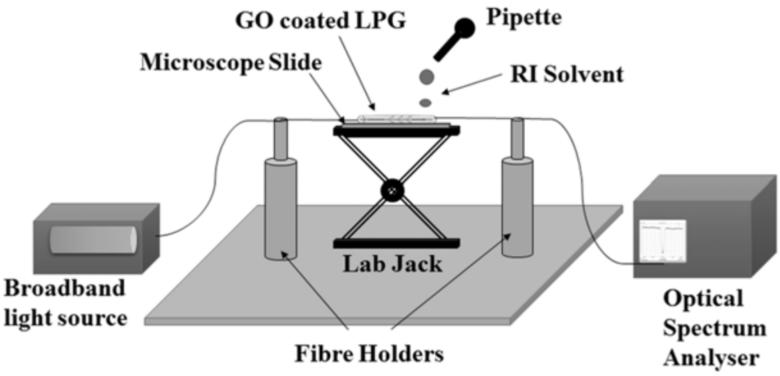

Figure 3. Experimental Setup

5. Results and Discussion

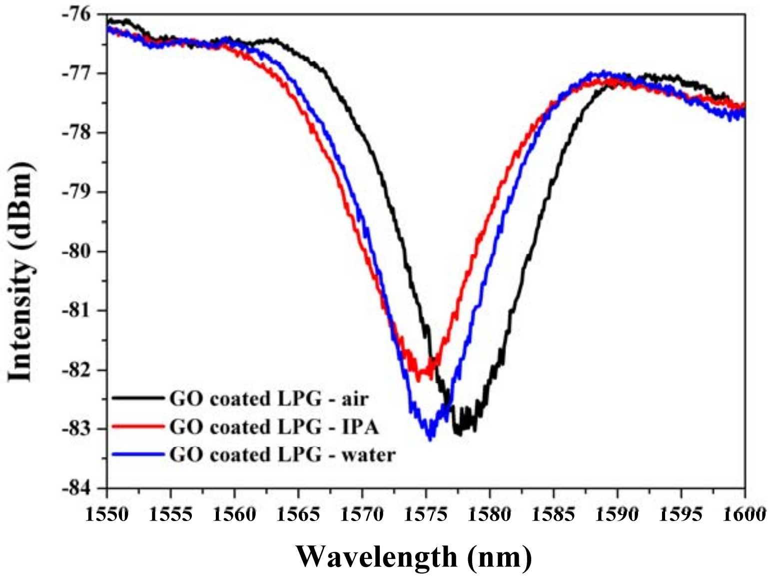

Figure 4. External refractive index response of the GO coated LPG

Highest order resonance loss band of the GO coated LPG with lowest attenuation point at 1578 nm wavelength was selected as the interrogating loss band due to its improved sensitivity compared to other attenuation bands present in the LPG. Using the above mentioned experimental setup, the external refractive index response of the GO coated LPG was investigated by submerging the sensor probe in water and IPA (Isopropyl alcohol), which contains refractive indices of 1.33 and 1.37, respectively. In the case of the sensor probe submerged in water (which corresponds to an external refractive index of 1.33), the peak resonance wavelength of the GO coated LPG performed a blue shift of 3nm while the intensity of the loss band remained constant. When the GO coated LPG probe was submerged in IPA (which corresponds to an external refractive index of 1.37), the peak resonance wavelength of the loss band exhibited a blue shift of 3.5 nm while the intensity of the loss band was increased by 1dB. Based on this initial experiment carried out, it was evident that the transmission spectrum of the GO coated LPG is capable of exhibiting intensity and wavelength variations based on the changes in external refractive index. As described in the introduction, the coated GO layer can be further functionalized with various kinds of biomolecules and nanostructures to develop selective biosensors as shown in [6]. Therefore, we propose this GO coated LPG sensor probe as a promising, comparatively cheap and easy to fabricate sensing platform for the development of biosensors.

6. Conclusion

A sensing probe based on a GO coated LPG was presented as a potential platform to develop biosensors by exploiting the unique features of GO. GO was immobilized on the LPG surface by functionalizing it with APTES to achieve electrostatic self-assembly of GO sheets. External refractive index response of the GO coated LPG was investigated at room temperature by submerging it in liquids with different refractive indices and it was proven that the GO coated LPG exhibits variations in the intensity and wavelength of the transmission loss bands based on the changes of the surrounding refractive index. Based on these promising results, we would like to present GO as a very attractive material for sensing. Furthermore, we also propose this GO coated LPG probe as a potential platform to develop biosensors by functionalizing the coated GO layer with other biomolecules and nanostructures to detect and measure biological species and parameters.

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