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Graphene oxide-coated Long Period Grating-based fibre optic sensor for relative humidity and external refractive index

Kasun Prabuddha Dissanayake, Weiping Wu, Hien Nguyen, Tong Sun and Kenneth T. V. Grattan

Abstract— **Graphene oxide is a very attractive material for refractive index and humidity sensing due to its unique two-dimensional structure, which results in faster response times and improved sensitivity over alternative materials. In this paper, response of a graphene oxide-coated long period grating-based sensor to changes in relative humidity and external refractive index (as well as temperature to provide a correction for any changes) is reported. In fabricating the probes, an improved Hummer's method was used to synthesis the graphene oxide dispersion used as its basis, allowing coating of a functionalized long period grating by using a dip-coating technique. A consistent and stable response of the resonance dip intensity of the graphene oxide-coated long period grating was observed to the change in humidity, achieving a sensitivity of 0.15 dB / %RH with a linear correlation coefficient of 0.980 over the relative humidity range from 60%RH to 95%RH, at room temperature (25 °C). A blue shift of the resonance band wavelength was recorded when the sensor was exposed to varying temperature conditions from 25 °C to 70 °C and the response was found to be linear, with a correlation coefficient of 0.997. When evaluating its performance as an external refractive index sensor, sensitivities of ~17dB/RIU in the lower refractive index region (1.33-1.38) and ~55 dB/RIU over the higher refractive index region (1.40-1.45) were achieved. The graphene oxide-coated long period grating sensor probe performed well, showing a good stability and repeatability over a number of test cycles in the performance evaluation carried out.**

Index Terms— **humidity sensor, refractive index sensor, long period grating, graphene oxide, optical fibre**

I. INTRODUCTION

OVER the past decade, fibre optic sensors have become popular in many practical applications due to their unique characteristics such as immunity to electromagnetic interference, biocompatibility, easy multiplexing capability, small size, and their ability to be used in very hazardous and corrosive environments [1]. One of these applications is measuring relative humidity (RH), which has particular significance in structural health monitoring, the semiconductor industry, the medical and health sectors, agriculture, food processing, storage, and many other industrial applications. The above mentioned features of fibre optic sensors illustrate their significant advantage over their electrical counterparts, when it comes to humidity measurement [2]. Various types of humidity sensors based on in-fibre gratings have been reported before [1]. In the area of refractive index (RI) sensing, fibre optic sensors based on Long Period Gratings (LPGs) have been preferred over other optical fibre sensor designs due to their high sensitivity to surrounding refractive index [3]. Due to the RI modulations present in LPGs, light propagating in the core interferes with some of the co-propagating cladding modes and the respective resonance bands can be noticed in the transmission spectra. These resonance bands depend on (and change with) the effective refractive index of the propagating medium, which comprises the known RI of the cladding, as well as the RI of the material coated on the fibre that surrounds the cladding. This coated LPG approach has been used as the basis of the sensor probes reported here.

Recently, research on various types of graphene oxide (GO)-based optical sensors has been reported [4, 5], especially in humidity sensing [6]. GO is a two-dimensional nanomaterial that contains sp^2 and sp^3 hybridized carbon atoms along with many oxygen rich functional groups, such as epoxides and carboxylic groups, on the edges and the basal plane of a single layered graphene sheet. These oxygen functionalized groups allow GO to undergo various kinds of chemical and bio-chemical interactions with other biological molecules, biological structures, and different kinds of nanostructures, leading to a very rich surface chemistry diversity. Even though GO as a material has been known for some time, it has attracted attention as a candidate material for range of applications, only after the discovery of graphene in 2004 [7]. GO can be easily synthesized by exfoliating graphite in a very strong oxidizing medium and it can be dispersed in a wide range of organic and inorganic solvents due to its unique surface chemistry, and thus these aqueous dispersions can be used to coat GO on fibre surfaces. The combination of unique and scientifically interesting characteristics such as these, combined with a low cost synthesis process, means that GO presents itself as a very attractive candidate for various kinds of chemical and bio-

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chemical sensors.

In this paper, a fibre optic sensor scheme that comprises a GO-coated LPG, which lies at the core of the essential sensing principle used, is presented. To achieve a stable coating, GO flakes were immobilized on the silica fibre surface by functionalizing the LPG surface area with polymerizable acrylate groups using 3-(trimethoxysilyl)propyl methacrylate, before coating GO onto the fibre surface. The transmission spectra of this GO-coated LPG can then be used as the basis of the sensor scheme illustrated, operating over a relative humidity range from 60%RH to 95%RH. Given the need for providing correction for temperature excursions in the use of this probe for the measurement of other parameters, the temperature dependency of the developed sensor was also investigated and its performance has been analysed. Additionally, the sensitivity of a GO-coated LPG-based sensor to changes in the surrounding RI, monitored over the important RI range from 1.329 to 1.453, was investigated and results on its performance are presented in this paper.

II. SENSING PRINCIPLE

The working principle of the developed sensor is based on the analysis of light propagating in the cladding modes, affected by the deposited thin film of GO. When the light from a broadband source is coupled to a LPG, the RI modulation in the core enables the light propagating in the fundamental LP_{01} core mode to be coupled with the LP_{0m} (m=1, 2, 3…) co-propagating cladding modes at discrete wavelengths. High attenuation of these cladding modes results in the formation of several resonance bands at these discrete wavelengths in the LPG transmission spectrum. The mode coupling mentioned above occurs when following phase matching conditions are satisfied [9]

$$\beta_{core} - \beta_{clad}^m = \frac{2\pi}{\Lambda} \qquad (1)$$

where β_{core} is the propagating constant of the fundamental core mode, β_{clad}^m is the propagating constant of the mth order cladding mode and Λ is the grating period. These propagation constants can further be expressed as follows [10].

$$\beta_{core} = \frac{2\pi}{\Lambda} n_{core}^{eff}, \qquad \beta_{clad}^m = \frac{2\pi}{\Lambda} n_{clad,m}^{eff} \qquad (2)$$

Based on (1) and (2), the resonance wavelength λ_{res} of an attenuation band caused by the light coupling between the fundamental core mode and the respective co-propagating cladding mode can then be written as

$$\lambda_{res} = (n_{core}^{eff} - n_{cladd.m}^{eff})\Lambda_{LPG} \qquad (3)$$

where n_{core}^{eff} and $n_{cladd.m}^{eff}$ are the effective refractive indices of the fundamental core mode and the mth cladding mode, respectively. Λ_{LPG} is the grating period of the LPG. While the RI of the core remains unaltered, when a thin coating of a

substrate is applied on the LPG, it can be seen (and thus modelled) as a double-clad waveguide. The effective refractive indices of the cladding modes are formed by the combination of both the cladding RI and the RI of its surrounding material, in this case GO. Based on this, the influence of the external RI of the material that surrounds the cladding can be expressed as [11]

$$\frac{d\lambda}{dn_{sur}} = \frac{d\lambda}{dn_{clad,m}^{eff}} \cdot \frac{dn_{clad,m}^{eff}}{dn_{sur}} \qquad (4)$$

where λ is the wavelength of the attenuation band and n_{sur} is the RI of the surrounding material. Due to this dependency, the changes occurring in the external RI can be monitored through the changes of the transmission spectra of the resonance loss bands, which enables a LPG to be used as a RI sensor.

When the external RI is smaller than that of the cladding, the phase matching conditions mentioned above are satisfied and according to (3) and (4), the effective RI of the cladding will increase while the core RI remains the same, which results in a blue shift of the resonance wavelength. However, when the external RI is higher than that of the cladding, such as is the scenario with GO, these phase matching conditions will no longer be satisfied. The cladding-surrounding medium interface will lose its total internal reflection condition, and thus guided modes in the cladding will act as radiation modes or leaky cladding modes. Under these conditions, the change in the intensity of resonance loss bands, which is affected by the amount of light reflected back at the cladding-surrounding medium interface, will supersede the change in the wavelength shift.

The minimum transmission of loss bands in a LPG is governed by the equation [12],

$$T_i = 1 - sin^2 (K_i L) \qquad (5)$$

where T_i is the minimum transmission of the ith cladding mode and K_i is the coupling coefficient of the ith cladding mode, which 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. L is the length of the LPG in the probe. As explained above, the RI change induced by the changes in the surrounding environment, e.g. the GO-coated layer, affects the evanescent field (mode field diameters and mode propagation constants of cladding modes), thus the coupling coefficients, and this eventually leads to noticeable (and measurable) intensity changes in the LPG loss bands. This underpins the working principle of the GO-coated LPG sensor schemes presented in this work.

III. MATERIALS AND EXPERIMENTAL METHODS

Graphite flakes and sodium nitrate ($NaNO_3$) used in the experiments were purchased from Alfa Aesar and Acros Organics, respectively. Hydrogen peroxide (H_2O_2) was purchased from Fisher Scientific. All other chemicals were purchased from Sigma-Aldrich (UK). All of these chemicals

were of analytical grade, and thus no further purification was needed.

A. GO Synthesis

An improved Hummer's method [13] was followed to synthesize the GO used in this work. In this approach, first, graphite flakes (500 mg) and $NaNO_3$ (500 mg) were mixed in concentrated H_2SO_4 (22.5 mL) in an ice bath (0 ℃ - 4 ℃) for 4 hours. Afterwards, 3 g of $KMnO_4$ was added very slowly to the mixer before adding 46 mL of DI water and stirring for another 1 hour in the same ice bath. The mixture then was stirred again for 2 hours at 35 ℃ and afterwards, kept in a reflux system at 98 ℃ for 10-15 minutes, following which the temperature was changed to 30 ℃. At this point, a brown coloured solution was obtained. The mixture was again stirred for 2 hours at room temperature. Afterwards, 13.3 mL of 30% H_2O_2 was added very slowly (due to high rate of reaction) and the mixture turned to a very bright yellow colour, as shown in Fig. 1 (a). Following that, 100 mL of DI water was added to the prepared solution and it was stirred for another 1 hour at room temperature. The mixture was then kept for 24 hours, without stirring, until the particles were seen to settle at the bottom of the vessel. At this point, water was filtered out using a paper filter and the resulting mixture was washed repeatedly by sonication and centrifugation with 10% HCl and DI water several times until a gel-like substance was formed. The resultant gel-like substance was vacuum-dried for 8 hours to create the GO flakes, as shown in Fig. 1 (b).
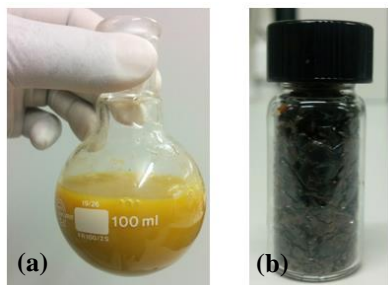

Fig. 1. (a) Bright yellow colour solution obtained after oxidizing graphite flakes first with $KMnO_4$ and then with H_2O_2 (b) GO flakes obtained

B. LPG Fabrication

A B/Ge co-doped photosensitive fibre (purchased from Fibercore, UK) was exposed to the light from a 248 nm Krypton Fluoride (KrF) excimer laser with a pulse energy of 10 mJ and a pulse frequency of 175 Hz through a metal amplitude mask with a period of 400 µm to create the 25 mm long LPG. After the inscription process, the LPG was annealed at 100 ℃ for 2.5 hours to stabilize its optical properties.

C. GO Deposition on the Fibre Surface

0.5 mg of GO flakes, obtained as described above, was dissolved in 10 mL of distilled water to prepare the GO aqueous solution. After stirring for 12 hours, the GO aqueous dispersion was sonicated for 1 hour before being centrifuged for another 20 minutes at 3000 rpm. This centrifugation process was repeated three times and the supernatant was taken to achieve a high percentage of single layered GO flakes in the solution. To

confirm that the prepared solution contained a high number of single layered GO sheets, the UV/Vis spectrum of the GO aqueous dispersion was measured, as shown in Fig. 2.
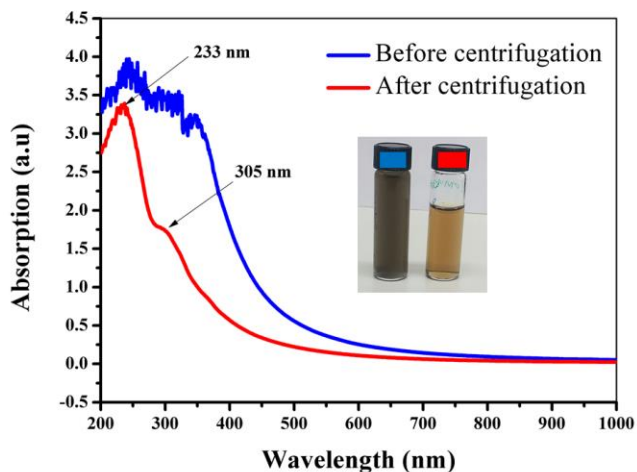

Fig. 2. UV/Vis spectrum of the prepared GO aqueous dispersion before and after centrifugation

UV/Vis spectrometry is a simple and easy way of measuring the quality of GO aqueous dispersions created. Two important characteristics of GO can be observed from its UV/Vis spectrum. The first is the 'shoulder' at a wavelength of ~310 nm, which corresponds to the $n - \pi *$ transitions. The other most important feature that can be observed is the peak at ~230 nm, which corresponds to the $\pi - \pi *$ transitions. This particular peak is related to the C-O bonds and number of nanoscale sp^2 clusters appeared on the GO sheets, which is directly related to the amount of single layered GO sheets present in the aqueous dispersion [14]. Therefore, the intensity of this peak at ~230 nm can be used as an indicator of the quality of the prepared GO aqueous dispersion. As is illustrated in Fig. 2, a distinctive absorption peak at 233 nm was observed after the GO dispersion was centrifuged, which confirmed that a high number of single-layered GO sheets was obtained, avoiding the creation of multilayers.

Before coating the fibre, the LPG was immersed in a $H_2SO_4:H_2O_2$ (7:3) solution at room temperature for 1 hour, followed by washing it thoroughly in an ultrasonic bath with distilled water and drying at 100 ℃ for half an hour to clean the coating surface and leave it with exposed hydroxyl groups which facilitate bonding of a silane agent. Afterwards, the LPG surface was functionalized by immersing in a 3-(trimethoxysilyl)propyl methacrylate solution for 30 minutes followed by drying at 70 ℃ for half an hour to stabilize the immobilized silane layer. GO was then coated on the functionalized LPG surface by immersing in 200 µL of prepared GO aqueous dispersion and drying at 45 ℃. Deposition of GO sheets on the LPG surface occurs due to the π-π non-covalent interactions that take place between the π bonds present at the end of 3-(trimethoxysilyl)propyl methacrylate groups and those of the GO sheets. Immersing in the GO aqueous dispersion was repeated three times to achieve a brownish black coloured coating, which is clearly visible in the microscopic image (together with a bare LPG for cross-comparison) shown in Fig. 3. By comparing the microscopic

images taken, an approximation of the coating thickness was made, with this estimated to be ~ 1.5 µm. The transmission spectrum of the LPG, obtained both before and after coating, is illustrated in Fig. 4. These results indicated that the GO sheets prepared were successfully coated on the LPG surface in this key fabrication step for the probe.
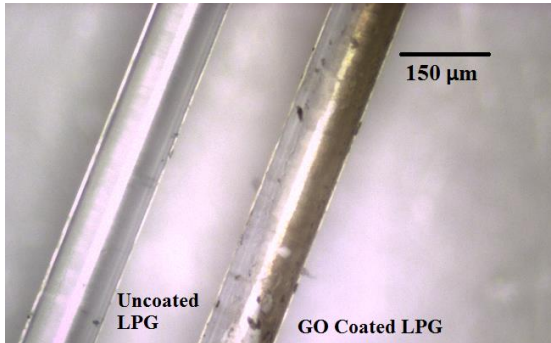

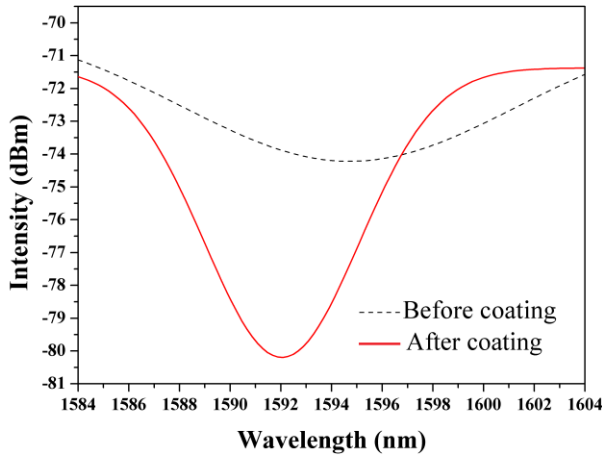Fig. 3. Microscopic image of GO-coated LPG (right) and a bare, uncoated LPG (left)

Fig. 4. Transmission spectra of the LPG before and after coating in the wavelength region 1584 nm to 1604 nm

D. Experimental Setup for Evaluating the Humidity Sensitivity

Fig. 5 illustrates the experimental setup used for evaluation of the sensor performance as a function of humidity (and temperature) changes.
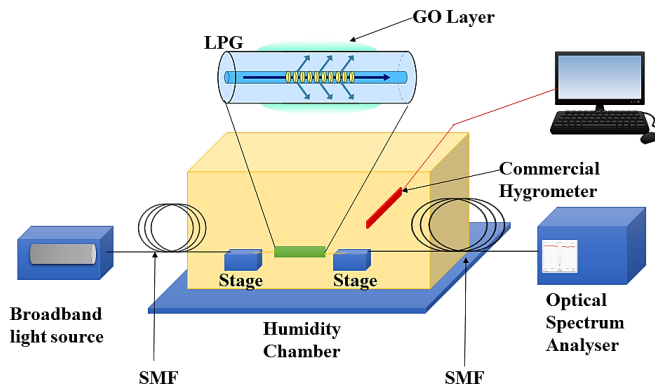

Fig. 5. Experimental setup used for humidity sensing

To do so, one end of the GO coated LPG was connected (via SMF-Single Mode Fibre) to a broadband light source (OceanOptics LS1) and the other connected to an Optical Spectrum Analyzer (Yogokawa AQ6370C) to record the transmission spectra. The spectrometer offers a wavelength measurement range from 600 nm to 1700 nm and a spectral resolution of 0.5 nm. The GO-coated LPG was mounted on two stages (as illustrated in Fig. 5) inside the humidity chamber (Binder KBF 115) to minimize the errors caused by external strain and bending effects. The humidity chamber was used as it was capable of rapid changes of relative humidity over the range from 5%RH to 95%RH, at a particular (and known) temperature value (25 ℃ to 100 ℃). The system was allowed to stabilize before each transmission spectrum was recorded at each particular humidity level chosen. A commercial hygrometer was used to cross-calibrate the sensors to shown performance, in terms of temperature and relative humidity.

E. Experimental Setup for Evaluating External RI Changes

To record the transmission spectra of the GO-coated LPG, seen to vary with changes in the external RI, one end of the coated sensor probe was connected to the broadband light source and the other to the Optical Spectrum Analyzer (both devices are as same as described above), as depicted schematically in Fig. 6. To evaluate the performance of the sensor at various external RI values, the GO-coated LPG sensor probe was carefully mounted on two fibre holders, which were stationed on an optical bench. A microscopic slide was placed on a height-adjustable laboratory jack and the coated LPG forming the basis of the sensor was placed carefully on top. This set up facilitated allowing a few drops of sample material (organic solvents with known refractive indices) to be applied on the fibre surface using a pipette, so that the coated area of the LPG was submerged in the solvent. After each test was completed, the LPG and the microscopic slide were cleaned and dried carefully with lint-free paper (Kimwipe) before being exposed to the next solvent sample of different refractive index. Prior to the next solvent sample being applied to the sensor probe, the spectral output was monitored using the spectrometer – this being observed until the spectrum of the coated LPG returned to its original position (i.e. when the coating was exposed to air), to ensure that no residue of the solvent used remained.
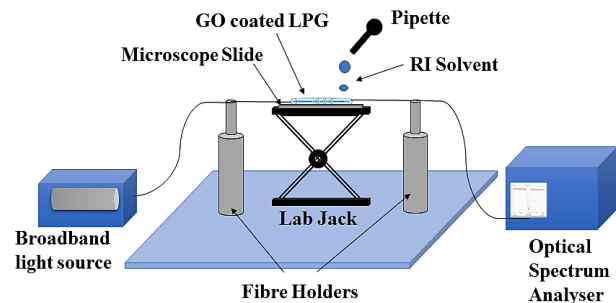

Fig. 6. Experimental setup used for external RI sensing measurements

IV. Experimental Results And Analysis

Preliminary data on the humidity and temperature response of this GO-coated LPG was originally presented in [15], but it is now extended showing the results of more extensive evaluation and testing.

A. Humidity Response

Before monitoring the humidity-sensitive response of the GO-coated LPG sensor probe, the humidity response of a bare LPG was recorded for cross-comparison purposes. The RH response of a bare LPG, fabricated using the same process mentioned above, is illustrated in Fig. 7.
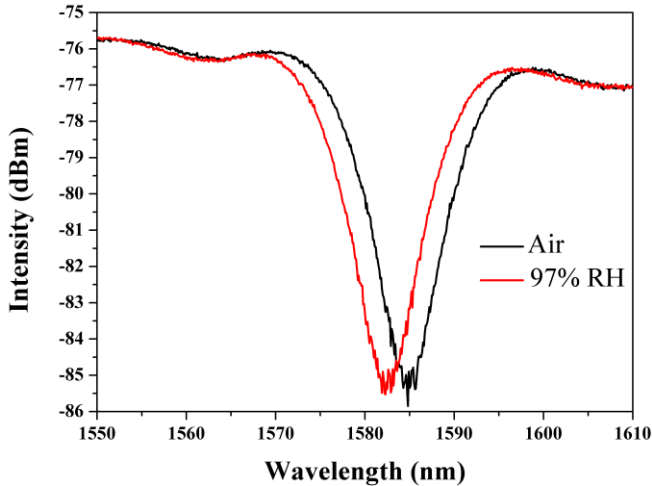

Fig. 7. Humidity response of a bare LPG used for cross-comparison

For this experiment, the same laboratory setup was used (as illustrated in Fig. 5), but this time, replacing the GO-coated LPG with a bare LPG. Initially, the temperature was fixed at 25 ℃. There wasn't any noticeable change observed in the transmission spectrum until the relative humidity level reached ~97% RH, at which point, a slight change in the resonance band intensity and wavelength was seen, as shown in Fig. 7. At very high humidity levels (such as >97% RH), water droplets can become deposited on the fibre surface, which leads to a measurement error due to strain being applied as a result, to the LPG. The wavelength shift observed in this test can therefore be explained by this effect.
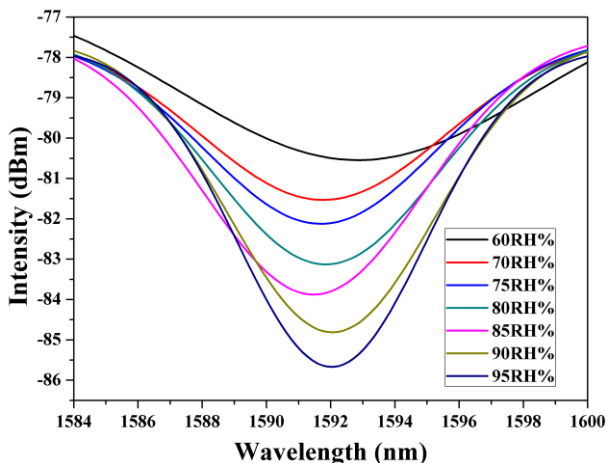

Fig. 8. Transmission spectra of GO coated LPG at different relative humidity levels

To observe the humidity response of the GO-coated LPG sensor, the temperature of the humidity chamber was initially fixed at 25 ℃ and the relative humidity level was changed over the range from 20%RH to 95%RH. Fig. 8 illustrates the transmission spectra of the resonance band of the GO-coated LPG, monitored at a centre wavelength of 1592 nm, for various different levels of humidity in the test chamber.

A minimal intensity or centre wavelength shift was observed for humidity levels lower than 60%, but beyond that, it was clearly observed that with increasing humidity, the intensity of the resonance dip increased, with an accompanying slight shift of the centre wavelength. This observed active response range of 60%RH to 95%RH corresponds well with the performance of a reduced GO-based humidity sensor, reported in the literature [16]. The unique characteristic of the GO used in this work is however, that by contrast it is super-permeable to water molecules [17]. When moisture molecules are present inside the GO material, a change of optical properties of GO layer (such as is reflected in the RI and coupling coefficient changes) is induced, and hence the change of centre wavelength and the intensity of the resonance loss band.
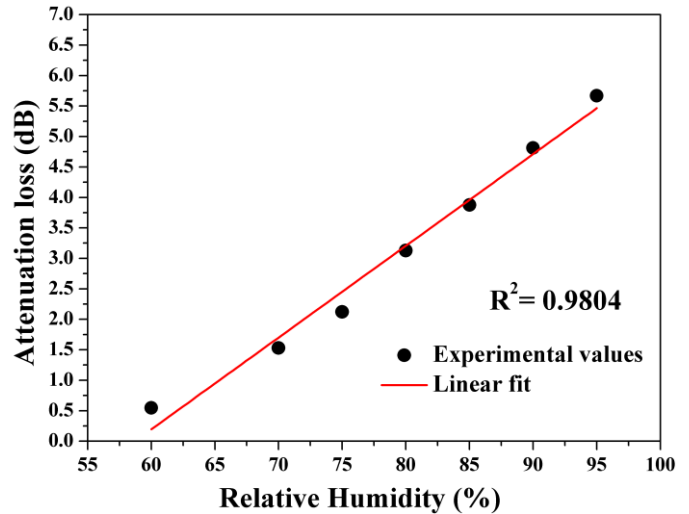

Fig. 9. Intensity variations of the resonance band centred at 1592 nm at different relative humidity levels

Fig. 9 depicts the intensity change of the resonance band centred at 1592 nm, for varying RH levels. The minimum intensities of -80.5 dBm and -85.7 dBm were recorded at 60%RH and 95%RH, respectively. A good linear correlation between RH level and the resonance band intensity was observed with a linear correlation coefficient of 0.980. This leads to a RH sensitivity of 0.15 dB/%RH over the range of 60%RH to 95%RH at 25 ℃.

Comparing the response of the bare LPG (illustrated in Fig. 7, which recorded a negligible intensity variation over a relative humidity range of 60% to 97%), with the response of the GO-coated LPG, tested over the humidity range recorded in Fig. 8, it is evident that the use of the GO coating has considerably improved the sensitivity of the LPG-based probe in these tests performed under varying RH conditions.

B. Temperature Response

The effect of temperature on the sensor probe further was investigated (as a correction for thermal changes may be needed in practical applications of the sensor) by fixing the relative humidity level at 45%RH, and changing the temperature from 25 ℃ to 70 ℃. Fig. 10 illustrates the transmission spectra, showing the resonance band dip at various discrete temperatures, while Fig.11 depicts the centre wavelength shift of the same resonance band over the same temperature range from 25 ℃ to 70 ℃. Centre wavelengths of 1594.2 nm and 1579.7 nm were recorded at 25 ℃ and 70 ℃, respectively, showing the spectral range of the device.
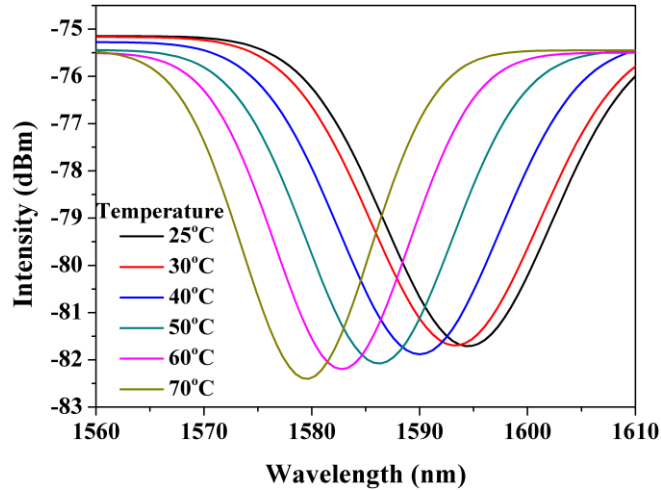

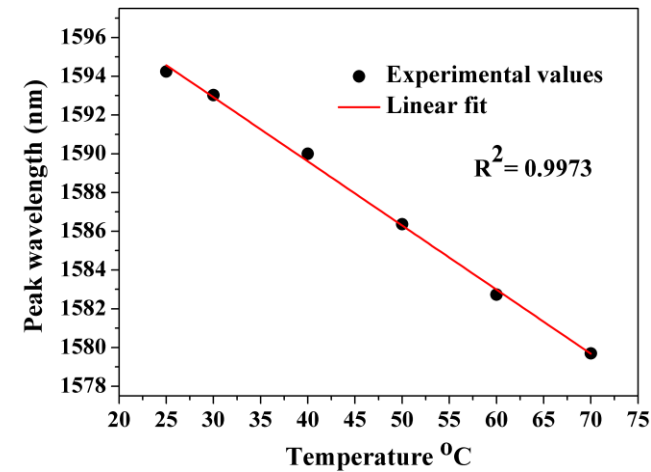Fig. 10. Transmission spectra of GO coated LPG at different temperatures

Fig. 11. Centre wavelength of the resonance band as a function of temperature

A very good linear correlation between the temperature change and the centre wavelength shift was observed, with a temperature sensitivity of 0.32 nm/℃ and a linear correlation coefficient of 0.997. A slight change of the resonance band intensity (0.6 dB) was observed when the temperature was increased from 25 ℃ to 70 ℃.

By comparison, when an uncoated LPG fabricated using the same setup (and the same amplitude mask) was exposed to increasing temperature from 25 ℃ to 70 ℃, a similar wavelength shift was observed (but without the small change in the resonance band intensity seen). The temperature change induces a variation in the grating period (by changing its length), and thus creating a shift in the resonance wavelength. The material, in this case GO, also contributes to the resonance band characteristics by changing its effective refractive index as a result of the temperature changes. The change in the resonance band intensity, which was observed for the GO-coated LPG (but not in the uncoated LPG), can thus be noted. The centre wavelength shift recorded as a function of temperature changes can be used for effective temperature measurement and, as needed, for temperature compensation of the humidity sensor, where humidity is measured under varying temperature conditions. The ability to measure both parameters (humidity and temperature) configured this way in the same sensor probe is an advantage of this approach over fibre optic humidity sensors that require two gratings [2] – in that way to compensate for the error caused by varying temperature conditions.
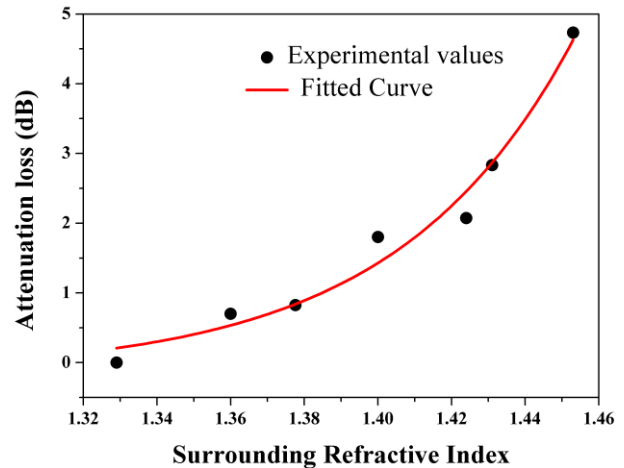
C. External RI Sensitivity

Fig. 12. Intensity loss of the resonance band against external RI

Fig. 12 illustrates the response of a GO-coated LPG to observed changes in the surrounding RI. To be able to measure a range of external RI values, different organic solvents (of known and different RI values ranging from 1.329 to 1.453) were used with the setup described in Fig. 6 to record the sensor performance. These measurements were carried out in an indoor environment at 25 ℃ where relative humidity levels were monitored throughout the experiment to make sure that the values remained constant (so that any wavelength shift or intensity loss of the resonance band caused by temperature and humidity changes was insignificant). Fig. 12 shows a nonlinear response of the resonance band intensity with increasing RI values. These observations are consistent with previously reported studies with GO [18] and carbon nanomaterial [19] based fibre optic RI sensors. Calculations based on the figure showed that in the lower region of RI values (from 1.33 to 1.38), an approximate sensitivity of 17dB/RIU has been achieved, compared to the higher RI region (values from 1.4 to 1.45), where an approximate sensitivity of 55 dB/RIU was achieved.

V. Discussion and Conclusions

A novel approach to the design of humidity sensor probes and an external refractive index monitors has been presented, based on a GO-coated LPG. The probes operate by exploiting the super-permeability of the GO used to water molecules, and its dependency on the thermo-optic properties in the presence of such water molecules. Results obtained show high sensitivity and where direct comparison can be made, comparability with the performance of probes using similar carbon nanomaterials [16, 18 and 19]. The fabrication of such probes has been made convenient by using an improved Hummer's method for the GO synthesis. 3-(trimethoxysilyl)propyl methacrylate was coated on the LPG surface to immobilize coated GO flakes to achieve a stable sensor performance. A satisfactory linear response for the humidity sensor was recorded over the 60%RH to 95%RH range, with a sensitivity of 0.15 dB / %RH at 25 ℃. A good linear correlation between temperature and the centre wavelength shift was also observed for a temperature range of 25 ℃ to 70 ℃. The sensor performed with a consistent repeatability and stability. Sensitivities of 17 dB/RIU and 55 dB/RIU have been achieved in two different external RI regions as stated above.

Research is continuing to enhance the performance of this sensor design, proposing improvements to achieve a better performance, which include optimizing the coating thickness, to achieve the highest sensitivity. Previous research has shown that there is an optimal thickness for a coating material that has a greater RI than that of cladding [20] and especially carbon nanomaterials [21]. Thus, with an optimization of coating thickness, changes in the wavelength shift can also be achieved along with the intensity shift of the resonance bands, allowing for the development of dual parameter sensors. Further refinement can come from the variation of the period of the grating, to achieve a higher sensitivity, as higher order modes are bound to be more sensitive to the external RI. Another potential approach is to functionalize the GO layer deposited on the fibre surface with various molecules with functional groups or bio-molecules, to develop highly selective chemical and bio chemical fibre optic sensors [22]. Furthermore, GO could be reduced to achieve the characteristics of graphene by applying a chemical or thermal reduction process and in that way, reduced GO could add another dimension to optical fibre sensors, as has been introduced by Sridevi et al. in [23].

In conclusion, based on the promising results obtained and considering the potential of GO as a sensing material as discussed above, GO has been proved to be a very attractive sensing material for optical fibre sensors. The on-going work shows considerable promise to increase the device sensitivity by varying the RI of the GO solution, and also by experimenting with the effects of different coating thickness.

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