Nonadiabatic Tapered Optical Fiber with GO/PVA Nanostructured Sensitive Coating for Humidity Sensing Application

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Abstract. A non-adiabatic tapered fiber sensor coated with humidity-sensitive graphene oxide (GO)/ Poly (vinyl) alcohol (PVA) nanocomposite film for humidity sensing application was proposed. GO/PVA film was deposited onto the tapered region by dip-coating technique. The surface morphology of the coating film was characterized through Field Emission Scanning Electron Microscope (FESEM). When exposed to percent relative humidity (RH) ranging from 20 to 99.9 % RH, the sensor exhibited sensitivity for both untapered and tapered fibers at $-0.00132 \pm 0.00043$ a.u. (%)$^{-1}$ and $0.00106 \pm 0.00008$ a.u. (%)$^{-1}$, respectively. The contribution of GO/PVA composite film in enhancing sensor sensitivity was proven, which was $0.00624 \pm 0.00033$ a.u. (%)$^{-1}$ with percentage of sensitivity boosting up to 15.86 % when compare to the uncoated ones.

1. Introduction

Relative humidity (RH) sensing application through fiber-based sensor has been discovered as early as 1989 by Mitschke [1]. Fiber-based sensor has become a favor than the conventional sensors due to its outstanding features including its immunity to electromagnetic interference, easy fabrication and compact structure. Among many types of fiber-based sensor, tapered optical fiber sensor is considered one of the most facile and easy to fabricate. This type of sensor is fabricated by reducing the diameter of the fiber to allow the existence of evanescence waves that often manipulated for various sensing application. Thus, increasing its interaction with the surrounding medium, allowing the measurements of humidity responses. The process of reduction or tapering of optical fiber is often achieved through laser ablation, polishing, chemical etching and focused ion beam etching and flame brushing technique [2]. Nevertheless, the flame heating technique has proven to be the most versatile technique. It can fabricate tapered fiber with good physical techniques to produce the optimum condition for tapered optical fiber [3]. Flame brushing technique is a technique where a uniform heat is applied onto a section of the fiber while it is being pulled, involving a pulling rig system [4].

A tapered optical fiber is considered adiabatic if and only if the main portion of light remains in the fundamental mode (LP01) as it propagates through the fiber. The tapering angle should must be small so that the guided mode will not couple with other undesirable mode. Maximum angle that is allowable is

$$\Omega_z = \frac{r(z)}{z_b} = \frac{r(z)(\beta_1 - \beta_2)}{2\pi}$$

(1)
Given $\Omega_x$ is the local half-taper angle, $r(z)$ is the local radius of taper transitions, $\beta_1$ and $\beta_2$ are the local propagation constants of the propagating mode and the nearest symmetric mode, respectively. Whereas the beating length is given by

$$z_b = \frac{2\pi}{(\beta_1 - \beta_2)}$$  \hspace{1cm} (2)$$

A condition for low loss fiber is when the local taper length scale

$$z_t \gg z_b, \text{ where } z_t = \frac{r(z)}{2\pi}$$  \hspace{1cm} (3)$$

However, if the local taper length scale change is much larger, coupling happens between the fundamental mode of the un-pulled fiber and the first two modes of the taper waveguide ($LP_{01}$, $LP_{02}$) [5]. This condition creates a non-adiabatic tapered fiber. Light propagates at the air-cladding interface, changing the light characteristics from single mode fiber to multi-mode fiber. The energy transfer from the fundamental mode to higher order modes, depending on the rate of diameter changes. A large portion of evanescent field extended to the external medium. As a result, characteristic mode travelling in the tapered region become dependable with the surrounding medium. Hence, the resultant intensity at the end of the fiber, $I$, is given by

$$I = I_{core} + I_{cladding} + 2\sqrt{I_{core}I_{cladding}\cos\Phi}$$  \hspace{1cm} (4)$$

Where $I_{core}$ and $I_{cladding}$ is the intensity of light at the core and cladding mode, $\Delta\Phi$ is the phase difference between core and cladding. Different sensing material has been used as the sensitive coating for many sensing purposes. This additional film serves unique characteristics believed to boost the sensor sensitivity. Among the coating materials includes gold [6], zinc oxide [7], graphene oxide (GO) [8], Poly (vinyl) alcohol (PVA) [9] and TiO$_2$ [10]. Therefore, this research focuses on the potential of tapered optical fiber sensor coated with GO/PVA nanocomposite to be employed as improved humidity sensor.

2. Materials and methods

2.1 Fabrication of non-adiabatic tapered fiber coated with GO/PVA ultrasensitive thin film

Fabrication of tapered fiber was done by reducing the waist diameter of a fiber in a short section of distance of a standard silica glass optical fiber. In this practice, the tapered structure was achieved through flame brushing technique, where constant heat was applied onto a stripped section of single mode fiber (SMF) causing it to elongate with reduced waist diameter. The polymer buffer coating was removed from a 2 cm long section in the middle of a 500 cm length of the SMF using a mechanical stripper. The section was cleaned with acetone to eliminate any remaining buffer coating or dusts. During the tapering, the stripped section of the optical fibre was exposed to the flame produced by the butane gas burner (max temperature 1300 °C) while the fiber was gently stretched. The section was flame brushed back and forth horizontally until a region of uniform reduced diameter was formed in between the fiber.

Commercially purchased GO dispersion with concentration of 6 mg/ml was dissolved until it reached concentration of 2 mg/ml. 0.3 g PVA with 630 degree of polymerization was added into the GO solution before the mixture was immediately stirred and heated for 12 hours in 30 °C. The fiber was then coated with GO/PVA by dip-coating technique. The coated fiber was left to dry overnight at room temperature of 25 °C.
2.2 Humidity experimental setup
The experimental setup for the proposed sensor to detect humidity responses consists of a light source, experimental medium, humidity meter and spectrometer as in figure 1. The light source used in this experiment is Tungsten-Halogen white light source operated in wavelength of 360 nm-2400 nm. Light form the light source transmitted through the fiber. At the tapered section, a portion of evanescence wave interacted with the surrounding medium with different humidity level ranging from 20 to 99.9 % RH in correlation with the humidity meter. The transmission signal of the fiber was collected by a spectrometer in wavelength of 200 nm – 1000 nm. Data was analysed using Thorlabs software and Origin pro 8 software.

![Figure 1. Experimental setup of involving light source, experimental medium, humidity meter and spectrometer.](image)

3. Result and discussion

3.1 Microstructure of the fiber sensor
Based on figure 2 (a), the structure of tapered optical fiber experience gradual decrease in the microfiber structure ranging from 35.96 μm to 26.93 μm, showing non-adiabatic microfiber structure. In figure 2 (b), a smooth taper with constant diameter of 8.61 μm was successfully achieved on the thinnest region of the tapered region.

![Figure 2. The structure of tapered SMF under 10/0.25 resolution showing (a) the non-adiabatic structure of taper fiber and (b) uniform tapered part of the fiber sensor.](image)

By referring to the evanescent field theory, the evanescent wave that propagates on the outer waveguide-surrounding interface is inversely proportional to the tapered waist diameter. With higher number of evanescence waves interact with the experimental medium, the sensor becomes more
sensitive to any changes happen in the external refractive index. Stronger evanescent field provides high improvements to the sensing responses of the fiber sensor.

3.2 Performance of non-adiabatic tapered optical fiber sensor

The significance of tapering process in the humidity sensing application is proven from the difference in performances of un-tapered and tapered SMF as represented in figure 3.

An experiment was carried out to determine the performance of un-tapered fiber sensor and how it differs with the tapered one. A standard SMF with diameter of 125 μm and a tapered SMF with diameter of 8.61 μm were tested with different percentage of humidity level, ranging from 20.0 % RH to 99.9 % RH. The data was taken in the spectrum range from 500 nm to 1100 nm. As shown in figure 3 (a), the spectra of un-tapered fiber sensor overlapped each other and any significant difference between the humidity levels are hardly seen. To conclude, light rays were perfectly confined in the fiber and there was no presence of evanescent waves to detect any refractive index changes happening on the surrounding medium. By means, no sensing information can be gathered by the output transmission due to the lack of signal interaction with the surrounding. Whereas in figure 3 (b), the transmission intensity increases with response towards the increasing surrounding RH. When water molecules presented in the container increased, the effective refractive index of the surrounding medium increased. Relating with total internal reflection principle, more light rays continued to reflect in the core thus increasing the light rays confined within it. The transmission signal therefore higher at increasing RH. From the figure, intensity at wavelength 700.00 nm was taken to measure the sensitivity. A graph of relative intensity versus percent relative humidity in linear fitting for both sensors was plotted in figure 4 for the ease of comparison.

Figure 3. The performance of (a) untapered fiber sensor and (b) tapered fiber sensor under varying humidity of 20 % RH to 99.9 % RH.
Figure 4. Graph of relative intensity versus percent relative humidity in linear fitting.

Based on the graph, the sensitivities are determined from the slopes of the graph for both untapered and tapered fibers, which are calculated at $-0.0003 \text{ a.u.} (\%)^{-1}$ and $0.005 \text{ a.u.} (\%)^{-1}$ respectively. The slope of untapered fiber is too small (almost negligible) and not in any specific order.

3.3 Surface morphology of GO/PVA nanocomposite coating

FESEM analysis was carried out on sample of GO/PVA nanocomposite prepared on a glass substrate in order to understand how the structural behaviour of the samples may affect the performance of the sensor. Figure 5 (a) shows an overview of the surface of GO/PVA nanocomposite in low magnification of 50 K. The nanocomposites surface appears to be uniformly distributed under low magnification image, indicating that the PVA were homogeneously dispersed into the GO layers. In higher magnification of 150 K referring to figure 5 (b), the surface of the nanocomposites showed noticeable roughness with no specific shape identified and the appearance of minimal cracks. The grain structure improved gradually with the increasing amount of cross-linking network between GO and PVA, resulting in the formation of well-textured and oriented molecular structure of the surface.

Figure 5. Surface morphology of GO/PVA nanocomposites in (a) low magnification and (b) high magnification.
3.4 Enhancement of sensitivity through GO/PVA coating

The role of GO/PVA nanocomposite coating in enhancing the sensitivity was investigated by measuring the performance of tapered SMF with and without coating. A standard tapered SMF with diameter of 9.03 μm were exposed in humidity level varying from 20.0 to 99.9 % RH before and after coating to observe any difference in the output spectra. The intensity spectra were taken from wavelength 500 nm to 1100 nm as presented in figure 6.

Figure 6. The performance of (a) uncoated and (b) coated tapered SMF under varying humidity of 20 % RH to 99.9 % RH.

Roughly, both graphs acquire varying spectra corresponding to different percent relative humidity. However, the dissimilarity in between the graphs lay in the order of the transmission intensity. Referring to figure 6 (a), the transmission intensities of uncoated tapered fiber sensor for percent relative humidity ranging from 20.0 to 99.9 % RH are not in any specific order. As compared to transmission intensity of coated tapered fiber sensor in figure 6 (b), the intensities are in good ascending order. The graph of relative intensity versus percent relative humidity in linear fitting of both sensors are plotted in figure 7 for the ease of comparison and determination of the sensitivities.

Figure 7. Graph of relative intensity taken at 700 nm versus percent relative humidity in linear fitting.
Based on the graph, the sensitivities are determined from the slopes of the graph for both uncoated and coated tapered fiber sensors, which are calculated at \(0.0053 \text{ a.u.} \text{ (%)}^{-1}\) and \(0.0062 \text{ a.u.} \text{ (%)}^{-1}\) respectively. By comparison, coated tapered fiber sensor has higher sensitivity than the uncoated tapered fiber sensor, with percentage of sensitivity boosting up to 15.86 %. The reason behind the increasing performance of coated tapered fiber sensor is due to the structural behaviour of both materials in supporting humidity sensing mechanism. GO provides high surface area because of its high surface to volume ratio properties. Due to its layered honeycomb structure, abundant of reactive oxygen functional groups (carboxyl, hydroxyl and epoxy groups) contained in each layer [11]. The existence of these functional groups makes GO hold hydrophilic property, making it excellent in humidity sensing. The presence of oxygen functional groups on GO structure also make it possible for intercalation of polymers to happen between the layers of GO to form a composite film. Thus, allowing more PVA molecules to be trapped in between GO layers, forming a firm composite film on the surface of the tapered region. In the other hand, PVA provides stability to the whole structure [12], thus creating a stable nanocomposite film. To some extent, the duo complements each other as good moisture absorbent.

4. Conclusion

In summary, the proposed tapered fiber coated with GO/PVA nanocomposite film was successful in detecting humidity ranging from 20 to 99.9 % RH. Performance of the non-adiabatic tapered fiber sensor has been determined and the sensitivity recorded for both untapered and tapered fibers were \(-0.0003 \text{ a.u.} \text{ (%)}^{-1}\) and \(0.005 \text{ a.u.} \text{ (%)}^{-1}\), respectively. This work demonstrated that sensor with GO/PVA coating exhibited higher sensitivity, which was \(0.0062 \text{ a.u.} \text{ (%)}^{-1}\), when compared to uncoated ones with sensitivity of \(0.0053 \text{ a.u.} \text{ (%)}^{-1}\) owing to the hydrophilic properties of GO and film stability contributed by PVA.

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