SINGLE MODE MODIFIED TAPERED STRUCTURE WITH GRAPHENE OXIDE/POLYVINYL ALCOHOL COMPOSITE LAYER FOR RELATIVE HUMIDITY SENSING

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ABSTRACT

A moisture-sensitive sensor based on tapered optical fiber coated with graphene oxide (GO)/polyvinyl alcohol (PVA) nanocomposite film was studied and experimentally demonstrated. Modification on waveguide structure has been made by reduction of the waist diameter to promote evanescent wave for sensing purposes. In this study, standard silica single mode optical fiber with diameter of 125 microns was successfully tapered down to a smaller beam waist around 9 microns by flame-brushing technique. The tapered section of the fiber was then functionalized with additional deposition of GO/PVA nanocomposite layers through dip coating technique. The sensing principle is based on ability of the moisture-sensitive coatings to undergo changes in refractive index and modulates the intensity of light propagating through the fiber. Three different concentrations of nanocomposites comprise of GO/0.2 g PVA, GO/0.3 g PVA and GO/0.4 g PVA were prepared and characterized using field emission scanning electron microscope (FESEM) and Raman spectroscopy. The presence of oxygen functional groups (such as -OH, COOH) on the GO structure creates an invitation for the attachment of PVA through hydrogen bonding as evidenced by the FTIR analysis, thus providing strong adhesion between the GO and PVA layers. The performance of the sensor was investigated over a wide range of 20.0 to 99.9% relative humidity (RH), where the sensor showed good sensitivity as its signal increased linearly with respect to the surrounding RH. Tapered optical fiber sensor with coating of GO/0.3 g PVA recorded the highest sensitivity, which was found at 0.5290 % absorption/% RH. The sensor shows good repeatability with precision error of 3.11%. The sensor acquired good reversibility in both increasing and decreasing relative humidity with close points recorded at the same % RH. Stability of the output signals suffers from high fluctuation at extreme surrounding condition of 99.9% RH with fluctuation rate of 0.009/90 minutes. Finally, response time of 141 seconds corresponding to full range of RH spread from 20.0 to 99.9% has been experimentally achieved.

ABSTRAK

Satu pengesan yang peka-kelembapan berdasarkan gentian optik tirus yang disalut dengan nanokomposit filem grafin oksida (GO) / polivinil alkohol (PVA) telah dikaji dan ditunjukkan secara eksperimen. Pengubahsuaian pada struktur pandu gelombang telah dibuat melalui pengurangan diameter pinggang untuk menggalakkan gelombang evanesen bagi tujuan pengesanan. Dalam kajian ini, gentian optik mod tunggal silika piawai dengan diameter 125 mikron telah berjaya ditiruskan kepada pinggang pancaran yang lebih kecil sekitar 9 mikron dengan menggunakan teknik berusan-api. Bahagian tirus gentian kemudiannya difungsikan dengan pemendapan tambahan lapisan nanokomposit GO/PVA melalui teknik penyalutan celup. Prinsip pengesan adalah berdasarkan kepada keupayaan celupan peka-kelembapan menjalani perubahan indeks biasan dan memodulatkan keamatan cahaya melalui gentian. Tiga kepekatan nanokomposit berlainan yang terdiri daripada GO/0.2 g PVA, GO/0.3 g PVA dan GO/0.4 g PVA telah disediakan dan dicirikan menggunakan pelepasan bidang mikroskop elektron (FESEM) dan spektroskopi Raman. Kehadiran kumpulan berfungsi oksigen (seperti -OH, COOH) di atas struktur GO mewujudkan jemputan untuk pelekatan PVA melalui ikatan hidrogen seperti yang dibuktikan oleh analisis FTIR, lalu menyediakan lekatan yang kuat antara lapisan GO dengan PVA. Prestasi pengesan itu dikaji pada julat kelembapan relatif (RH) yang lebar, di antara 20.0 ke 99.9%, dengan pengesan menunjukkan kepekaan yang baik apabila isyarat meningkat secara linear dengan RH persekitaran. Pengesan gentian optik tirus dengan salutan GO/0.3 g PVA merekodkan kepekaan yang tertinggi, yang didapati pada 0.5290 % kelembapan/% RH. Pengesan menunjukkan kebolehulangan yang baik dengan ralat kejituan 3.11%. Pengesan memperoleh kebolehulangan yang baik dalam kedua-dua peningkatan dan pengurangan kelembapan relatif dengan titik berhampiran direkodkan pada % RH yang sama. Kestabilan isyarat keluaran mengalami turun naik yang tinggi pada keadaan persekitaran lampau pada 99.9% RH dengan kadar turun naik 0.009 / 90 minit. Akhirnya, masa tindak balas 141 saat sepadan dengan julat penuh penyebaran RH daripada 20.0 ke 99.9% telah dicapai melalui uji kaji.

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LIST OF ABBREVIATIONS

UTM	-	Universiti Teknologi Malaysia
RH	-	Relative Humidity
CAGR	-	Compound Annual Growth Rate
FBG	-	Fiber Bragg Grating
GO	-	Graphene Oxide
PVA	-	Poly (Vinyl) Alcohol
HF	-	Hydrofluoric
FIB	-	Fiber Iron Beam
MZI	-	Mach-Zehnder Interferometer
MEMS	-	Micro-Electro-Mechanical Sensor
FTIR	-	Fourier Transform Infrared Radiation
FESEM	-	Field Emission Scanning Electron Microscope
SMF	-	Single Mode Fiber

LIST OF SYMBOLS

P	-	partial pressure
f	-	fraction of evanescence wave
m	-	mass
v	-	volume of air in m^3
μm	-	micrometre
%	-	percentage
nm	-	nanometre
n	-	refractive index
g	-	gram
L	-	litter
Θ	-	angle
E	-	evanescence wave intensity
a	-	absorption coefficient
λ	-	wavelength
b	-	attenuation coefficient
d_p	-	penetration depth
l	-	length
N	-	number of reflection
d	-	fiber diameter
I	-	intensity
C	-	concentration of medium
$\Delta\Phi$	-	phase difference
Ω	-	local angle
r	-	local radius
V	-	volt
°C	-	degree Celsius
β	-	local propagation constant
ppm	-	parts per million

s	-	second
π	-	pi-value
r	-	reduction factor of attenuation
cm	-	centimetre
a.u.	-	arbitrary unit

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Humidity plays a great role in every part of humans' life, be it for the industrial purposes or for our own comfort. Humidity is described as the moisture content or amount of water vapour presents in the air while relative humidity (RH) is defined by the ratio amount of moisture content in the air to the maximum moisture level that it can achieved in constant pressure and temperature [1]. RH measurements are often stated in percentage because it is simpler and widely applied in many sensors measurements. The relation is determine by the equation

$$\% RH = \frac{P_v}{P_s} \times 100 \tag{1.1}$$

Where the actual partial pressure of moisture content in air is describes as P_v while P_s is the saturated pressure of moisture content in air in the constant temperature (Bar or kPa). Meanwhile, saturation humidity, SH is described as the ratio mass of water vapour at saturated level to the volume of air. Given m_{ws} is the mass of water vapour at saturated value in gram and v is the volume of air in m^3. The maximum amount of moisture content (mass) in unit volume of gas at constant temperature is

$$SH = \frac{m_{ws}}{v} \tag{1.2}$$

The increase in demands for highly efficient and reliable humidity sensor shows how important the device is to the humankind. It is expected that the humidity sensor market will reach approximately USD 1.8 billion by 2023 with Compound Annual Growth Rate (CAGR) growing at the rate of 15% over the period starting from

2016 [2]. From agriculture, food processing, semiconductor processing, mining, textile and extended to pharmaceuticals industries; all of the stated industries depends greatly on humidity sensors to monitor the air moisture circulating around them. Although there have been variety of humidity sensors established worldwide, the rise in demands showed that there is always room for further improvements to be made with the aid of new advance technology.

The established humidity sensors that have been used for decades in various industries possessed their own limitations. As an example, capacitive-based humidity sensor experienced limited long-term stability and certain substances can deteriorate the performance of the sensor [3]. Since the sensor need to be in direct contact with gaseous, any composition of the gas that may not compatible with the sensor will result in the alteration of sensor's performance. Take small droplets of oil and dusts for instance, the accumulation of these unwanted substances will interfere the nature of dielectric material. This sensor works best in environment conditions with varying high and low temperature. However, it is not ideal to be use in critical condition such as for the detection of soil moisture content. Condensation might happen and destroy the whole measurements of the sensor [4].Psychometric humidity sensor is a sensor working based on evaporative cooling and it has been used for a long time in the measurements of relative humidity. It is a sensor with advantages of large range of measurement values and it can withstand rough environment. Yet, it also has weaknesses in which it has low accuracy, not suitable for long-term usage since it requires water reserve and wick maintenance. Besides, skilled operators are required to handle the operation of the sensor [5]. In addition, the conventional electric sensors present several drawbacks such as high cost, need for maintenance and inability to be use in hazardous or explosive nature environments, in which electromagnetic interference immunity is required.

In these days, the attention for sensor application had shifted to fiber-based sensor. Fiber-based sensing offers advantages over electrical and electronic sensors because of the photonic behaviour of light that is predictable and controllable. It is lightweight and robust. Thus, it can work normally even in the places with high magnetic fields, high radiation fields and in extreme temperature environments. Fiber based sensor has been used in many application involving measurements of quantities like temperature, vibration, pH and humidity. There are three types of fiber-based sensor, which are intensity-based fiber sensor, wavelength-based fiber sensor and phase-modulated fiber sensors [6]. The intensity-based fiber sensor rely on the loss of intensity signal with variety of mechanisms such as micro bending loss, attenuation and evanescent fields. This type of sensor has been used widely in the detection of gases and chemicals with different refractive index. Among the types of fiber that falls under this category are D-shaped fiber, U-shaped fiber and tapered optical fiber. The wavelength-modulated based sensor operates when there is shifting in the wavelength of light. Sensors using this kind of detection are fluorescence sensor, black body sensor and Bragg grating sensor. Fiber Bragg Grating (FBG) sensor is the most commonly used sensor, which operates by reflecting particular wavelengths or light and transmits the others. The phase modulated-based sensor works by measuring phase changes involves in the light detection. The examples of this type of fiber are Mach-Zehnder, Fabry-Perot and grating interferometer.

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. Among the technique involves in the reduction of fiber diameter includes chemical technique by applying strong concentrated acids, or mechanical technique involving moulding of the fiber and heat and pull technique where constant heat is applied onto a section of fiber. Tapered optical fiber has been publicize in the research field where its potential is being explored in many kinds of measuring purposes [7-10]. Incorporation of fiber-based sensor with nano-structured elements is relatively a good idea because it provides high-impact improvements in the accuracy, reliability, stability, and practicality. The difference in effective refractive index in between the cladding will allow more light to be refracted out of the fiber and interacting with the experimenting medium. Different sensing material has been used as the sensitive coating for many sensing purposes. Among the coating materials includes gold [11], zinc oxide [12], graphene oxide (GO) [13], Poly (vinyl) alcohol (PVA) [14] and TiO_2 [15].

Therefore, this research focuses on the potential of tapered fiber optical sensor coated with GO/PVA nanocomposite to be employed as improved humidity sensor compared to the conventional ones. The interest of combining PVA with GO arises due to the large surface area property of GO with plenty of oxygenated functional groups attached onto it. Mixture of GO and PVA allow the dispersion of PVA in between GO sheets due to the strong hydrogen bonding interaction [16]. The features possess by the combination of these duo offers promising results.

1.2 Problem Statement

In the manufacturing of industrial products such as electronic devices, some of the parameters are being controlled to observe the product potential performances under certain condition. These parameters includes temperature, pressure and relative humidity. Among the parameters stated, only the measurement of RH is needed to be in contact with the environment whereas temperature and pressure are invariably insulated from the process through the beneficial usage of thermowell and diaphragm, respectively. The constantly changing environment through various manufacturing processes makes it impossible for any humidity sensor to work efficiently in a long period of time without experiencing contamination and degradation. Thus, recalibration and periodic replacement of the sensor is needed, which involved huge amount of time and cost. Furthermore, most of the conventional sensor that based on the measurement of electrical resistivity or capacitance has a tendency to experience the state of 'gone to sleep' in which the sensor become less sensitive to any changes revolving around it and becomes too saturated with the environmental condition. This type of sensors also suffers mainly from inability to be used in hazardous or explosive nature environments, in which electromagnetic interference immunity is required.

. Therefore, the need to invent a vivid sensor to replace the conventional ones is crucial in order to meet the industrial needs. Among several options, tapered optical fiber sensor is considered as promising solution to these problems, owing to its unique characteristic such as low cost, electrically passive operation, long life-time and practically suitable to be used regardless the condition of surrounding. Most importantly, this kind of sensor is unaffected by any electromagnetic interference produced by electronic devices placed nearby the sensing area, make it very accurate and reliable method. In particular, the tapered section of the fiber enables the evanescent fields to interact directly with the surrounding medium, providing a descent sensing mechanism. Overall performance of the proposed sensor could be further improved by the implementation of sensitive material as outer coating layer where the evanescent field propagating at. The uses of specific coatings open up the new challenge and possibilities of designing particular sensor for detection of measures of interest. Properties of the moisture-sensitive material coating play an important role towards the process and need to be carefully examined beforehand. Comprehensive research work needs to be carried out in order to fully explore the potential of GO/PVA composite film as the outer layer of tapered optical fiber for humidity sensing purposes.

Above and beyond all other consideration, the optimum condition for the sensor to perform sensing task is a crucial goal that needs to be achieved. Relation between key parameters with the output performance of the sensor needs to be carefully examined in order to contribute towards improving any particular characteristic useful for RH sensing purposes.–Other important features such as stability, repeatability and response time are yet to be optimized before realization of such sensor. Analysing and examining the results establish a better understanding on the physics of such system, which give a significant contribution to our body of knowledge.

1.3 Research Goal

The main objectives of this study is to develop a new tapered optical fiber sensor coated with GO/PVA nanocomposite film for the detection humidity sensing purpose.

1.3.1 Research Objectives

In order to achieve the stated goal, specific objectives are listed as below;

a) To characterize and analyse different ratio of GO/PVA nanocomposite and deposit as the ultrasensitive coating layer on the tapered section of optical fiber.
b) To record and analyse the absorbance spectrum from the output signals of optical fiber sensor with respect to changing surrounding humidity.
c) To determine and optimize the sensitivity, stability, reversibility, repeatability and response time of the designated sensor.

1.4 Scope of Research and Limitations

This research is categorized into three main parts; fabrication and optimization of tapered optical fiber, optimization and characterization of GO/PVA nanocomposite film as the ultrasensitive coating layer and data analysation. In the fabrication of tapered optical fiber, Silica based single mode fiber (SMF) with standard core-cladding diameter of 9/125 μm is used. The fabrication process involves tapering of optical fiber with flame-brushing technique. Fiber is stretched and one side of the fiber is pulled with pulling speed of 1mm/s. Optimization of the tapered fibre is done by tapering in waist diameter in range 8.15 μm up to 46.14 μm and waist length of maximum 3cm. The best dimension is further use in the coating step. The GO sample is purchased from Graphene Supermarket with concentration of 6.2 g/L. Flake size in the range of 0.5 to 5 μm and the solid content is 1.085 g. PVA sample is purchased from Sigma Aldrich with 630 degree of polymerization. The coating is done by dip coating the tapered region with GO/PVA solution involving three sets of samples; GO/0.2 g PVA, GO/0.3 g PVA and GO/0.4 g PVA. Characterizations involving Fourier Transform Infrared Radiation (FTIR), Field Emission Scanning Electron Microscope (FESEM) and Raman spectroscopy are done onto the samples and coating film.

Ocean Optics Halogen-Tungsten white light source with wavelength in between 400 nm to 1000 nm is used as the light source, transmitting light onto the fiber and interacting with the medium with relative humidity ranging from 20.0 % RH to

99.9 % RH. Thorlabs Spectrometer CCS200 spectrometer in the range of 200 nm to 1000 nm in the visible light region with resolution of 2 nm is use to capture the signal transmission of the light. The intensity based-spectra is represented in Thorlabs software and the absorbance response is calculated. The analysation of data is done by using Origin 8 Pro software. Parameters investigated in the research works includes the reversibility, repeatability, stability and response time. Reversibility is done by increasing and decreasing value of RH in the range of 20.0 % RH to 99.9 % RH. Repeatability involves three times data collection and error measurements. Stability is conducted to measure the data in long duration of 90 minutes while the time taken to evaluate the response time is 189 seconds.

There are some limitations in this experiment involving the taper profile of the fiber sensor. The tapering setup involved the pulling of only one side of the fiber while it is being heated. Another side of the fiber in which is fixed onto its position resulted in abrupt (non-adiabatic) taper profile. Moreover, the down taper and up taper of the fiber could not be set, thus the diameter of the tapered fibers could not be fixed into any preferred integral numbers. As a consequence, it is quite impossible for the realisation of adiabatic taper profile.

1.5 Research Significance

Humidity sensor is known as the most commonly used sensor in industries involving semiconductor, agriculture, structural, climatology and automated system. The amount of water content in the air can greatly affect the performance of a product. In soil management for instance, crop productions depends greatly on soil moisture content. Vegetables and fruits need to meet certain humidity level to ensure the products are marketable. In Japan for example, fruits are often treated as luxury items given as gifts and melon are among the most expensive fruit. A good quality musk melon can easily cost more than 10,000 yen or 100 USD. There must be a good reason behind it. The farmers need to maintain the most conducive environment for the plant to grow and produce juicy and sweet melons. The key factor is to keep the musk melon at optimum temperature and humidity level since the early stage involving the preparation of seeds and soil. Another good example is in the production of semiconductor components. Humidity level is controlled and monitored in the wafer production because too much moisture might destroy the physical and chemical process of the wafer. In this case, the relative humidity should be maintained at 30 % RH at 20 °C (70°F) or else it might cause failure to the semiconductor assembly. Among the problems arises due to increase in humidity level are corrosion to the circuit points, condensation on microchips circuit surface and improper adhesion of photo resist. The components of wafer production are often comprises of polymers, which are hygroscopic thus, it can cause the polymers to swell and harm the wafer surface.

In recent days, we have witnessed variety of sensors are being developed and produced to cater the needs for humidity sensor. However, the commercialized sensors are still lacking in stability, accuracy in long terms and weak endurance against rough surroundings. The need for improvement in humidity sensor is undeniable as it is important to have a good humidity sensor with good characteristics of; repeatability, accuracy, long-term stability, interchangeability, ability to recover from condensation in a short time, resistance to physical and chemical contaminants, small compact size and low in production cost. Thus, execution of this research work will discover the huge potential of exploiting tapered optical fiber sensor into humidity sensing by proposing the application in real time. Tapered optical fiber sensor provides long-term stability, high sensitivity and it is chemically inert, hence it can withstand hazardous environments. It is small, compact, easy to install hence making it practical to be use in any system. With added feature of GO/PVA nanocomposite coating at the tapered region, it is expected that it will boost the sensitivity of the sensor and provide extra stability towards the sensing region.

Conclusively, the significances of this study mainly contribute towards the increment of our underlying knowledge on the RH sensing by tapered optical fiber coated with GO/PVA nanocomposite. Understanding and quantifying the physics of such system gives an insight into the field of optical sensor. Successful development of the proposed sensor leaves a direct benefit for scientific awareness of the country, and the whole research activities can be used for future references.

1.6 Thesis Organization

The flow of the research works is further elaborated in this section. Chapters comprising of introductions, literature studies, methods involved, data collection, data analysation and future perspectives are sorted in this thesis.

Chapter 1 gives a brief idea of what the research is all about. A simple introduction with problem statements are important to raise the issues about why this research need to be done in the first place. Research questions and research objectives are listed as goals that should be achieved at the end of research works. The scope of the research is explained in details to highlight the limit of the research. The significance of the study is briefly described to give a clear vision of how the research can benefit people especially in industries with controlled humidity environment.

Chapter 2 is the literature studies related to the research. It comprises of the working principle of fiber and how does it differ in tapered optical fiber sensor. Fundamental concept of sensing parameters are also explained as well as the adiabatic condition that need to be achieved by the fiber sensor to reduce signal attenuation. Since the research involves coating of GO/PVA nanocomposite film at the tapered region as the ultrasensitive layer. Thus, the chemical structure of each material and their characteristics that contribute to the sensing development are briefly explained.

Chapter 3 is the where the methodology of the experiment is explained in details. Starting from the fabrication of tapered optical fiber, preparation of three sets of GO/PVA nanocomposite with different mass of PVA, characterization of the samples and coating technique implemented on the sensing region. All techniques, materials and apparatus involved are revealed in this section.

Chapter 4 is where the entire results of the research is represented in the form of tables and graphs. Material characterization is included in this chapter. Spectra of tapered optical fiber with three different fraction of GO/PVA nanocomposites are observed and the sensitivity each sensors are compared. Sensor with the highest performance went for further measurements of reversibility, repeatability, stability and response time.

Chapter 5 is the last chapter where it concluded the research based on experimental results. In this chapter, the objectives of the research are revised whether the objectives are fulfilled or not. This chapter also discussed the sensor potential to be employ into the related industries; whether it is marketable or not. It also discussed the future improvement of sensor that can be made, if any.

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