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doi:10.15199/48.2022.04.16

Tapered plastic optical fiber loop coated with ZnO nanorods using multiple channels for relative humidity sensing

Abstract*. To utilize the evanescent wave on a plastic optical fiber (POF), a humidity sensor was fabricated based on a tapered POF with a loop shape and coated with zinc oxide (ZnO) nanorods. The POF was tapered manually to a diameter of 0.90 mm by using the polishing method. ZnO nanorods were synthesized using the hydrothermal method. The obtained results revealed that the increase in the length of the tapered POF loop coated with ZnO nanorod had provided an excellent sensing performance as an RH sensor in terms of sensitivity and repeatability properties.*

Streszczenie. *Aby wykorzystać falę zanikającą na światłowodzie z tworzywa sztucznego (POF), wykonano czujnik wilgotności oparty na stożkowym POF w kształcie pętli i pokryty nanoprętami tlenku cynku (ZnO). POF zwężano ręcznie do średnicy 0,90 mm metodą polerowania. Nanopręty ZnO zsyntetyzowano metodą hydrotermalną. Uzyskane wyniki wykazały, że zwiększenie długości zwężającej się pętli POF pokrytej nanoprętem ZnO zapewniło doskonałe wyniki wykrywania jako czujnik wilgotności względnej pod względem czułości i właściwości powtarzalności.* (***Zwężająca się pętla światłowodowa z tworzywa sztucznego pokryta nanoprętami ZnO wykorzystująca wiele kanałów do wykrywania wilgotności względne***)

Keywords: Humidity sensor, Zinc Oxide nanorods, Plastic optical fiber
Słowa kluczowe: czujnik wilgotności, światłowód.

Introduction

Almost every industry nowadays emphasize the importance of humidity detection and control. Applications such as agriculture, health, food processing, and meteorology require accurate, high speed and economic measurement of humidity [1]–[3]. Relative Humidity (RH) is used to narrate the amount of water vapor in the air. In definition, relative humidity is defined as the ratio of the actual water vapour pressure to the saturated vapour pressure at a certain temperature [4]. In recent decades, optical fiber humidity sensor development has shown an outperform from the conventional electrical sensors where optical fiber is immune from the electromagnetic field and harsh environment [5].

Optical fiber-based sensors have been widely researched in recent years because of its properties that suits as a sensing medium [6]–[9]. In optical fiber sensor, microfiber resonator attracts great attention due to its simple structure, high sensitivity, and good refractive index sensing. The microfiber loop resonator is easily fabricated as the loop is maintained by van der Waals or electrostatic forces in the joint area[10]. A previous study proves that microfiber loop resonator is another possible route to fabricate a robust micro-resonator for sensing applications.[11]–[15] However these method introduce drawback with hardness in fabrication and handling a very small core of glass fiber are very tricky because it could easily break. Plastic optical fiber (POF) can be a good replacement since POF is known to has a great flexibility and hostility to impacts and vibration while the greater coupling of light from the source to fiber [16].

Recent research has been done widely on Zinc Oxide (ZnO) in biological sensing and gas sensing application. ZnO is a semiconductor material with a wide bandgap of 3.37 eV at room temperature and high excitation energy of 60mV[10], [16]–[19]. ZnO is an applicable, biocompatible and biosafe material; thus, incorporating them as sensing elements ensures promising results. The preferable method for synthesizing ZnO nanorods on the surface of a POF is known as the hydrothermal method. The hydrothermal method, among other methods, require low temperature with straight forward control of nanorod morphology by variation of the experimental condition [19]. This method features a simple and environmentally friendly method.

This paper presents the design and development of a tapered POF loop coated with ZnO nanorods for relative humidity sensing by using visible light spectrum. The sensing mechanism of the designed sensor coated with ZnO nanorods upon exposure to the different humidity level was demonstrated, leading to a reduction of light intensity guided in the POF, thus changing the output voltage. The performance of the humidity sensor in sensing different humidity level also was analyzed in this work.

Material, fabrication and characterization

A POF covered by a fluorinated polymer jacket with 1880 to 2120 μm of the inner-outer diameter range, and the core diameter of the POF is in the range from 1840 to 2080 μm was used in this setup. The POF was first tapered using sandpaper and acetone until the core diameter reach 900 μm. Next, the tapered POF was converted into a loop. This process is needed to be done before ZnO nanorods coated onto the tapered POF loop. Fig. 1 shows the tapered POF loop without coated with ZnO nanorods. Each end of the fiber will be connected to the LED and Photodetector in the humidity sensing setup. The length of the end fiber to the loop was fixed to 8.6 cm; therefore, the loop diameters (D) can be varied. The loop diameter was varied to 4 different loop circumference length 11cm, 13 cm, 15cm and 19cm, with diameter length D, calculated was 3.5 cm, 4.33 cm, 4.93 cm and 6.05cm.

The synthesis of ZnO nanorods was carried out using the hydrothermal method [19], [20]. The growth time of the ZnO has been optimized in the previous work in Ref. [17]. The synthesis of ZnO nanorods begins with the seeding process. This process is important as the resulting coated ZnO characteristic depends on the seeding process in term of diameter, length and uniformity of the nanorods. Two solutions were prepared, which are the ZnO nanoparticle solution and the pH-controlled solution. Initially, 0.0044 g of zinc acetate dihydrate [Zn(O2CCH3)2(H2O2)2] was dissolved in 20 ml of ethanol under slow stirring at 50℃ temperature for 30 minutes to produce 1mM of ZnO nanoparticle solution. Then, 20 ml of ethanol was added to the solution. Next, the pH control solution was prepared by dissolving sodium hydroxide (NaOH) in 20 ml of ethanol, forming a 1mM solution by heating the solution at 50℃ temperature while slowly stirring the solution for 10 minutes. The prepared pH control solution was added into ZnO nanoparticles solution using a pipet. This method is needed to generate hydroxyl ions (OH-) into the seeding solution. For each drop of pH control solution, the ZnO nanoparticles solution was stirred slowly for 1 minute. The seeding solution was then kept in a water bath at 60℃ temperature for 3 hours.

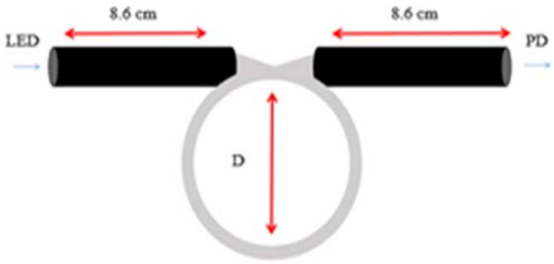

Fig.1. The schematic of the tapered POF loop

The tapered POF loop seeding process was conducted using a slow stirring method and followed by the dipping and drying method. In the first step, the POF was immersed in the prepared seeding solution for 30 minutes under slow stirring. Next, the POF was dipped into the prepared seeding solution for 1 minute and then dried on a hot plate at the temperature of 70℃ for another 1 minute. This process repeated at least ten times. Then, the seeded POF was annealed for 3 hours at 70℃.

Growth solution was prepared by dissolving of 2.97g zinc nitrate hexahydrate [Zn(NO3)2.6H2O] and 1.40 g hexamethylenetetramine (HMT) [(CH2)6N4] into 1000 ml of deionized water (DI) to produce 10 mM solutions for each of the compound [19]–[21]. The seeded POF was placed in the solution and heated at 90℃. In order to maintain constant growth, the solution was discarded and replaced with a new solution every 5 hours. The growth time was set to be 12 hours to obtain ZnO nanorods on the tapered POF loop. Upon completion of the growth time, the tapered POF loop was removed and rinsed with DI water.

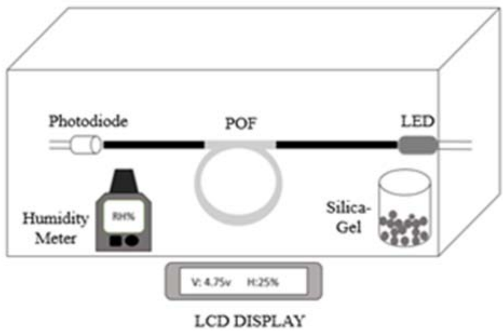

Fig. 2. Setup for humidity sensing

Setup for the tapered POF loop in a humidity chamber is shown in Fig. 2. During the test, the POF was placed inside the chamber, and the level of humidity was controlled using a wet tissue and silica gel. The relative humidity level was measured from 30 %RH – 90 %RH, and a humidity meter was used to monitor the range of relative humidity level. The experiment was conducted in a laboratory with a controlled temperature at 26°C.

Sensing Mechanism

For equations it is recommended to use standard equation editor existing in Word editor (usually it is Math Type editor). The equation editor is defined as follows: font Times New Roman italic, matrix bold, for letters font 10, Fig. 3 illustrates the sensing mechanism of the tapered POF loop coated with ZnO nanorods. The evanescent field and refractive indexes are interrelated and play a major role in enhancing the sensor sensitivity. The output results were based on the intensity-variation scheme. Evanescent field absorption varies with the change in the surrounding medium when the light propagates through the sensing region.[22] Variation of humidity level will change the refractive index of the fiber and the surrounding medium that modulate the output light intensity.

The ZnO layer in the exposed core surface of the POF act as a changeable refractive index layer with respect to the change of the sensing element [23]. When water vapor was absorbed onto the sensing element, which is Zinc Oxide, it leads to the change in the refractive index of the respective nanomaterial. Another factor the microfiber is highly sensitive to the parameter changes of the external medium was due to the strong evanescent field along the surface of the exposed core POF coated with zinc oxide nanorod. The design has shown several advantages, which is quick in response, high resolution and stable measurement[24]

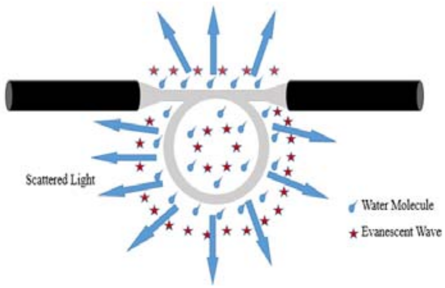

Fig. 3. Sensing mechanism of tapered POF loop coated with ZnO nanorods

Characterization of ZnO Nanorod using Scanning Electron Microscopy (SEM)

The coating layer on the tapered POF loop was observed using a scanning electron microscope (SEM). Fig 4 shows the morphology of ZnO nanorods on the tapered POF loop using SEM. It can be seen that the ZnO nanorods were evenly distributed on the surface of the tapered POF region. The length and diameter of the ZnO nanorods were approximately 250 nm and 1000 nm, with the growth time of the ZnO nanorod was set to 12 hours. This result also reported in Ref. [17]. Fig. 4 and 5 shows Energy Dispersive Spectroscopy (EDS) elemental analysis, revealing Zinc and Oxygen peak at the coating layer on the POF surface

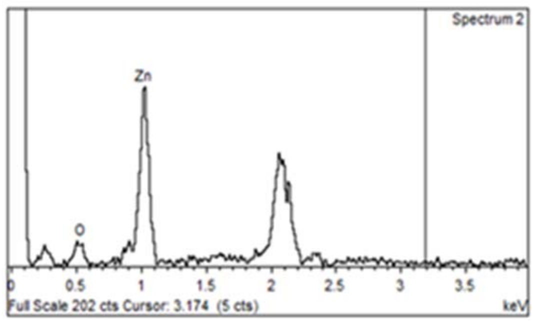

Fig. 4. EDS elemental analysis showing zinc and oxygen peak

Result and Discussion

Fig. 6A shows the output voltage against relative humidity for four different lengths of tapered POF loop coated with ZnO nanorods. Initially, the output voltage for D1, D2, D3 and D4 was approximately at 1.17, 1.21, 1.51, 1.46 V, respectively, at 90%RH. It was observed that the output voltage for D1 started to increase slowly when the relative humidity decrease, up to 30 %RH. The highest average output voltage for D1 was recorded at 30%RH, with the values was 1.58V. These patterns also were observed for D2, D3 and D4, in which the output voltage was increased as the relative humidity inside the chamber decreased. It also was revealed that D3 and D4 indicated a significant difference in output voltage compared to the D1 and D2 when using the blue LED. This might be because the D1 and D2 has lower exposed region length compared to D3 and D4 with exposed core region. This results to D3 and D4 to become a more sensitive to environmental changes sensor as the humidity decrease.

The result shows a similar pattern with a different range A) ut voltage. At 90 %RH, the output voltage for D1 was 1.33 V, and D2 was 1.42 V. The difference in range of output voltage recorded for each D1, D2, D3 and D4 as the relative humidity decrease from 90 to 30 %RH with D1 from 1.17 to 1.58V, D2 from 1.21 to 1.81V, D3 from 1.51 to 2.69V and the highest recorded is D4 from 1.46 to 3.11V. From Fig. 6A, it was observed that as the length of the exposed POF increased, the output voltage increased

The sensitivity recorded for D1, D2, D3 and D4 calculated from the slope of the plotted graph in Figure 10. D1 recorded the lowest sensitivity with 0.0066V/%RH, which was higher than using red LED as the input but slightly lower than green LED. Then, followed by D2 with 0.0108V/%RH and D3 with 0.0211V/%RH. The highest sensitivity was achieved by D4 with 0.0283/%RH, where this result also similar based on the red channel and green channel. The sensitivity was observed to be higher with respect to the length of the exposed core POF, where D4 has the highest length of tapered POF loop coated with ZnO nanorods, thus gaining the highest sensitivity value compared to D1, D2 and D3.

Fig. 6B shows the output voltage of D4 based on red, green and blue channel towards different humidity level. The graph pattern shows similar trend for the three different spectra. The output voltage for range from 90 – 30 %RH varies differently for each of the spectra with the highest value was generated by blue spectra with the value was 1.66 V, and followed by red channel with 1.5V, and the green channel produced the lowest output range with the value was 0.95V. The repeatability percentage was calculated as the sensor was exposed to different humidity levels from 30% B) %RH. A high value MR% obtained for blue is 13.5%, g vith 9.52% and red with 9.4%.

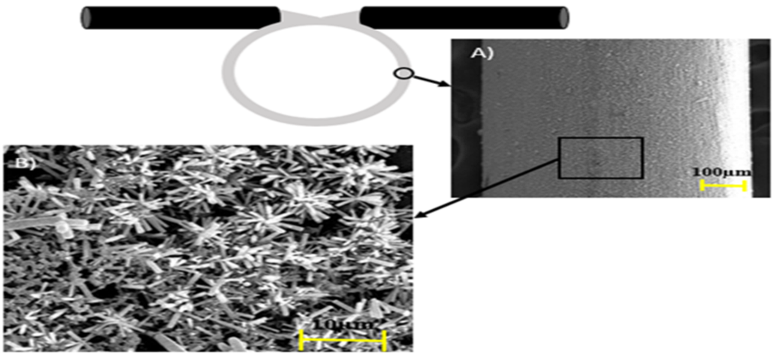

Fig. 5. Morphology of ZnO nanorods on the tapered POF loop using SEM A) ZnO coating on POF surface, B) the morphology of ZnO nanorod growth at 2.0k magnification

A(B)

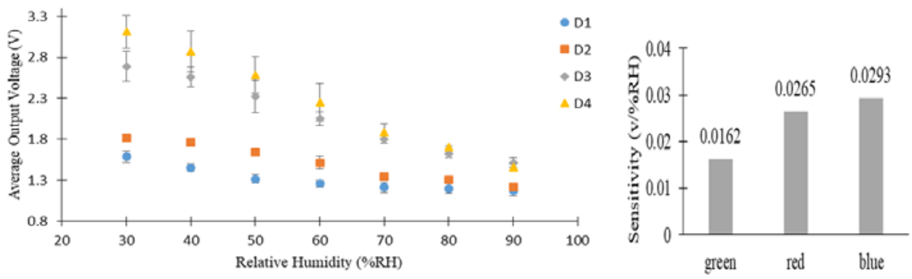

Fig. 6. A) Output voltage vs Relative humidity for Red Channel B) Sensitivity of D4 based on three different channels

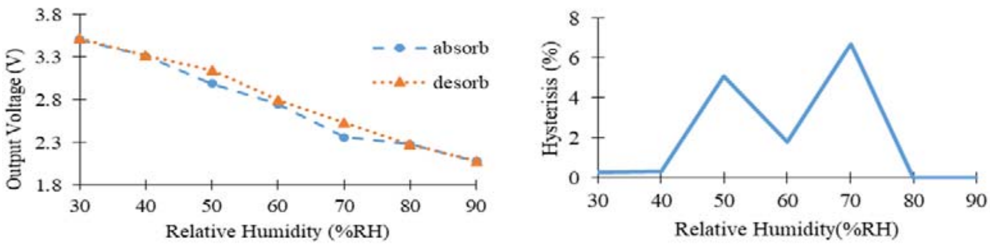

Fig. 7. A) Hysterisis curve of tapered POF loop coated ZnO absorption and desorption B) Hysterisis curve of tapered POF loop coated ZnO percentage.

. The sensitivity values were calculated based on the slope of the plotted graph in Fig. 6A. Blue spectra showed the highest sensitivity with 0.0293 V/%RH, followed by red spectra, with a slightly lower value which is 0.0265 V/%RH. Green spectra produced the lowest sensitivity with 0.0162 V/%RH. It was revealed that the blue spectra showed the highest slope value, thus resulting in higher sensitivity compared to the red and green channel.

Hysteresis curves of the tapered POF looped coated with ZnO is displayed in Fig. 7. A test was carried out by observing RH adsorption and desorption processes in the range of 30 %RH to 90 %RH for D4. The calculated humidity hysteresis (γH) at 60% RH was approximately 1.79%. The humidity hysteresis was calculated using $\gamma H = \pm (\Delta V_{max}/V_{FS})100\%$, where ΔV_{max} is the maximum voltage variance at a certain humidity level, and VFS is the full-scale output of voltage [25] Small hysteresis produced in a sensor result in higher reliability and measurement consistency [26]. As seen, D4 has a low humidity hysteresis; thus, this sensor produced better measurement consistency in the voltage stability

The output voltage against RH for coated POF and the uncoated POF sensor with the same exposed core length using the optimized length was investigated. In this testing, D4 was chosen because it produced the highest sensitivity compared to others sample. Fig. 8 shows output voltage vs relative humidity for uncoated POF and coated POF for D4 sensor. The result obtained for both sensors was decreased linearly with the increase of RH level. However, the coated POF sensor produces a much higher sensitivity which is 0.0285 V/%RH, compare to the uncoated POF with 0.0115 V/%RH. The linearity obtained for coated POF is 99.43%, while for the uncoated POF is slightly lower with 97.32% linearity. The presence of the coating ZnO layer on the exposed core surface results in higher sensitivity obtained. When POF was exposed to the humidity, the coated layer of ZnO nanorods experienced rapid surface adsorption of water molecule onto nanorods. This will result in a change of the effective reflective index and absorption coefficient of the ZnO nanorod surface as the optical properties of the nanorod was modulated. In this phenomena, as the light hit the water absorbed nanorods, more light will randomly be scattered rather than a loss at the nanorod area.

The stability performance for both coated and uncoated D4 sensor is shown in Fig. 9. The stability for both sensors was analyzed by prolonging both sensors at the same humidity level, which is 90 %RH, for 300s to investigate the response in term of output voltage. Based on the graph shown, both sensors showed a relatively consistent output voltage in 300s.

Table 1 summarizes the characteristics of uncoated and coated ZnO POF with the same exposed core length sensors. Both sensors (uncoated and coated) showed superiority in sensing performance with > 97% linearity. Standard deviations were found to be 0.002V and 0.7017 %RH resolution for the coated ZnO POF, while uncoated ZnO POF produced 0.02 V and 1.7391% RH, respectively.

Table 1. Sensing of uncoated and coated ZnO POF loop towards humidity

Parameters	Uncoated POF	ZnO coated POF
Standard deviation (v)	0.0200	0.0200
Resolution (%RH)	1.7391	0.7017
Sensitivity (V/%RH)	0.0115	0.0285
Linearity (%)	97.3200	99.4300

The interaction between the sensing region and the environment significantly increases the sensing response for the coated ZnO POF [27]

Conclusion

A relative humidity sensor using a tapered plastic optical fiber loop coated with ZnO nanorod was successfully demonstrated in this work. The designed sensor showed an improvement in sensitivity with an increase in the exposed region length. Based on the collected data by experimental work, the D4 sensor with the longest length with 19 cm exposed region produces the highest sensitivity, which is 0.0285 V/%RH and linearity of 99.43% when using the blue channel as the input. The result showed improvement compared to the uncoated POF sensor with the factor of 2.5. The designed sensor shows promising result features with a simple design with an easy synthesis approach, reducing the complexity in the fabrication stage. The proposed sensor could be applied in other sensing applications such as chemical or temperature sensing in future work.

Acknowledgments

The authors would like to acknowledge the University of Malaya, Universiti Teknikal Malaysia Melaka and Ministry of Education, Malaysia, for their financial support (FRGS/2018/FKEKK-CETRI/F00359).

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