

Relative Humidity Sensor Employing Optical Fibers Coated with ZnO Nanostructures

Z. Harith^{1,2,5*}, N. Irawati^{1,2}, M. Batumalay^{2,4,5}, H. A. Rifaie³,
G. Yun Il⁵, S. W. Harun^{2,4}, R. M. Nor³ and H. Ahmad^{2,4}

¹Institute of Graduate Studies, University of Malaya - 50603, Kuala Lumpur, Malaysia

²Photonics Research Centre, University of Malaya - 50603, Kuala Lumpur, Malaysia

³Department of Physics, University of Malaya - 50603, Kuala Lumpur, Malaysia

⁴Department of Electrical Engineering, Faculty of Engineering,
University of Malaya - 50603, Kuala Lumpur, Malaysia

⁵INTI International University - 71800, Nilai, Malaysia

Abstract

We demonstrate two simple relative humidity sensors using a tapered Plastic Optical Fiber (POF) and silica microfiber. The POF is tapered by chemical etching method whereby the fiber is immersed into acetone and polished by a sand paper to reduce the fiber's waist diameter from 1 mm to about 0.4 - 0.5 mm. The silica microfibers were fabricated using flame brushing techniques. Both tapered fibers are then coated with zinc oxide (ZnO) nanostructures using sol-gel immersion on ZnO seeded and non-seeded fiber methods before it is used to sense relative humidity. It is found that the tapered POF coated with seeded ZnO performed better compared to tapered silica microfiber. The tapered POF coated with seeded ZnO based sensor has linearity and sensitivity of 94.41 % and 0.176 mV/%, respectively.

Keywords: Fiber Optic Sensor, Relative Humidity (RH) Sensor, Refractive Index (RI), Tapered Plastic Optical Fiber, Tapered Silica Microfiber, Zinc Oxide (ZnO)

1. Introduction

Tapered optical fibers are famous for sensing applications as it allows a higher portion of evanescent waves to interact with its surrounding^{1,2}. On the other hand, the need to sense moisture in moisture-sensitive environments such as semiconductor manufacturing and packaging has become essential. To date, a number of sensors based on evanescent wave have been used to measure humidity. For example, Muto et al. demonstrated a humidity sensor based on reversible absorption of water (H₂O) from the ambient atmosphere into a porous thin-film interferometer that sits on the tapered Plastic Optical Fiber (POF)³. In another work, Gaston, et al³. demonstrated a humidity sensor based on the interaction of the evanescent field in

side-polished standard Single Mode Fibers (SMFs) with the surrounding ambient⁴.

In this paper, two evanescent wave based Relative Humidity (RH) sensor are demonstrated using a tapered POF and silica microfiber as a probe. The tapered POF and silica microfiber are obtained by chemical etching method and flame brushing technique, respectively. Both fiber probes are coated with ZnO nanostructures sensitive material since its optical properties change in response to surrounding humidity⁵.

1.1 Preparation of Sensor Probes

Firstly, POF is tapered using chemical etching technique. POF-based sensor was chosen because it has

*Author for correspondence

several advantages such as easy to handle, mechanical strength, disposability and easy mass production of components and systems compared to silica based fiber⁶. Besides that, POFs stand out for their greater flexibility and resistance to impacts and vibrations, as well as greater coupling of light from the light source to the fiber⁷. In this work, the POF used has a diameter of 1 mm, with the refractive index of the core is 1.492 and the refractive index of the cladding is 1.402. Cotton buds are used to apply the acetone, followed by de-ionized water in order to neutralize it. Then, milky white foam can be observed on the outer cladding, as the results of reactions between the acetone and the surface of the polymer. Sand paper was used to polish the cladding. Then tapered POF was cleansed using de-ionized water. These etching, polishing and cleaning processes were repeated until the diameter of designated region was reduced between 0.4 to 0.5 mm. The total length of the tapered section was 10 mm. The fabricated tapered POF probe was then coated with ZnO nanostructures using sol-gel immersion method. To prepare the ZnO nanostructures, 0.01M zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and 0.01M hexamethylenetetramine (HMTA) were dissolved in 100 ml deionized water. To deposit ZnO nanostructures, the prepared tapered POF is immersed and suspended into the solution at 60°C for 15 hours. Following this, another coating method was applied to the bare fiber; the seed layer to grow the ZnO nanostructures on the fiber was developed using a simple manual dip coating technique. The seeded solution was prepared using zinc acetate dehydrate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$) as a precursor dissolved in isopropanol with molarity of 0.025 M. The solution was stirred at 60° C for 2 hours in ambient to yield a clear and homogenous solution. Then, the solution was cooled down to room temperature for the coating process. The fiber was manually dipped into the seeding solution and was dried at 50° C to evaporate the solvent and to remove the organic residuals. These coating and drying methods were repeated for 5 times in order to increase the thickness of the fibers.

The silica microfiber was fabricated from a standard SMF using a flame brushing technique⁸. An oxy-butane burner was used as the heat source and the gas pressure was controlled at the lowest level of 5 psi to ensure that the convective air flow from flame is very low. Prior to tapering process, a small region of the fiber protective polymer buffer jacket was stripped and mounted onto a pair of motorized translation stages. During the tapering

process, the fiber was being stretched out by pulling while heating by a moving torch to ensure the consistent heat is applied to the uncoated region of the fiber. The repeated heating process produced good uniformity of microfiber. The transmission spectrum of the microfiber is then monitored in real time using an Amplified Spontaneous Emission (ASE) source and Optical Spectrum Analyzer (OSA). ZnO nanostructures coating onto the silica microfiber was done using sol-gel immersion method as previously described.

Both POF and microfiber probes were then characterized using Field Emission Scanning Electron Microscope (FESEM) to investigate the morphology of ZnO nanostructures on the tapered fibers. Figure 1(a) and (b) show the morphology of ZnO nanostructures that are coated on the tapered POF and silica microfiber, respectively. As shown in Figure 1(a), the ZnO structure on the tapered POF is a nanorod type with a hexagonal cross-section. These nanorods absorb water and increase the sensitivity of the sensor as reported by Liu et al. and Batumalay et al.^{9,10}. For tapered silica microfiber as shown in Fig. 1(b), the homogenous particles of ZnO nanostructures can be observed.

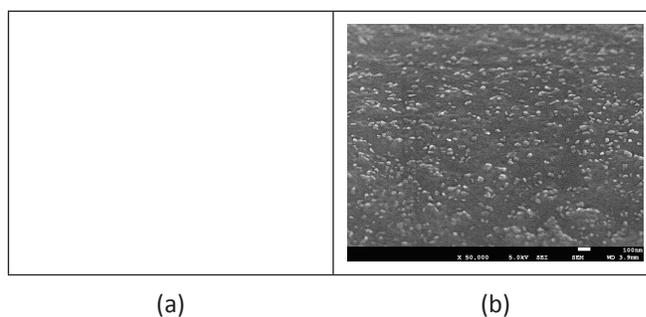


Figure 1. FESEM images of ZnO nanostructures coated on (a) tapered POF (b) silica microfiber.

2. Experimental Setup

Figure 2 shows the experimental setup for the RH measurement. The setup comprises of a He – Ne light source (wavelength of 633 nm with an average output power of 5.5 mW), an external mechanical chopper, a tapered fiber coated with ZnO nanostructures, a silicon photo-detector, a lock-in amplifier and a computer. The light source was chopped by a mechanical chopper at a frequency of 113 Hz to avoid the harmonics from the line frequency which is around 50 to 60 Hz. The 113 Hz frequency was

selected because it is an acceptable value of output with greater stability. Noted that, the output voltage stability degrades as the chopper frequency increases.

The 633 nm light was launched into the tapered POF, which is positioned in a closed compartment, and a dish filled with saturated salt solution was placed underneath the POF. The closed compartment was fabricated with a hole and the tapered POF was introduced through it into the closed compartment and suspended to saturated salt solutions in order to simulate different values of relative humidity. In the practical work done, the performance of the proposed sensor was calibrated for relative humidity between the ranges of 50 to 70 % using 1365 data logging humidity-temperature meter. The output lights were sent into the 818 SL, Newport silicon photo-detector and the electrical signal was fed into the SR-510, stanford research system lock-in amplifier together with the reference signal of the mechanical chopper. The output that resulted from the lock-in amplifier was connected to a computer and the signal received was processed using delphi software. The function of chopper was to match the input signal with the reference signal, in order to permit sensitive detection system and remove noise.

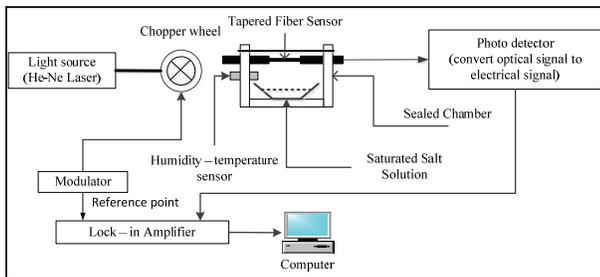


Figure 2. Experimental setup for the proposed RH sensor for POF.

Figure 3 shows the experimental setup to measure RH using a silica microfiber coated with ZnO nanostructures as a probe. As shown in the figure, ASE light from the Erbium-Doped Fiber Amplifier (EDFA) is launched into the silica microfiber probe placed in a closed compartment with a dish filled with saturated salt solution while monitoring the output spectrum using an OSA. The closed compartment was made with a hole and the tapered silica microfiber was introduced through it into the closed compartment and suspended to saturated salt solutions in order to simulate different values of RH. In the experiment, the performance of the proposed sensor was calibrated for relative humidity ranging from 50

to 70 % using 1365 data logging humidity-temperature meter.

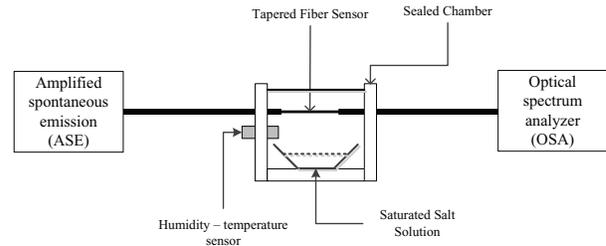


Figure 3. Experimental setup for the proposed RH sensor silica microfiber.

3. Results and Discussion

Figure 4 shows the variation of the transmitted light from both tapered POF and silica microfiber coated with non-seeded ZnO nanostructures with data of output voltages against the RH. As shown in the figure, the tapered POF based sensor has a linearity of 97.61 % and sensitivity of 0.0222 mV/% while the linearity and the sensitivity of the silica microfiber based sensor are 94.10 % and 0.0011 mV/% respectively. This sensors work based on the refractive index changes. The variation of refractive index between core, cladding and sensitive material provides changes in the output voltage which has been discussed. The changes shown by both tapered fibers indicate that the ZnO coating has successfully functioned as a sensitive material and thus the performance of the sensor is significantly improve with the coating.

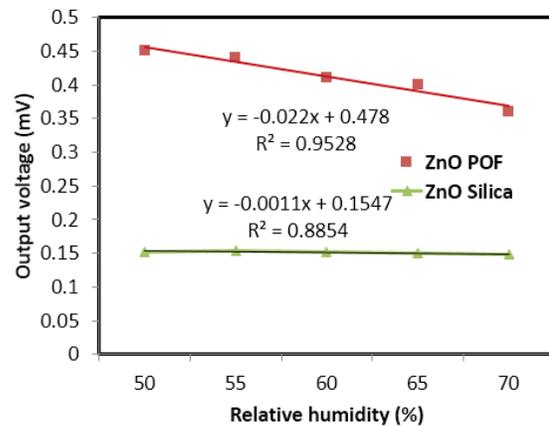


Figure 4. Output voltage against relative humidity for the proposed tapered POF and silica microfiber coated with non-seeded ZnO nanostructure.

Figure 5 shows the variation of the transmitted light from both tapered POF and silica microfiber coated with

seeded ZnO nanostructures with data of output voltages against the RH. As shown in the figure, the tapered POF based sensor has a linearity of 94.41 % and sensitivity has improved to of 0.176 mV/% while the linearity and the sensitivity of the silica microfiber based sensor are 96.72 % and 0.0038 mV/% respectively.

As reported by Liu, et al.⁹, the RI of ZnO composite changes from 1.698 to 1.718 as RH changes between 10 to 95%. When ZnO composite exposed to humid environment, it leads to rapid surface adsorption of water molecules and causes changes in optical properties. The RH value increases linearly with the amount water molecules being absorbed on ZnO composite and leads to larger leakage of light⁹. Liu, et al. also reported that the increasing water molecules cause an increase in both effective refractive index of surrounding medium and absorption coefficient of the ZnO composite surfaces and leads to a larger leakage of light or losses⁹. In addition, the interaction within the fiber and the target analyte results in refractive index change and as an approach for evanescent wave sensing. The higher the portion of evanescent wave travelling inside the fiber cause it to become more sensitive to physical ambience of its surrounding.

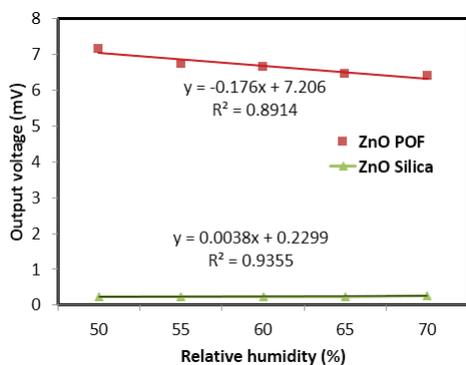


Figure 5. Output voltage against relative humidity for the proposed tapered POF and silica microfiber coated with seeded ZnO nanostructure.

The performance comparison for both sensors is summarized in Table 1 and Table 2. As seen in the Table 1, tapered POF fiber coated with non-seeded ZnO has performed better as a RH sensor compared to that of silica microfiber. Tapered POF coated with non-seeded ZnO nanostructures exhibited better linearity and sensitivity with 97.61 % and 0.0222 mV/% respectively, while tapered silica microfiber coated with ZnO nanostructures shows linearity of 94.10 % and sensitivity of 0.0011 mV/%. Meanwhile, when the tapered fibers were coated with seeded

ZnO nanostructures, sensitivities of the sensors have improved further, as shown in Table 2. It shows that sensitivity of tapered POF has increased from 0.0222 mV/% to 0.176 mV/% and sensitivity of tapered silica has improved from 0.0011 mV/% to 0.0038 mV/%. However, tapered POF still performed better as a RH sensor when coated with seeded ZnO, compared to that of silica microfiber.

The differences in the obtained results are most probably due to the different tapering method applied to the fibers. The diameter of the POF core will not change when chemical etched method is applied in the tapering process and the coated ZnO nanostructures acted as external stimulus. The ZnO nanostructures on the tapered region play an important role by causing rapid adsorption of water molecules. Apart from that, the gradual process of the chemical etching enables much simpler monitoring of the waist diameter. In contrast, the flame brushing technique that was used to taper silica microfiber evenly reduced further the core and cladding diameters and altered the refractive index profile. Due thinning of the core, light propagated through the tapered region will be more distorted.

On the other hand, different methods of coating have shown different results to both of the tapered fibers. Seeded method has exhibited better performance as the sensitivities of proposed sensors have improved compared to the non-seeded method. This probably due to more nanostructures were able to be formed as the dip and dry process produces the base for the ZnO nanostructures to grow.

Table 1. The performance comparison for both RH sensors with non-seeded ZnO nanostructure

Performances	Tapered POF	Tapered Silica Microfiber
Sensitivity	0.0222 mV/%	0.0011 mV/%
Linearity	97.61 %	94.10 %
Standard deviation	0.0356 mV	0.0018 mV

Table 2. The performance comparison for both RH sensors with seeded ZnO nanostructure

Performances	Tapered POF	Tapered Silica Microfiber
Sensitivity	0.176 mV/%	0.0038 mV/%
Linearity	94.41 %	96.72 %
Standard deviation	0.2947 mV	0.0061 mV

4. Conclusion

Simple sensors are demonstrated and compared using tapered POF and tapered silica microfiber. The tapered POF was fabricated using chemical etching method while tapered silica microfiber was fabricated using flame brushing technique. These tapered fibers were coated with non-seeded and seeded ZnO nanostructures and then were used to detect changes in RH. When the tapered region is coated with ZnO, visible nanorods structures can be observed on POF. These hexagonal cross-section nanorods can increase the area of water adsorption and improve the sensitivity of the sensor. From the data collected, it was found that performances of tapered POF coated with seeded ZnO nanostructures is better compared to silica microfiber. The output voltage of the sensor using tapered POF coated with ZnO nanostructures shows better sensitivity of 0.176 mV/% and 94.41 % slope linearity. The ZnO nanostructures exposed to humid environment leads to rapid surface adsorption of water molecules and causes changes in optical properties, any change in optical provokes a change in effective index of the optical fiber, changing its transmission properties.

5. References

1. Yeo TL, Sun T, Grattan KTV. Fibre-optic sensor technologies for humidity and moisture measurement. *Sensors and Actuators A: Physical*. 2008; 144(2):280–95.
2. Batumalay M, Lokman A, Ahmad F, Arof H, Ahmad H, Harun SW. Tapered plastic optical fiber coated with HEC/PVDF for measurement of relative humidity. *Sensors Journal. IEEE*; 2013; 13(12):4702–5.
3. Muto S, Suzuki O, Amano T, Morisawa M. A plastic optical fibre sensor for real-time humidity monitoring. *Measurement Science and Technology*. 2003; 14(6):746.
4. Gaston A, Lozano I, Perez F, Auza F, Sevilla J. Evanescent wave optical-fiber sensing (temperature, relative humidity, and pH sensors). *Sensors Journal. IEEE*; 2003; 3(6):806–11.
5. Wei A, Pan L, Huang W. Recent progress in the ZnO nanostructure-based sensors. *Materials Science and Engineering*. 2011; 176(18):1409–21.
6. Rahman HA, Harun SW, Yasin M, Phang SW, Damanhuri SSA, Arof H, Ahmad H. Tapered plastic multimode fiber sensor for salinity detection. *Sensors and Actuators A: Physical*. 2011; 171(2):219–22.
7. Zubia J, Arrue J. Plastic optical fibers: An introduction to their technological processes and applications. *Optical Fiber Technology*. 2001; 7(2):101–40.
8. Jung Y, Brambilla G, Richardson DJ. Optical microfiber coupler for broadband single-mode operation. *Optics express*. 2009; 17(7):5273–8.
9. Liu Y, Zhang Y, Lei H, Song J, Chen H, Li B. Growth of well-arrayed ZnO nanorods on thinned silica fiber and application for humidity sensing. *Optics express*. 2012; 20(17):19404–11.
10. Batumalay M, Harith Z, Rafaie HA, Ahmad F, Khasanah M, Harun SW, Ahmad H. Tapered plastic optical fiber coated with ZnO nanostructures for the measurement of uric acid concentrations and changes in relative humidity. *Sensors and Actuators A: Physical*. 2014; 210:190–6.