DOI: 10.11591/ijeecs.v6.i1.pp97-103

Optical Humidity Sensor Based on Tapered Fiber with Multi-walled Carbon Nanotubes Slurry

■ 97

Habibah Mohamed¹, Ninik Irawati², Fauzan Ahmad^{3*}, Mohd Haniff Ibrahim⁴, Sumiaty Ambran⁵, Mohd Azizi Abdul Rahman⁶, Sulaiman Wadi Harun⁷

1,3,5,6 Malaysia-Japan International Institute of Technology, Universiti Teknologi Malaysia, Jalan Sultan Yahya Petra, 54100 Kuala Lumpur, Malaysia

^{2,7} Photonic Research Centre, University of Malaya, 50603 Kuala Lumpur, Malaysia
⁴ Lightwave Communication Research Group, Faculty of Electrical Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia

Corresponding author, e-mail: fauzan.kl@utm.my*, sumiaty.kl@utm.my

Abstract

We demonstrated performance comparison of optical humidity sensor for bare and Multi-walled carbon nanotubes (MWCNTs) slurry coated tapered optical fiber. The starting material for MWCNTs slurry is MWCNTs- acrylonitrile butadiene styrene (ABS) based fused deposition modeling (FDM) 3D printer filament. The ABS was dissolved using acetone to produce MWCNTs-acetone suspension. The MWCNTs-acetone suspension was drop-casted on the tapered fiber to produce MWCNTs slurry by evaporation process at room temperature, which resulted the MWCNTs slurry attach to the tapered fiber. The MWCNTs slurry acts as the cladding for humidity changes measurement. The experimental works showed improvement of sensitivity from 3.811 μ W/% of bare tapered fiber to 5.17 μ W/% for the coated tapered fiber with MWCNTs slurry when the humidity varied from 45% to 80%.

Keywords: Optical humidity sensor, tapered optical fiber, multi-walled carbon nanotubes, sensitivity

Copyright © 2017 Institute of Advanced Engineering and Science. All rights reserved.

1. Introduction

Nearly forty years ago, the study of optical fiber sensors has been started. Optical fiber sensors have some advantages such as lightweight, ease in signal light transmission high sensitivity, small size, and immunity to electromagnetic interference (EMI), to name a few [1]. The researchers have found optical fiber sensor structures could be bi-conical tapers, fiber Bragg gratings (FBG), circular cavities, Mach-Zehnder interferometer (MZI), functionally coated microfibers and much more.

A tapered optical fiber or microfiber proposes low bending loss, high fractional evanescent fields, tight optical confinement, flexibility and more sensitive to the environment. The evanescent wave sensing method enables the optical fiber to be exploited as an intrinsic sensor. The evanescent field formed at the boundary merges with the target analyte neighboring the fiber, therefore conferring data as a result of the changing of refractive index, optical absorption, or dispersion [2].

The tapered optical fiber needs to be coated with nanostructured materials to optimize the sensitivity of the sensor [3, 4], as illustrated in Figure 1. It is by reason of the differences of the effective refractive index of the cladding and allows more lights to be disseminated from the tapered optical fiber. In the previous research, there are a few of nanostructured materials have been used as a coating material, such as agarose gel [5], zinc oxide (ZnO) [6], graphene [7] and hydroxyethyl cellulose/polyvinylidene fluoride (HEC/PVDF) [8], recently.

Carbon nanotubes (CNTs) comprise of one or more graphite sheets rolled up to form a cylinder infinitely extended along the axis. One of the fascinating elements is their potentials to exhibit metallic or semiconducting performance rely on their diameter and on the orientation of the carbon hexagons with respect to the nanotube axis. This definite electronic property is presented without transforming the local bonding and sets nanotubes afar from any other nanostructured materials (NSMs). The exceptional optical properties monitored in single-walled CNTs (SWCNTs) and multi-walled CNTs (MWCNTs) are the van Hove singularities [9]-[10]. In addition to their electronic properties, CNTs also retain an array of unprecedented structural and

98 ■ ISSN: 2502-4752

mechanical properties such as high tensile elasticity and strength [11], together with an excellent thermal and chemical constancy. CNTs film based humidity sensors had been successfully demonstrated by directly grow CNTs on electrodes and drop-cast a CNT suspension on a substrate with electrodes [12]-[13]. The realization of the CNTs as humidity sensor is due to drastic changes in the electrical conductivity upon the adsorption of water molecules. Basically, the percentage of humidity will vary with the changes of water molecules.

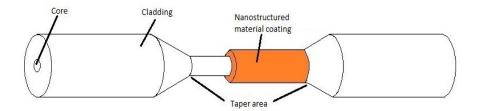


Figure 1. Schematic illustration of the tapered fiber coated with nanostructured material coating

A number of evanescent wave sensors have been demonstrated by experiment for humidity measurement. The sensing process is based on the desorption and adsorption activity, and the outer layer region culminated from the water vapor to clarify the sensing properties [6], [14]–[16]. Silica optical fiber (SOF) has been established in various applications including sensors and these features make it remarkable in resistance and flexibility to impacts and chemical reaction [17]. The range of the relative humidity in the industries is between 45% to 85%. For example, the textile industry needs the temperature in between 15 °C to 25 °C and 50% to 85% for relative humidity and the confectionery industry needs the humidity between 55% to 90% [19]. In Malaysia itself, the environment's relative humidity is quite high, ranging between 55% to 80%. It it based on daily report on the Meteorological Department, Ministry of Science, Technology and Innovation (MOSTI), Malaysia.

Recently Ahmad et al, [18] demonstrated a 4.04 μm tapered single mode fiber loop resonator (MLR) with a diameter of 3.4 mm coated with reduced graphene oxide (rGO) as the optical humidity sensor. The optical humidity sensor works within humidity range from 30% to 50% and they reported sensitivity improvement for coated tapered fiber. Basically, the preparation of the rGO required complex process and the diameter of the MLR structure depends on the low van der Waals force between the tapered fibers with higher loss compare to the straight tapered fiber.

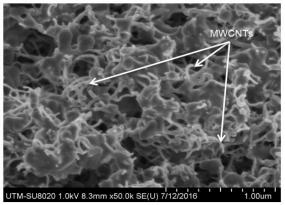
Nevertheless, there are a few works have been carried out regarding tapered silica optical fiber (SOF) with MWCNTs. In this work, SOF was tapered using flame-brushing technique and the tapered region is coated with MWCNTs slurry to detect the changes of the humidity without any structure formation. We also presented a simple approach to prepare the MWCNTs slurry as the coating material and the performance of both bare and MWCNTs-slurry coated tapered fiber are studied and discussed.

2. Material Preparation and Characterization

2.1. Multi-walled Carbon Nanotubes Slurry

In this work, we used 3D printing filament that utilized multi-walled carbon nanotubes (MWCNTs) dispersed in acrylonitrile butadiene styrene (ABS) resin (MWCNTs-ABS) purchased from 3DXTech. The filament diameter and weight is about of 2.85 mm and 750 g, respectively. To develop MWCNTs slurry, MWCNTs-ABS filament was extruded using 3D printer with a nozzle diameter of 0.4 mm at 210 $^{\circ}\text{C}$ to reduce the diameter from 2.85 mm to 200 μm . Next, the extruded MWCNTs-ABS with the weight of 25 mg was mixed with 1 ml acetone, then it went through the ultrasonic process for 5 minutes in order to dissolve the ABS and produced the MWCNTs-acetone suspension. The morphology of the MWCNTs slurry was investigated using Field-Emission Scanning Electron Microscope (FESEM) (Hitachi SU8020) by drop-casted the MWCNTs-acetone suspensions on a glass slide and produced MWCNTs slurry by the

evaporation process. Figure 2 showed the MWCNTs slurry after the acetone evaporated and it showed that the MWCNTs agglomerated and the surface is not smooth. It is clearly can see the MWCNTs slurry is a nanoparticles of MWCNTs that exhibit individual nanoparticles with the low degree of dispersion. The Raman spectroscopy of MWCNTs slurry was performed using LabRAM HR Evolution (Horiba) as shown in Figure 3. The D-band originates from a double resonance process is observed at 1348.71 cm⁻¹. The G-band, which corresponds to tangential stretching C-C vibrations in the nanotube wall plane with the distinct peak at 1582.42 cm⁻¹. The G' peak originates from two-phonon scattering phenomena was observed at 2690.34 cm⁻¹ [20]. A peak at 1453.14 cm⁻¹ was also observed due to Si effect of the glass slide.



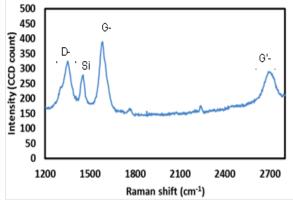


Figure 2. Morphology of the MWCNTsacetone droplet on glass slide

Figure 3. Raman spectroscopy of the MWCNTs-acetone droplet on glass slide

2.2. Tapered Optical Fiber

The tapered silica optical fiber (SOF) is prepared by using the standard single mode fiber (SMF-28) and being heated by using the flame-brushing technique. To start the tapering process, the protective buffer coating of the SOF was removed from a certain segment using the fiber stripper. The exposed region of the SOF is cleaned by wiping with a tissue paper soaked with iso-propanol to prevent the impurities remaining on the tapered optical fiber. The fiber is placed onto the fiber holders to start the tapering process after cleaning the fiber. The oxygen gas and butane gas of the oxy-butane torch are supplied from separate gas cylinders. The flame is about 10 mm long with a pinpoint tip and it was fixed just below the fiber. Two stepper motors (LINIX Stepping Motor) were deployed in order to control the linear stage and sliding stage traveling speed and position. The linear stage is responsible for stretching the heated SOF. The flame brushed the exposed fiber and moved from side to side to produce the uniform length and diameter of the fiber, as shown in Figure 4.

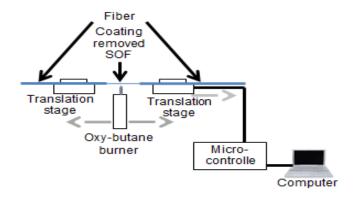


Figure 4. Schematic illustration of flame-brushing technique

100 ■ ISSN: 2502-4752

The length and diameter of the tapered fiber are 7 mm and 6 μ m, respectively. The MWCNTs slurry based coating was realized by dropping MWCNTs-acetone suspension to the tapered fiber and left for about 20 minutes for acetone to evaporate at room temperature. Figure 5(a) showed the bare tapered fiber with 6 μ m of diameter and 5(b) showed the coated tapered fiber with MWCNTs slurry. Both figures were captured using the optical microscope. It was discerned that the MWCNTs slurry was attached the tapered fiber. The drop-casted MWCNTs slurry is scattered after the acetone is evaporated and it caused the fiber is not fully coated with MWCNTs slurry. At the same time, it acts as cladding that interacts with the evanescent wave since the diameter of tapered fiber is very small.

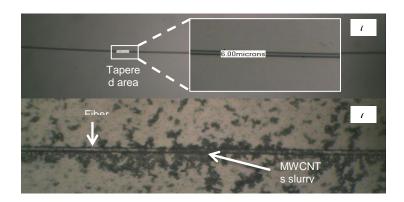


Figure 5. (a) Bare tapered fiber with diameter of 6 μ m (b) Droplet of the MWCNTs-acetone on the tapered optical fiber with diameter of 6 μ m.

Figure 6 showed the experimental set-up for the proposed sensor to sense various the percentage of humidity using the tapered SOF coated with and without the MWMCNTs slurry. The experimental set-up consists of Amplified Spontaneous Emission (ASE) as the light source, a closed chamber with a small container of saturated sodium hydroxide (NaOH) solutions for varying the humidity contents and the humidity-temperature detector was used to monitor the humidity changes in the closed chamber (a plastic airtight container). The output power was recorded using Optical Power Meter (OPM) and wavelength changes were observed using Optical Spectrum Analyzer (OSA). In this experimental set-up, the humidity changes were observed in the range of 45% to 80% with a constant room temperature.

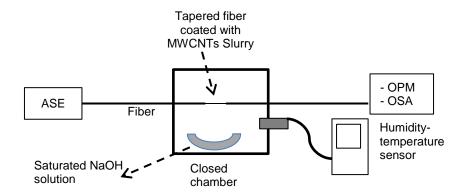


Figure 6. The experimental set-up for humidity sensing

3. Results and Discussion

The output power for both tapered optical fiber, bare and MWCNTs slurry coated fiber is shown in Figure 7. For both fibers, the output light intensity is increased almost linearly, with the

increasing of the percentage of the humidity. The output power of bare tapered fiber was increasing from 1867 μW to 1994 μW when the humidity changes from 45% to 80 %. Meanwhile, the MWCNTs coated tapered fiber produced a lower output power, which varies from 618 μW to 793 μW for the same percentage changes of humidity. The reduced output power is due to loss induces by the coating material, which in this case a MWCNTs slurry, that attached along the tapered fiber. Meanwhile, the loss of light is higher in MWCNTs-coated tapered fiber compared to bare tapered fiber, because of the higher refractive index of MWCNTs. Additionally, the refractive index increased when the humidity increased, resulting more light propagation inside the optical fiber.

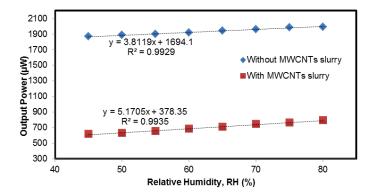


Figure 7. Output power in function of humidity for the bare tapered fiber and MWCNTs coated tapered fiber.

Figure 8 showed the changes of the central wavelength of the bare tapered fiber and the coated tapered fiber. It is observed that the bare and coated tapered fiber showed the same trend with the central wavelength shifted to lower wavelength when the percentage of humidity increasing from 45% to 80%. The central wavelength of bare tapered fiber shifted from 1532.20 nm to 1531.88 nm, with 0.32nm range. The MWCNTs-coated tapered fiber showed a wider range of 1.03 nm, from 1534.22 nm to 1533.19 nm, when the humidity changes.

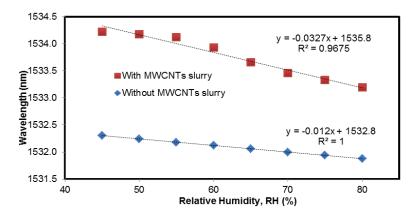


Figure 8. Variation of central wavelength in function of humidity for the bare tapered fiber and MWCNTs coated tapered fiber.

Table 1 tabulated the performance of the bare tapered fiber and MWCNTs slurry coated tapered fiber as an optical humidity sensor. The resolution and standard deviation of bare tapered fiber are higher than the coated tapered fiber with the same linearity. Meanwhile, coated tapered fiber with MWCNTs slurry produced a higher sensitivity of 5.17 μ W/% compared to 3.811 μ W/% for bare tapered fiber, when the humidity varies from 45% to 80%. The

102 ■ ISSN: 2502-4752

performance of the proposed works showed better results from works reported in [18] which the proposed sensor sensed the humidity changes from 30% to 50%, with simpler coating material preparation and require minimum handling.

Table 1. The performance of the proposed humidity sensor

Parameters	Bare Tapered Fiber	Tapered Fiber Coating with MWCNTs Slurry
Sensitivity (µW/%)	3.811	5.17
Linearity (%)	More than 99%	More than 99%
Standard Deviation (µW)	3.05	0.9154
Resolution (%)	0.801	0.177
Linear Range (%)	45 - 80	45 - 80

4. Conclusion

Optical based humidity sensor was demonstrated using bare tapered fiber and coated tapered fiber using multi-walled carbon nanotubes (MWCNTs) slurry. The MWCNTs slurry was fabricated from MWCNTs-ABS filament and extruded using the 3D printer to reduce the diameter. Acetone was employed to dissolve the ABS to produce MWCNT-acetone suspension, and then drop cast to the tapered fiber. The acetone evaporated and left the MWCNTs slurry to attach to the tapered fiber. The bare tapered fiber and coated tapered fiber with MWCNTs slurry were demonstrated as the optical humidity sensors. The experimental works showed the improvement of sensitivity of optical humidity sensor using coated tapered fiber with MWCNTs slurry compared to bare tapered fiber.

Acknowledgements

The authors thank Universiti Teknologi Malaysia (UTM) and Ministry of Higher Education (MOHE) Malaysia for supporting this research work under Research University Grant (RUG) Scheme grant no: 10J99 and 15H81, University Grant vote Q.K130000.2643.11J51 and Photonics Research Centre, University of Malaya.

References

- [1] B Lee. Review of the present status of optical fiber sensors. Opt. Fiber Technol. 2003; 9(2): 57–79.
- [2] G Stewart. Fiber optic sensors in environmental monitoring. Optical Fiber Sensor Technology: Chemical and Environmental Sensing, KTV Grattan, BT Meggitt, Eds. Dordrecht: Springer Netherlands. 1999: 87-112.
- [3] JM Corres, FJ Arregui, IR Matias. Design of humidity sensors based on tapered optical fibers. *J. Light. Technol.* 2006; 24(11): 4329-4336.
- [4] JM Corres, FJ Arregui, IR Mat??as. Sensitivity optimization of tapered optical fiber humidity sensors by means of tuning the thickness of nanostructured sensitive coatings. *Sensors Actuators, B Chem.* 2007; 122(2): 442–449, 2007.
- [5] IR Matias, FJ Arregui, and M. Lopez-amo. Optical fiber humidity sensor based on a tapered fiber coated with agarose gel. 2000; 127–131.
- [6] Z Harith, N Irawati, HA Rafaie, M Batumalay, SW Harun, H Ahmad. Tapered Plastic Optical Fiber Coated With Al-Doped ZnO Nanostructures for Detecting Relative Humidity. 15(2): 845–849.
- [7] HW Qiu, SC Xu, SZ Jiang, Z Li, PX Chen, SS Gao, C Zhang, DJ Feng. A novel graphene-based tapered optical fiber sensor for glucose detection. *Appl. Surf. Sci.* 2015; 329: 390–395.
- [8] M Batumalay, A Lokman, F Ahmad, H Arof, H Ahmad, SW Harun. Tapered Plastic Optical Fiber Coated With HEC/PVDF for Measurement of Relative Humidity. *IEEE Sens. J.* 2013; 13(12): 4702– 4705.
- [9] R Saito, M Fujita, G Dresselhaus, MS Dresselhaus. Electronic structure of chiral graphene tubules. Appl. Phys. Lett. 1992; 60(18): 2204–2206.
- [10] N Hamada, SI Sawada, A Oshiyama. New one-dimensional conductors: Graphitic microtubules. *Phys. Rev. Lett.* 1992; 68(10): 1579–1581.
- [11] JP Salvetat, JM Bonard, NH Thomson. Mechanical properties of carbon nanotubes. *Appl. Phys. A.* 1999: 69(3): 255–260.
- [12] WF Jiang, SH Xiao, CY Feng, HY Li, XJ Li. Resistive humidity sensitivity of arrayed multi-wall carbon nanotube nests grown on arrayed nanoporous silicon pillars. Sensors Actuators, B Chem. 2007; 125(2): 651–655.

- [13] JTW Yeow, JPM She. Carbon nanotube-enhanced capillary condensation for a capacitive humidity sensor. Nanotechnology. 2006; 17(21): 5441.
- [14] S Muto, O Suzuki, T Amano, M Morisawa. A plastic optical fibre sensor for real-time humidity monitoring. Meas. Sci. Technol. 2003; 14(6): 746–750.
- [15] L Xu, JC Fanguy, K Soni, S Tao. Optical fiber humidity sensor based on evanescent-wave scattering. Opt. Lett. 2004; 29(11): 1191–3.
- [16] LH Chen, T Li, CC Chan, R Menon, P Balamurali, M Shaillender, B Neu, XM Ang, P Zu, WC Wong, KC Leong. Chitosan based fiber-optic Fabry-Perot humidity sensor. Sensors Actuators, B Chem. 2012; 169: 167–172.
- [17] B Culshaw. Optical fiber sensor technologies: opportunities and perhaps pitfalls. 2004; 22(1): 39–50.
- [18] H Ahmad, MT Rahman, SNa Sakeh, MZa Razak, MZ Zulkifli. Humidity sensor based on microfiber resonator with reduced graphene oxide. *Opt. Int. J. Light Electron Opt.* 2016; 127(5): 3158–3161.
- [19] Hattingh J. The importance of relative humidity measurements in the improvement of product quality. NCSL International Workshop & Symposium. Washington D. C. 2001. 6 pages.
- [20] R Saito, A Jorio, AG Souza Filho, G Dresselhaus, MS Dresselhaus, MA Pimenta. Probing phonon dispersion relations of graphite by double resonance Raman scattering. *Phys. Rev. Lett.* 2002; 88(2): 274011–274014.