TITANIUM DIOXIDE COATING MICROFIBER FOR HUMIDITY SENSOR APPLICATION

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APPLICATION

Field of Study: Fiber Optic Sensors

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ABSTRACT

In this project, the effects of Titanium dioxide coating and fiber diameter on the performance of optical fiber humidity sensor were investigated. To start off, each fiber is tapered with the help of flame brushing on the waist region in order to reduce the fiber diameter. Then, each tapered fiber is sealed in a humidity chamber with RH increased from 45% to 90% and the power output against this change is noted. In the first experiment, the 7 µm coated sample was compared with a 7 µm non-coated sample to see the effects the titanium dioxide coat brings. The coated sample showed displayed better performance as it showed greater sensitivity and greater linearity. For the second experiment, three diameters 3 µm, 7 µm and 10 µm respectively and all coated with titanium dioxide in order to check the result of varying diameter on sensor performance. The smallest diameter (3 µm) was found to show greatest sensitivity and linearity, followed by 7 µm whereas the 10 µm had the least numbers of the three. Hence, the 3 µm sample was considered as ideal choice as a RH sensor.

ABSTRAK

Dalam projek ini, kesan salutan Titanium dioxide dan ukur lilit gentian optik bagi tujuan mengesan kadar kelembapan telah disiasat. Di permulaan, setiap gentian optik akan dikurangkan ukur lilitnya dengan bantuan nyalaan api. Kemudian, setiap gentian optik dimeterai dalam ruang kelembapan dengan RH meningkat daripada 45% hingga 90% dan keluaran kuasa terhadap perubahan ini dicatatkan. Dalam percubaan pertama, sampel 7 µm bersalut telah dibandingkan dengan sampel 7µm yang tidak bersalut untuk melihat kesan titanium dioksida yang dihasilkan. Sampel yang bersalut menunjukkan prestasi yang lebih baik kerana ia menunjukkan sensitiviti yang lebih tinggi dan kecerunan garis lurus yang lebih besar. Untuk eksperimen kedua, tiga ukur lilit 3 µm, 7 µm dan 10 µm dihasilkan dan semuanya disalut dengan titanium dioksida untuk dikaji hasil kesan diameter yang berlainan pada prestasi pengesan. Ukur lilit terkecil (3 µm) didapati menunjukkan sensitiviti dan kecerunan garis lurus yang paling tinggi, diikuti oleh 7 µm sedangkan 10 µm mempunyai bilangan paling kurang diantara ketiganya. Oleh itu, sampel 3 µm dianggap sebagai pilihan ideal sebagai sensor RH.

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TABLE OF CONTENTS

ABSTRACT .... **iii**

ABSTRAK .... **iv**

ACKNOWLEDGEMENTS .... **v**

TABLE OF CONTENTS .... **vi**

LIST OF FIGURES .... **viii**

LIST OF TABLES .... **x**

LIST OF SYMBOLS AND ABBREVATIONS .... **xi**

Chapter 1 : INTRODUCTION **1**

1.1. Background 1
1.2. Problem Statement 2
1.3. Objective 3
1.4. Report Outline 3

Chapter 2 : LITERATURE REVIEW **4**

2.1. Optical Fiber 4
2.1.1 Single-mode fibers 6
2.1.2 Multi-mode fibers 7
2.2. Optical Fiber Sensors 7
2.3. Total Internal Reflection 8
2.4. Micro Fiber 8
2.5. Tapered Fiber 9
2.5.1. Tapering Techniques 9
2.5.1.1. Flame-brushing technique 10
2.6. Parameters 11
2.6.1. Relative Humidity 11
2.6.2. Sensitivity 11
2.6.3. Linearity 11
2.6.3.1. Self-Phase Modulation 12
2.6.3.2. Cross-Phase Modulation 12
2.6.3.3. Four-Wave Mixing 12
2.6.4. Repeatability 12

2.6.5. Stability 13
2.7. Titanium Dioxide 13
2.8. Relative Humidity sensing 14
Chapter 3 : TITANIUM DIOXIDE COATING MICROFIBER FOR HUMIDITY SENSING APPLICATION 16
3.1. Introduction 16
3.2. Experimental Setup 17
3.3. Performance of MBR and No-MBR microfiber as Humidity Sensing . 20
3.4. Results and Discussion 23
Chapter 4 : EFFECT OF MICROFIBER DIAMETER OF TITANIUM DIOXIDE COATING MICROFIBER FOR HUMIDITY SENSING APPLICATION 25
4.1. Introduction 25
4.2. Experimental Setup 26
4.3. Performance of MBR and No-MBR microfiber as Humidity Sensor ... 30
4.4. Results and Discussion 34
Chapter 5 : CONCLUSION 36
REFERENCES 38

LIST OF FIGURES

Figure 2.1: Optical Fiber Structure..4
Figure 2.2: Different uses of Optical Fiber 6
Figure 2.3: Single-Mode Fiber 6
Figure 2.4: Multi-mode Fiber..7
Figure 2.5: Total Internal Reflection...8
Figure 2.6: Tapered Fiber 10
Figure 2.7: The Flame Brushing Technique 10
Figure 2.8: Resultant Waves from FWM..12
Figure 3.1: Optical microfiber fabricated with Lb = 7 µm and Ds = 125 µm 17
Figure 3.2: Transmission modes of 7 µm non-coated microfiber 18
Figure 3.3: Transmission mode of 7 µm coating Microfiber.........................19
Figure 3.4: The setup of experiment for humidity sensing by using coated and non-coated microfiber 20
Figure 3.5: Transmitted power value for non-coating and titanium dioxide coating microfiber varies with percentage of humidity.................................21
Figure 3.6: Repeatability observed for 7 µm non-coating sample................22
Figure 3.7: Repeatability observed for 7 µm titanium dioxide Coating sample...22
Figure 3.8: Stability performance of non-coating and titanium dioxide coating microfiber varies with time...23
Figure 4.1: Optical microfiber fabricated with Lb and Ds and titanium dioxide coating area of microfiber...27
Figure 4.2: Transmission mode of 3 µm coated microfiber 28
Figure 4.3: Transmission mode of 7 µm coated microfiber 28
Figure 4.4: Transmission mode of 10 µm coated microfiber 29
Figure 4.5: The setup of experiment for humidity sensing by using coated and non-coated microfiber 30
Figure 4.6: Transmitted power value for three different diameter..............31
Figure 4.7: Repeatability performance of 3 µm coated microfiber..............32
Figure 4.8: Repeatability performance of 7 µm coated microfiber..............32
Figure 4.9: Repeatability performance of 10 µm coated microfiber............33

Figure 4.10: Stability performance of 3 µm, 7 µm and 10 µm titanium dioxide microfiber varies with time...34

LIST OF TABLES

Table 3.1: Analysis of non-coating and titanium dioxide coating microfiber performance in humidity sensing activity. 21

Table 4.1: Analysis of different diameter of microfiber with titanium dioxide performance in humidity sensing activity. 31

LIST OF SYMBOLS AND ABBREVATIONS

$TiO2$	:	Titanium dioxide
µm	:	Micrometer
nm	:	nanometer
OPM	:	Optical Power Meter
TLS	:	Tunable Laser Source
TIR	:	Total Internal Reflection
RH	:	Relative Humidity
RFI	:	Radiofrequency Interference
EMI	:	Electromagnetic Interference
LED	:	Light Emitting Diode
FBG	:	Fiber Bragg Grating
HEC/PVDF	:	Hydroxyethylcellulose / Polylinylidenefluoride
MLR	:	Microfiber Loop Resonator
rGO	:	Reduced Graphene-oxide

Chapter 1 : INTRODUCTION

1.1. Background

In the recent decades, the use of optical fibers has immensely increased in every aspect of the consumer industry. Mainly we see that in the telecom sector, with the advent of 5G, it is gaining preference over the current communication infrastructures, as they shift from cable to fiber in order to rectify any possible latency or loss issues in the present generation network. The reasons for these replacements are the advantages that silica-based fiber glass offers over conventional wires: high performance, protection against external hazards i.e. damage, corrosion, moisture and very minor attenuation per unit length. An optical fiber is composed of three parts: core, cladding and the outer buffering. The core transmits light by the principle of total internal reflection. This protects the cable from any form of contaminant and minimizes cross talk between the fibers (Malathy, 2014).

Another similar case is with the electronic sensors as conventional sensing devices are soon to be fully replaced by fiber-based sensing devices. The sensing process involves the emission of light from a waveguide source, followed by its passage through the fiber while it is exposed to the physical quantity being measured and finally detection and process of the received light (Irawati, 2017). Optical fiber sensors are smaller in size, are cheaper and more found to be more efficient. Some other features are its capabilities for distributed and quasi-distributed sensing; meaning that after suitable multiplexing is done, it can even measure over regions that have multiple discrete points (Thyagarajan, K., & Ghatak, A.K. 2007).

Multiple studies have showed that if the size of microfiber is reduced, the sensitivity increases with lesser power being consumed. This process is carried out by stripping the jacket followed subsequent heating and pulling of the fiber. This fiber form becomes more responsive to the environmental changes. The reason for this is due to greater percentage of evanescent fields that can travel inside the fiber and in the cladding. Thus, the importance of microfibers was recognized. They serve as an ideal choice for sensing parameters such as temperature, humidity, pressure, acceleration, displacement and RI to name a few (Irawati, 2017).

However, in order to further increase the sensitivity, coating can be added to the tapered region. Especially in the case of relative humidity (RH) sensing, it has been shown that the coating material can alter the fiber's refractive index with changing RH, as one of the chemical properties a compound would include absorption of water molecules (Zhao, Tong, Chen & Xia, 2019). The taper diameter has been found to have an inverse relation with the sensitivity. Arguably, a 2 µm taper microfiber (Chen, Chen, Zhang, Li, & Lian, 2019) would display more responsiveness to the change in humidity than a diameter of 4 µm (Ahmad, Rahman, Sakeh, Razak, & Zulkifli, 2016).

For this project, different tapered diameters 10 µm and below, coated with Titanium (IV) oxide solution will be compared for their respective power responses to RH variations. One particular diameter will be experimented with bare taper (no coating) then tested after applying TiO2 to examine the effect of the coating layer.

1.2. Problem Statement

There is no doubt that optical fiber sensors are considered a breakthrough technology and offer a better alternative for costlier, fragile and hard to handle devices. Despite that however, the demands for better efficiency sensors by the consumers are never ending and thus always call for further innovations in this scientific field. We know that tapered fibers are more sensitive to any form of changes in the measurands, hence why the latest R&D focus has shifted towards studying the different forms of fiber decreasing methods such as chemical etching and flame brushing etc. and the behavior of the evanescent waves in those decreased regions. At the moment, there is much more work being done on the chemical coating characteristics e.g. rGO. The proposed coating solution i.e. Titanium dioxide has been theorized to have better absorption characteristics along with its ability to dry way quicker than rGO or Agarose gel. Therefore, it is of major importance to study and experiment with new compound and see the outcomes.

1.3. Objective

The objective of this work is to learn exactly how much practical can a coated tapered microfiber prove to be in response to the changing RH by the following steps:

i. Studying the response to the changing humidity on a bare fiber.

ii. Applying coating to the same tapered region to study the effects of the chemical solution.

iii. Comparing three different diameters coated with the same solution to study the effect of diameter reduction on sensor performance.

1.4. Report Outline

This project is based on five chapters. Chapter 1 focuses on the background, problem statement and the objectives of this work. Chapter 2 consists of the literature review: a study of the elements, components and parameters to be used in this project. In chapter 3, the first experiment will be presented. In this experiment, a 7 µm bare fiber will be compared to a 7 µm coated version to observe the effect made by the coating on RH sensing. RH will be ranged from 90 – 45%. Titanium (IV) oxide solution will be used. Chapter 4 will include the second experiment which will check the effect of diameter on sensor's performance. In this one, three diameters of 10 µm, 7 µm and 7 µm, all coated with TiO2 will be compared with each other under same procedure to observe the difference made by varying diameter on RH sensing. Last but not the last, chapter 5 will conclude the work and outline the future direction where this study can be applied.

Chapter 2 : LITERATURE REVIEW

2.1. Optical Fiber

Optical Fibers are strands of microscopic length, made out of the purest quality glass (silica). Its use is to transmit information in the form of light signals over large distances. Their diameter (including the jacket on cladding) is usually about 250 µm. There are three layers in the fiber; the buffer coating, the inner cladding and the core. The coat provides protection against damage and moisture, as well as against radiofrequency interference (RFI). Going from core to cladding to buffer, refractive indexes of the become smaller. This is to ensure that the light source does not refract out of the material, this is so to avoid cross-talk. Another purpose it serves is carrying out the transmission through total internal reflection. The use of glass also ensures that attenuation remains minimal. This means that instead of bending away from the medium, travelling light stays inside the core. LED or Laser are usually used as the light source. Typically, infrared light from 1.3 - 1.9 µm wavelength is used. Today, a standard silica fiber gives loss even less than 0.15 dB/km. Optical Fibers are divided into single-mode and multi-mode fibers.

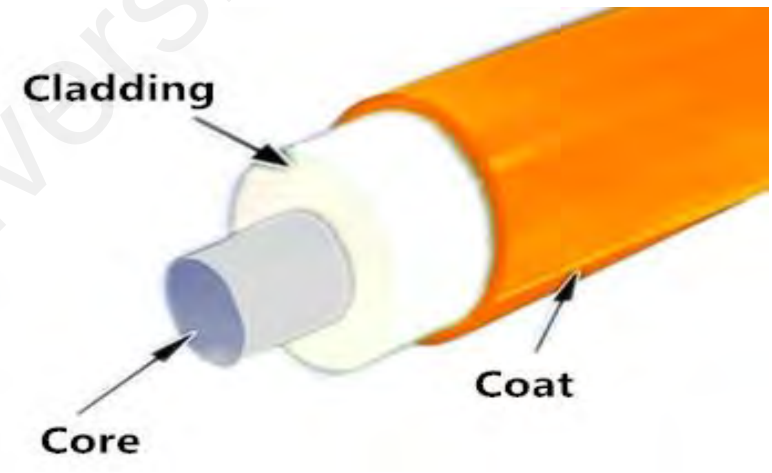

Figure 2.1: Optical Fiber Structure

The initial idea of transmitting light through a bent medium came in 1840, when physicists Jacques Babinet and Daniel Collodon found that light can travel through water jets that be used as fountain displays. Later in 1854, John Tyndall reported light can also travel through a curved stream of water, meaning that a light signal can also be bent. In 1880, this theory was carried forward by William Walter who designed light pipes with reflective coating to carry light throughout the house.

In 1960, laser was considered as a light source for the first time. This was the time when optical fiber communication started. In 1966, Charles K. Kao and George A. Hockham used glass as medium for laser light emission. They promoted the idea that attenuation in fiber optics can be decreased to as much as 20dB/km. To attain the minimum amount of attenuation, silica was proposed for the fabrication. Finally, in 1970, researchers from Corning Glass invented the first Single-Mode fiber. Only a decade later, this mode of communication was put up for commercial use, after telecom companies replaced the electrical wire with the new infrastructure. In 1988, Amphenol Fiber Systems International provided fiber optic products to both military and commercial consumers.

Today, the technology aids the military, broadcast, industrial, networking and medical industries where its many applications are used as sensing devices e.g. medical/surgical instruments, heavy machinery checkup & maintenance and etc.

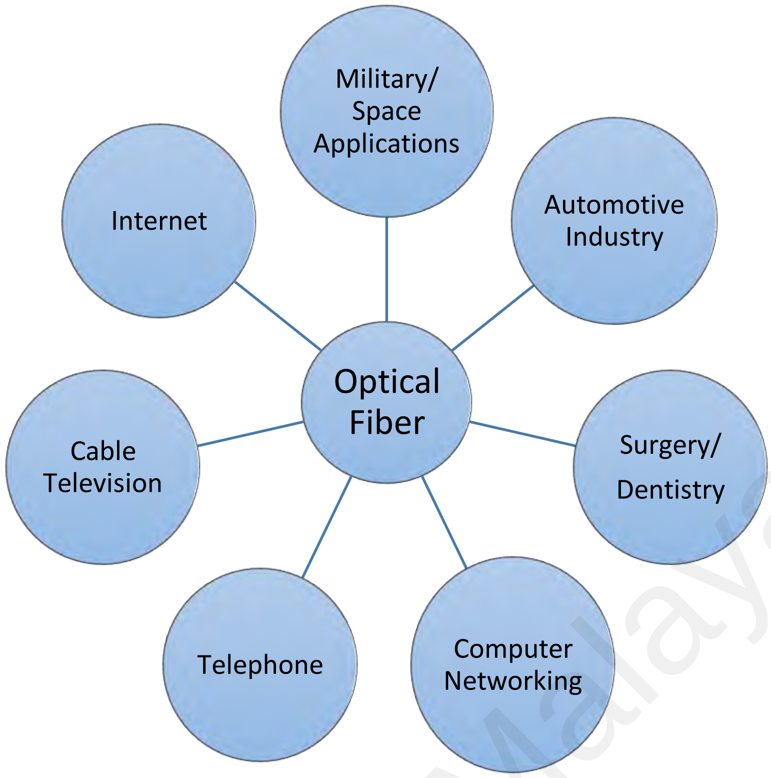

Figure 2.2: Different uses of Optical Fiber

2.1.1 Single-mode fibers

These fibers have core diameter of 9 µm. This means that only one mode of wavelength 1310 nm or 1550 nm can be transmitted at a time. They are used for long distance applications that have high bandwidth and also provide low attenuation.

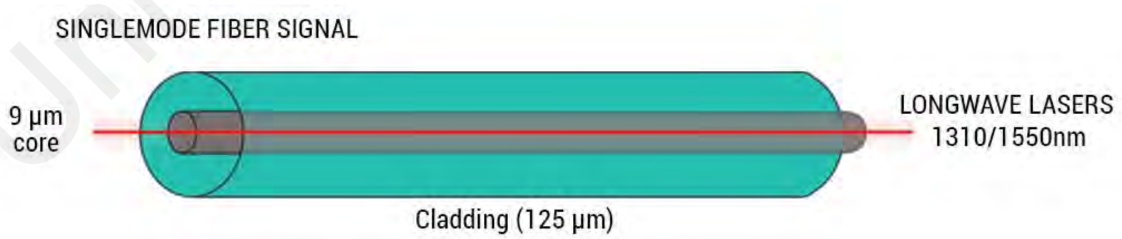

Figure 2.3: Single-Mode Fiber

2.1.2 Multi-mode fibers

The core diameter in these fibers is 50µm or 62.5µm. This means it can transmit multiple modes i.e. more data can pass at a single time. One side-effect of this is that it will increase the ray reflections, giving more attenuation and dispersion. That is why these types of fibers are ideal for short distance LAN data applications.

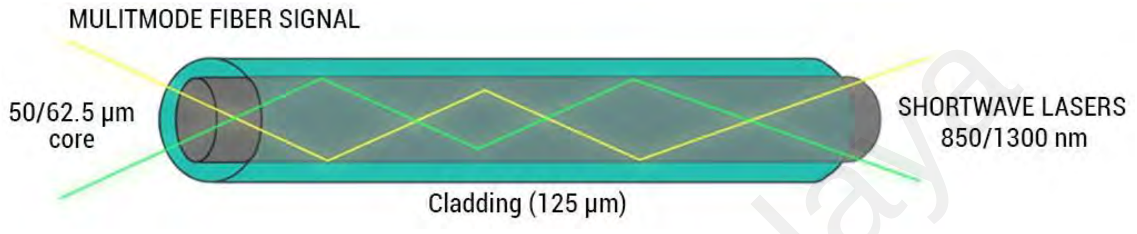

Figure 2.4: Multi-mode Fiber

2.2. Optical Fiber Sensors

Given their toughness and passive measurements, optical fibers are also used as sensing devices (Gouveia, Marcelo & Pellegrini, Paloma & S. dos Santos, Juliana & Jr, Ivo & Cordeiro, Cristiano, 2014). Fiber sensors can be categorized either with respect to the distance in which they transmit or with the fabrication process. They are mainly divided into two groups: point-based sensors and distributed sensors. For point-based sensors, measurements are taken at a certain location to analyze the changes in the surroundings. In distributed sensing, the measurements are taken and transmitted over long distances. Light is transmitted through a free-standing fiber and then the light propagated back will be measured at the same end. With regards to fabrication, sensors are shaped using many techniques such as splicing, tapering, polishing, coating etc. The methods are used to enhance fiber sensitivity and performance.

One such basic use of optical fiber is as RH sensor. Due to its high sensitivity, it is an ideal replacement traditional RH devices (Ahmad et al., 2016). One such reason for this is that the average performance of traditional sensors is not ideal under harsh environments due to factors such as EMI, deformation and corrosion. Fiber sensors are known to withstand such conditions for longer periods. Another significant application of fiber sensing is bio-sensors, which can be used to measure physical quantities in living species such as proteins, toxins and real-time DNA detection and also detection of uric acid. (Madhavan, 2014; Malathy, 2014) Other applications such as liquid sensing, temperature sensing, strain and magnetic field sensing are also in use.

2.3. Total Internal Reflection

TIR is used in modern photonic devices for transmission. It is a phenomenon that occurs when a ray travelling in a medium of high refractive index crosses the interface of a medium of lower refractive index (Jahani & Jacob, 2015). That is, ($n_{core} > n_{cladding}$) and when the angle of incidence is greater than the critical angle $\emptyset c$. This light is then fully reflected into the first medium.

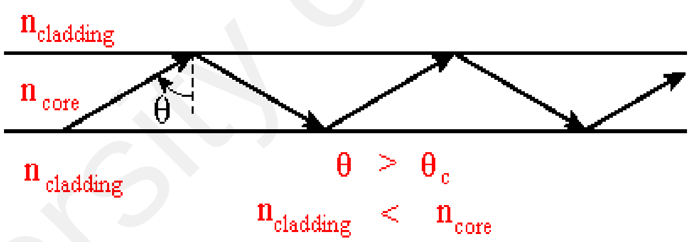

Figure 2.5: Total Internal Reflection

2.4. Micro Fiber

Micro fiber is also a type of waveguide that is developed from the combination of nanotechnology and fiber optics. With the passage of time, demands of efficiency and cost-effectiveness in the telecommunication industry are becoming ever increasing. Given its minute size, it gives high sensitivity, faster response time while low power is consumed in comparison with conventional fiber. In order to minimize the different effects of signal loss, e.g. absorption, Rayleigh scattering, that typically result in propagation loss, tapered fiber technology became significant (Jahani et al., 2014). With the help of chemical etching and from tapering optical fibers, the small diameter of micro fiber is retrieved.

2.5. Tapered Fiber

The main drive of the optoelectronics research has always been to develop more advanced sensors in order to push the limits of chemical, physical and biomedical measurements. For this, waveguides that have sub-wavelength dimensions are a requirement (Malathy, 2014).

Regular optical fibers are immune to environmental factors due to the thick protective cladding surrounding the core. This results in negligible effect of the evanescent component outside the core on the power transmitted (Corres, Arregui, & Matias, 2006). But tapered fibers are found to be very sensitive to those same factors. This is because now after tapering, high portion of the evanescent component can travel inside the cladding, increasing the sensitivity (Malathy, 2014). Hence, it is ideal for measuring humidity, temperature, strain and refractive index. It has also known to give the smallest possible value of loss at 0.015 dB/mm. (JB, & DR, 2004).

2.5.1. Tapering Techniques

This is used to reduce the diameter of the fiber so that its light propagation properties can be altered. Both the core and cladding are reduced in equal proportions (Malathy, 2014). Tapering of micro fibers can be performed through multiple techniques such as flame brushing or self-modulated taper drawing of standard fiber. However, both of these methods involve heat given to the fiber along with stretching, that forms a very thin diameter of length normally of a few hundred nanometers (Monro et al., 2010). Heat is then applied to the waist region, which is exposed by stripping the cladding.

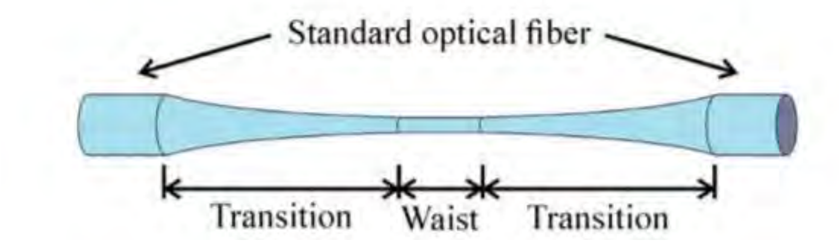

Figure 2.6: Tapered Fiber

2.5.1.1. Flame-brushing technique

This method involves heating and subsequently pulling the fiber. A short region of the buffer is stripped off the optical fiber, which is mounted on (computer-operated) two motored powered fiber holders which stretch the fiber. The heat is provided from a butane torch that is also moving. This means that evenly distributed heat as well as starching is provided to the uncoated fiber, which results in a uniform, smooth and tapered region (Skelton et al., 2012).

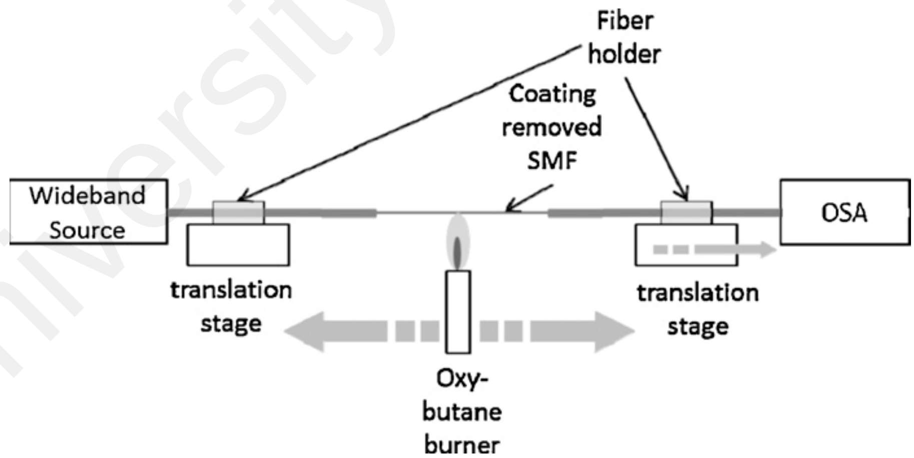

Figure 2.7: The Flame Brushing Technique

2.6. Parameters

Following are the parameters in RH sensing:

2.6.1. Relative Humidity

Relative Humidity denotes the amount of water vapor that is present in the mixture of air and water (Malathy, 2014). In other words, it is the ratio of the current amount of water vapor to the maximum amount that can be hold by the atmosphere (Yeo, Sun, & Grattan, 2008).

It can be expressed as: RH = $\frac{Pw}{Pws} \times 100$

Where Pw is the water vapor's partial pressure and Pws is the saturation water vapor.

2.6.2. Sensitivity

The sensitivity denotes the amount of change in the measuring variable caused by the change in the variable that is being monitored. The common measuring variables in different types of sensing are the wavelength (nm) or power (dBm). In the case of humidity sensing, sensitivity (S) will be denoted by: nm/%RH or dBm/%RH.

2.6.3. Linearity

The general definition of linearity is the proportionality between the change in input versus the change in output. It is expressed in percentage. In optical systems, non-linearity is defined by the dependence of the system on the beam power that is transmitted into the fiber (Waarts, Friesem, Lichtman, Yaffe, & Braun, 1990). As length of the fiber becomes greater, the more light interacts with it, causing non-linear effects to increase. But if power of the light travelling along the fiber is reduced, those effects are also decreased (i.e. linearity rises) (Engg & Sharda, 2008). After the usage of multi-wavelength systems started, following non-linear effects were seen:

2.6.3.1. Self-Phase Modulation

SPM is caused by ultra-short pulses inside the medium producing a change in the refractive index, which modules the phase of a beam triggered by its own beam.

2.6.3.2. Cross-Phase Modulation

XPM happens when different channel pulses with different speeds overlap (Agrawal, 2001). Consequently, the intensity of a beam in the medium not only effects its own phase but also phase of another beam. (M. Sheik-Bahae, 1990).

2.6.3.3. Four-Wave Mixing

FWM takes place in a non-linear medium when at more than one frequency components propagate together and each input wave creates a sideband (e.g. v1 & v2 creating v3 & v4 respectively). Common examples are cross-talk and power imbalance between channels (Thiel, Charles, 2000).

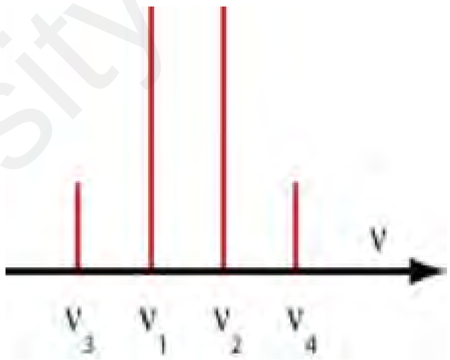

Figure 2.8: Resultant Waves from FWM

2.6.4. Repeatability

The repeatability is the change in the detection standards that is caused by taking several measurements of the same sample under same experimental conditions (e.g. room temperature, pressure, etc.). It denotes the limit in which those standards would remain constant under the constant environment factors.

2.6.5. Stability

There can be different forms of stability to observe in fiber optics. In an optical system, stability can be in the context of time, frequency, or even temperature, etc. As an example, in order to maintain and measure stability in the system with respect to time, it would be essential to identify all the time delays and cancel them. Time delay is a common occurrence for optical signals which may be caused by factors such as temperature variation or cross-talk (Chen et al., 2019). Similarly, factors that could cause instability in the frequency of the system are found to be unsuppressed phase noise (Williams, Swann, & Newbury, 2008), but factors like EMI or RFI have no effect due to the fiber's immunity against them (Lutes & Kirk, 1986). For thermal stability, the polymer coating and its characteristic to protect against external elements determines the outcome (Stolov, Wrubel, & Simoff, 2016).

2.7. Titanium Dioxide

TiO2, also written as Titanium (IV) oxide, is a naturally occurring substance that is bright white in color and is water insoluble. Its melting point is 1,843ºC and a boiling point of 2,972ºC. Because of its high refractive index, it possesses the ability to scatter the light and being resistant to UV wavelengths. These conditions make it suitable to be used from daily to commercial uses.

Based on experiments performed in the past with TiO2 (liquid solution) used as coating material for sensors, it can be found that usage of TiO2 coating layer enhances the sensitivity of the sensing device (Zotti et al., 2015). It also possesses an edge over most of the other coating agents due to its ability to dry quicker. Further cases where this substance was combined with other substrates, the dual-coating layers displayed both increased transmittance and reflectance with increasing wavelengths as the temperature rises, which serve as an ideal characteristic for optical switching (Gnawali, Haus, Reshetnyak, Banerjee, & Evans, 2018). These features render TiO2 as ideal choice for coating in this experiment.

2.8. Relative Humidity sensing

In the past, there have been numerous experiments done on the relative humidity on coated tapered fibers with varying diameters. (Ahmad et al., 2016) demonstrated the effects of reduced graphene oxide (rGO) coating on sensitivity a microfiber loop resonator for RH sensing. The comparison of bare fiber was made against coated fiber of same diameter (4µm). The results showed that MLR bare fiber had linear sensitivity of 0.0316nm/%RH whereas rGO coating fiber showed sensitivity of 0.0537%, both cases under RH of 30%-50%. Thus, it could be said that usage of coating material increases the sensitivity.

(Malathy, 2014) Compares the linearity and sensitivity (measured across voltage) of different coating material on the RH sensor. Agarose gel, HEC/PVDF composite and zinc oxide (ZnO) were the materials used. The tapered diameter was set at 0.45mm. It was found that Agarose gel displayed 0.0228 mV/% sensitivity and 98.36% linearity. ZnO had 0.0258mV/% sensitivity with 95.48% linearity. Whereas HEC/PVDF had 0.0231 mV/% sensitivity and 99.65% linearity. In conclusion, ZnO was shown to display better sensitivity than the other two.

Most recent work on RH was demonstrated by (Chen et al., 2019). A 2µm taper diameter macro-bend fiber was coated with Agarose gel coating to study the behavior of the new proposed sensor along the changing RH and temperature. The sensitivities of both temperature and humidity were found to increase due to the immense light absorption characteristics shown by the agarose film. Although, the author states that sensitivity the macro-bend sensor (314 pm/%RH) is lower than that displayed by a previous experiment of a resonator sensor (516 pm/%RH), it was still claimed to perform better than FBG sensors (22.07 pm/%RH and 1.2 dB/%RH respectively).

Chapter 3 : TITANIUM DIOXIDE COATING MICROFIBER FOR HUMIDITY SENSING APPLICATION

3.1. Introduction

Optical fibers have gained significant interest for a decade due to several noble applications in laser, plasmonic devices and optical sensors (Matsumoto et al., 2018; Zhao, J., et al., 2018; Ivraj et al., 2018; Cited, 2018; Polley, Basak, Hass, & Pacholski, 2019; Wang & Zhao, 2018). Optical microfiber is one of the recent structures sub-made from optical silica fiber, which has been a centre of research recently (Fu et al., 2018; Hou et al., 2019; Johari, Azize, Said, & Ngatiman, 2017b, 2017a). There are lots of research conducted using microfiber as sensors especially in biotechnology sensor, pharmaceutical and other related fields (A. Johari et al., 2018; M. A. M. Johari, Noman, Khudus, Jali, & Yusof, 2018; A. Johari, Imran, Abdul, Ha, et al., 2019; A. Johari, Imran, Abdul, Hafiz, et al., 2019). By manipulating total internal reflection and refraction during light travel, the use of microfiber could reach to the high level (Spillman, & McMahon, 1980). The structure of microfiber has recently become a centre of research due to capability of silica fiber, which is able to be structured in a very small diameter. A technique established as "flame-brushing" was used by manipulating temperature of fire to burn the silica fiber and stretch out the fiber from both sides to decease the size to be less than 10 µm (Brambilla, Jung, & Renna, 2010; Jasim, Harun, Arof, & Ahmad, 2013). This size and structure of microfiber has been an advantage of microfiber which can be applied in a limited working space area with the high performance (Li et al., 2014). By manipulating these advantages, this research applied the microfiber to be a humidity sensor.

This chapter studies the performance of titanium dioxide coated microfiber for humidity sensors (Awazu, Fujimaki, Rockstuhl, & Tominaga, 2008; Zulhakimi, Razak, Azzuhri, & Rahman, 2017). In this work, the performance between the titanium dioxide coated microfiber with non-coated microfiber humidity sensor is compared. The titanium dioxide coated microfiber was fabricated by the so-called “flame-brushing” technique from a standard SMF28 which was then surround coated with titanium dioxide (Brambilla, et al., 2010). The coated microfiber is first characterized by using a 7 μm size before the being employed for a humidity range of 45% – 90%, and then compared with non-coating microfiber for sensing performance.

3.2. Experimental Setup

The microfiber was fabricated from a standard silica fiber SMF-28 using a technique known by “flame-brushing” method (Brambilla, et al., 2010). A plastic cover around the SMF-28 was removed in a certain length for tapering process preparation. The stripped SMF-28 were then placed in the middle of heating area on the tapering machine. Every end of SMF-28 were held by stand holder. The holder also is used to grip the SMF-28 in the straight position during tapering process. The SMF-28 is than stretched out in the opposite axis, to reduce the diameter of silica fiber to be less than 10 µm. The resulting of microfiber is than physically characterized by two parameters, known as the stem diameter D_s and neck-to-neck length L_b, as shown in Figure 1. The microfiber now has the cladding and core path as waveguide, but in the micro width diameter. The experiment continues with two different microfiber, non-coating microfiber and titanium dioxide coating microfiber, where only the involved area is the Lb.

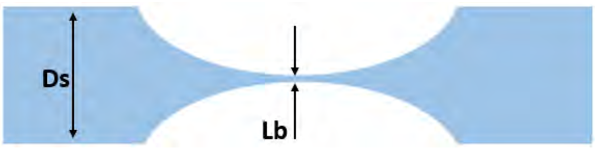

Figure 3.1: Optical microfiber fabricated with Lb = 7 um and Ds = 125 um

The microfiber is firstly characterized in the wavelength range between 1520 nm to 1620 nm by using tunable laser source (ANDO AQ4321D) for both non-coating and coating microfiber. The laser source was than tuned to have 1550 nm to 1555 nm with the wavelength interval of 0.001 nm. The output collected by using power meter (THORLABS S145C), was in the power form. There are two different transmission spectral collected overall. The first transmission spectral form is produced by non-coated microfiber as shown in Figure 3.2, while Figure 3.3 is from titanium dioxide coated microfiber. The insertion loss for non-coating is -5dBm, which is higher than coating microfiber. However, the insertion loss for non-coating is decreasing eventually from starting until the last wavelength value. The insertion loss of titanium dioxide coated microfiber is -18 dBm and remained the same all over wavelength. This is thought to be due to the microfiber with titanium dioxide coating, which allows to have higher loss during spectral transmission.

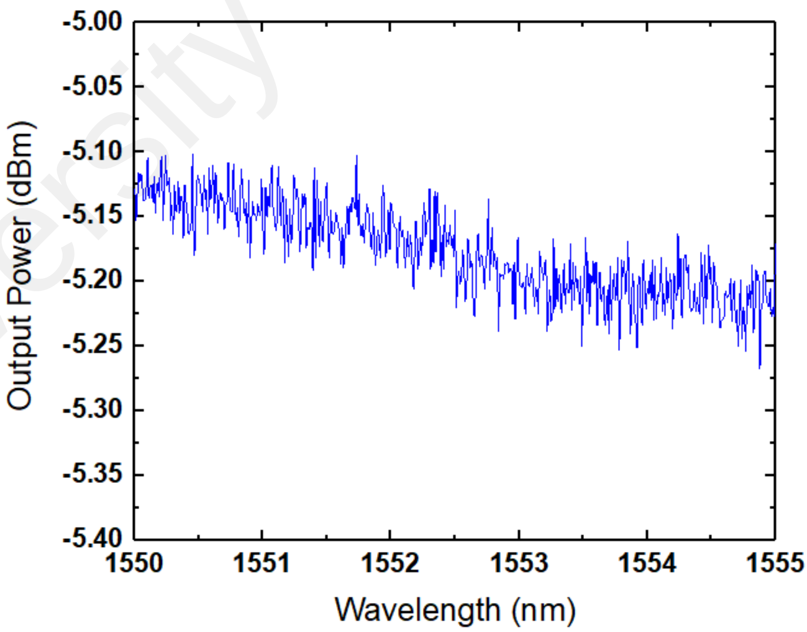

Figure 3.2: Transmission modes of 7 µm non-coated microfiber

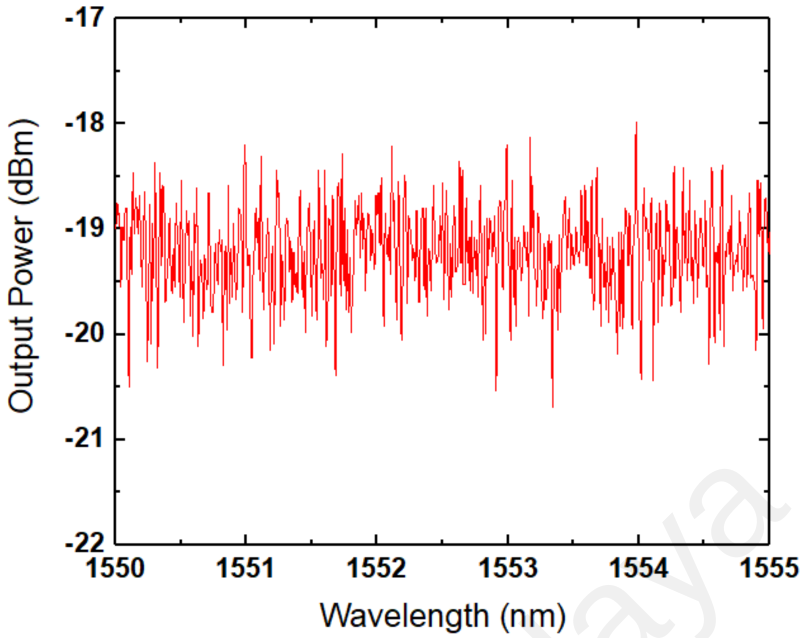

Figure 3.3: Transmission mode of 7 µm coating Microfiber

Figure 3.4 shows the setup of experiment used to investigate the performance of non-coating and coating microfiber for humidity sensing. The setup was placed inside the sealed chamber with the hygrometer (RS 1365) attached together, for monitoring the humidity level inside the chamber. The experiment was done at ambient room temperature of 25 °C and atmospheric pressure 1.0 atm. The setup was connected with tuneable laser source while the other end connected to optical power meter used to measure the transmitted power. The humidity level was varied from 45 %RH to 90 %RH by using silica gel.

The experiment for humidity sensing started with the wavelength set at 1550 nm and the transmission on every level of humidity is recorded. This has been conducted using two different microfiber, non-coating microfiber and titanium dioxide coating microfiber. The experiment was carried in three cycle of times which helps to reduce random error and also to investigate the repeatability performance as humidity sensor. The stability test is carried out for both conditions of microfiber for comparison, with 60 seconds period of time.

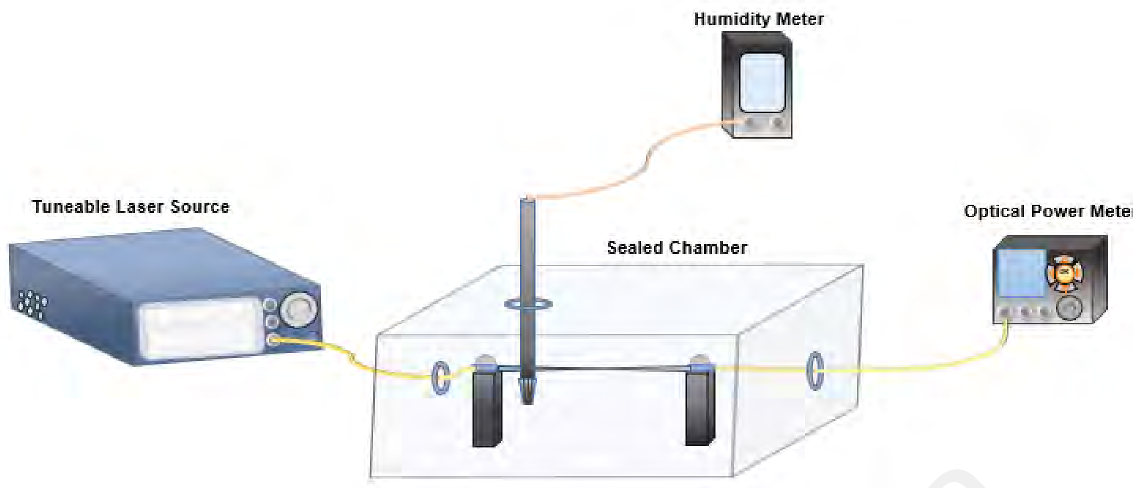

Figure 3.4: The setup of experiment for humidity sensing by using coated and non-coated microfiber

3.3. Performance of MBR and No-MBR microfiber as Humidity Sensing

The summary of spectral transmission for non-coating and titanium dioxide coating microfiber at the different humidity percentage is shown in Figure 3.5. Overall, both non-coating and coating microfiber show deceasing in transmission value with increasing level of humidity. The sensitivity, linearity and linear range of non-coating microfiber is slightly lower than titanium dioxide coating microfiber, as recorded in Table 3.1. The sensitivity of coating microfiber is 0.0254 dB/%RH, which is 0.0205 dB/%RH greater than non-coating microfiber, which was only able to have 0.0049 dB/%RH respectively. The linearity of coating microfiber is significantly 8.8% greater than non-coating. These result shows that the titanium dioxide coating microfiber shows absolute performance than non-coating microfiber as humidity sensor. This may explain that the titanium dioxide increases the degree of abortion results on the coating area, which allows to have great performance.

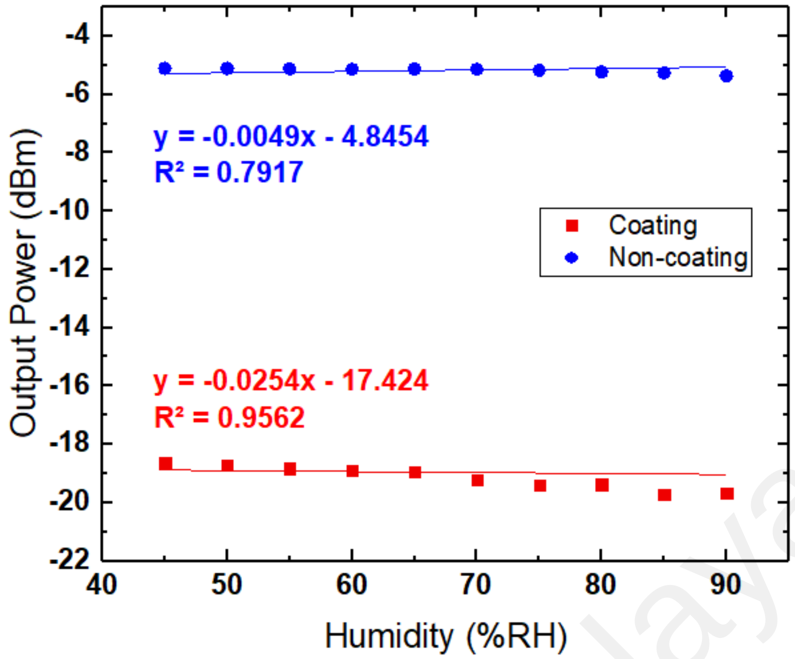

Figure 3.5: Transmitted power value for non-coating and titanium dioxide coating microfiber varies with percentage of humidity

Table 3.1: Analysis of non-coating and titanium dioxide coating microfiber performance in humidity sensing activity.

Parameters	Non-coating	Coating
Linearity (%)	88.98%	97.78%
Sensitivity (dB/%RH)	0.0049	0.0254
Linear Range (% RH)	45 - 90	45 - 90

As shown in Figure 3.6 & 3.7, the repeatability of the experiment is presented. This shows that the experiment was repeated three times for both conditions of microfiber. This is to decrease random error which may happen during experiment. For all repeating results, the sensitivity and linearity are consistently collected as evidence. For non-coating microfiber, the sensitivity value is more than 0.004 dB/%RH, with the linearity over than 50% respectively. The titanium dioxide coating microfiber sensitivity value is around 0.02 dB/%RH, with linearity is over than 70%.

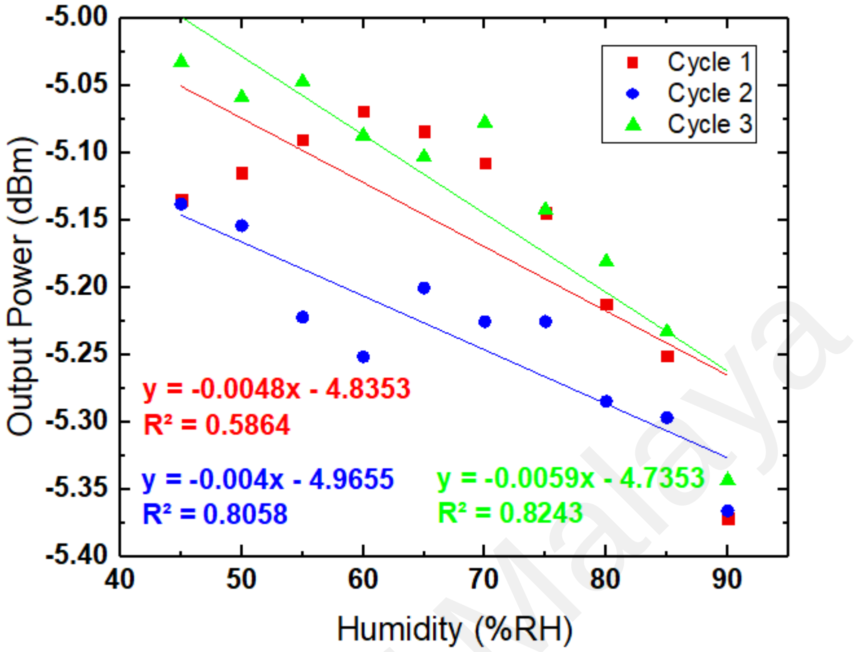

Figure 3.6: Repeatability observed for 7 µm non-coating sample

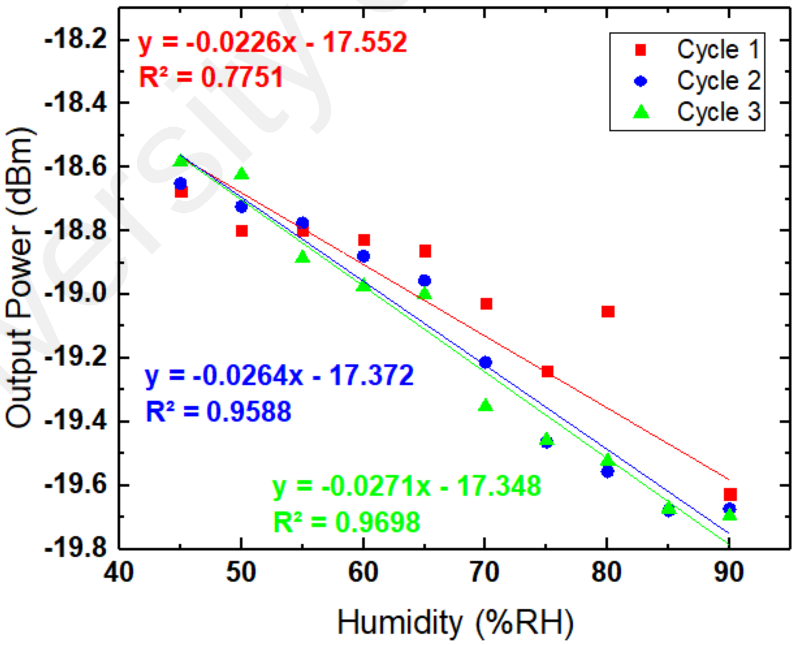

Figure 3.7: Repeatability observed for 7 µm titanium dioxide Coating sample

Repeatability performance of non-coating and titanium dioxide coating microfiber varies with humidity level.

The stability test is conducted for both condition of microfiber. Figure 3.8 shows the stability test over a period of 60 seconds interval. The humidity level of 50%RH is maintained for 60 seconds time interval. Both conditions showed that the transmission is less than 1% for coating and non-coating material. These results indicate that the coating and non-coating microfiber are stable over the humidity level.

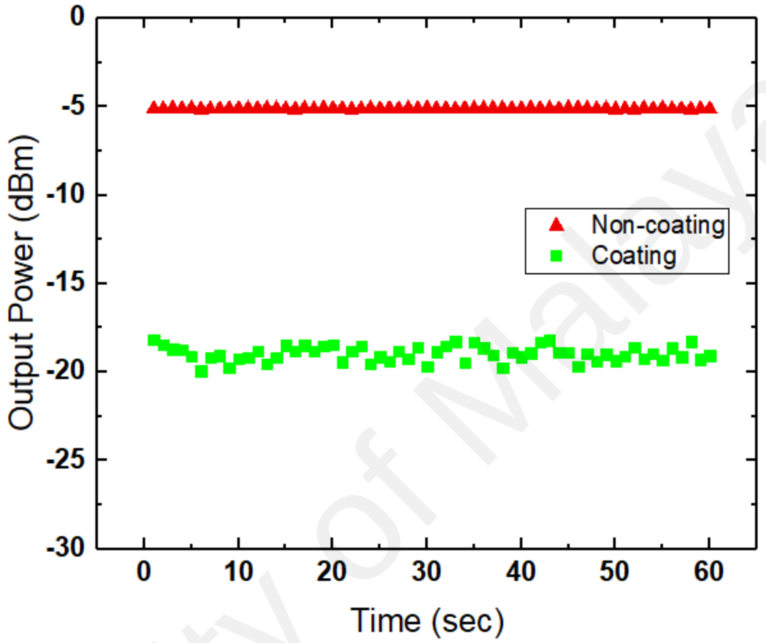

Figure 3.8: Stability performance of non-coating and titanium dioxide coating microfiber varies with time.

3.4. Results and Discussion

In this chapter the performance of titanium dioxide coating microfiber and non-coating microfiber as humidity sensor is investigated. The microfiber was fabricated using "flame-brushing" technique, creating micro size of silica fiber with the stem diameter D_s = 125 µm and neck-to-neck length L_b = 7 µm. The microfiber was than excited via TLS and characterized using wavelength range from 1550 nm to 1555 nm with the step interval of 0.001 nm bot both non-coating and coating microfiber. The performance of non-coating and coating microfiber as humidity sensor are then compare and evaluated. The sensitivity and linearity result for titanium dioxide coating microfiber is defined to have way better performance than non-coating microfiber. The linearity saw improvement due to the coating by 9.89% whereas sensitivity increased to a much greater extent.

To increase the accuracy and reduce random error, the experiment was conducted three times for both type of fiber respectively. Additional stability test on 50%RH over 60 seconds on time interval also show that both microfibers show a stable result as humidity sensor. The findings show that titanium dioxide coating microfiber is much more efficient performing as humidity sensor than non-coating microfiber.

Chapter 4 : EFFECT OF MICROFIBER DIAMETER OF TITANIUM DIOXIDE COATING MICROFIBER FOR HUMIDITY SENSING APPLICATION

4.1. Introduction

Fiber optic has received significant interest recently due to the wide applications in sensor, laser and also plasmonic devices (Matsumoto et al., 2018; Zhao, J., et al., 2018; Ivraj et al., 2018; Polley et al., 2019; Cited, 2018; Wang & Zhao, 2018). Due to technology development, fiber optic has made some changes with the size, where the micro size has gained more interest and become the centre of research recently (Fu et al., 2018; Hou et al., 2019; A. Johari et al., 2017b, 2017a). Applications of microfiber are related to some critical areas such as pharmaceutical, biotechnology and many other related fields (A. Johari et al., 2018; M. A. M. Johari et al., 2018; A. Johari, Imran, Abdul, Hafiz, et al., 2019). The concept of operation of microfiber is based on the total internal reflection and refraction, which would be having advantages for a microfiber to be used for multiple applications (Spillman, et al., 1980). Even though the size was decreased into micro range, the cladding still remained to be part with core fiber, which allowed light spectra to travel inside without any hesitation. The microfiber is made from a method known as "flame-brushing" where the high temperature flame was applied on silica fiber. The size of microfiber could made by controlling the flame and the starching process, where it could be less than 10 µm (Brambilla, et al., 2010; Jasim et al., 2013). This size and structure of microfiber has been an advantage of microfiber which is able to be applied in a limited working space area with the high performance (Li et al., 2014). By manipulating these advantages, this experiment applied the microfiber to be a humidity sensor.

For this chapter, the performance of titanium dioxide coated in three different size of microfiber for humidity sensors was investigated (Awazu et al., 2008; Zulhakimi et al., 2017). In this work, the performance between three different microfibers with titanium dioxide coating with as humidity sensor is compared. The “flame-brushing” technique was applied to standard SMF28 silica fiber and form three sizes of microfiber, 3 µm, 7 µm and 10 µm. These sizes of microfiber were finally coated with titanium dioxide respectively (Brambilla, et al., 2010). These coated microfibers were first characterized by using tuneable laser source and were then employed for a humidity range of 45% RH to 90% RH.

4.2. Experimental Setup

The microfiber was fabricated using the method known as “flame-brushing” on silica fiber SMF-28 (Brambilla, et al., 2010). The procedure started with removing a plastic cover around the fiber by stripping technique. However, the cladding part still remained on fiber even though the stripping process excellently removed the cover. On the stripping area, the flame with certain heat temperature is applied. During the heating process, the fiber which were held by stand holder were starched out the fiber on the left and right side, which is known as tapering process. By stretching out the fiber with the high heating temperature, the size of fiber is reduced to become less than 10µm. As shown in Figure 4.1, physical characterization of microfiber is based on two parameters, known as the stem diameter D_s and neck-to-neck length L_b. This procedure formed three different sizes of microfiber 3µm, 7µm and 10µm on the L_b side of tapering. These sizes of microfiber then undergo another process called coating procedure. A solution called titanium dioxide is used coated on microfiber and used for humidity sensing.

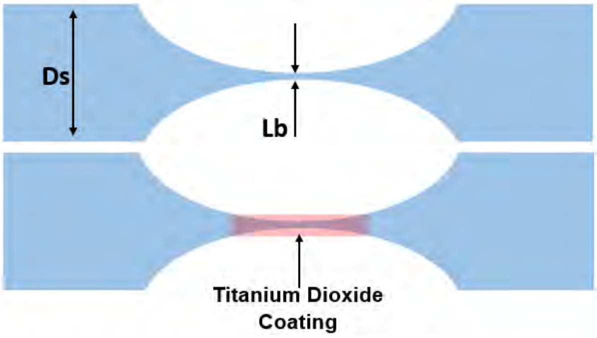

Figure 4.1: Optical microfiber fabricated with Lb and Ds and titanium dioxide coating area of microfiber

The characterization of three different coating microfibers was done by using tuneable laser source (ANDO AQ4321D) in the range of wavelength from 1520nm to 1620nm. During the experiment, the tuneable laser source was tuned to the range of 1550nm to 1555nm with the interval of 0.001nm. The output from the end of the cavity is collected by using optical power meter (THORLABS S145C, in the decibel form. The transmission spectral form is produced by these coated microfibers as shown in Figure 4.2, 4.3 and 4.5 respectively. The insertion loss for 3µm is -17.5dBm, while for 7µm is -18dBm and for 10µm is around -14.8dBm. By insertion loss value, 7µm coating microfiber remained the lowest among others, while 10µm coating microfiber become the highest insertion loss value. This is due to the diameter size of microfiber. Even though the same solution is used to coated on every size of microfiber, the value of insertion loss will not be the same on every microfiber due to different diameter of microfiber.

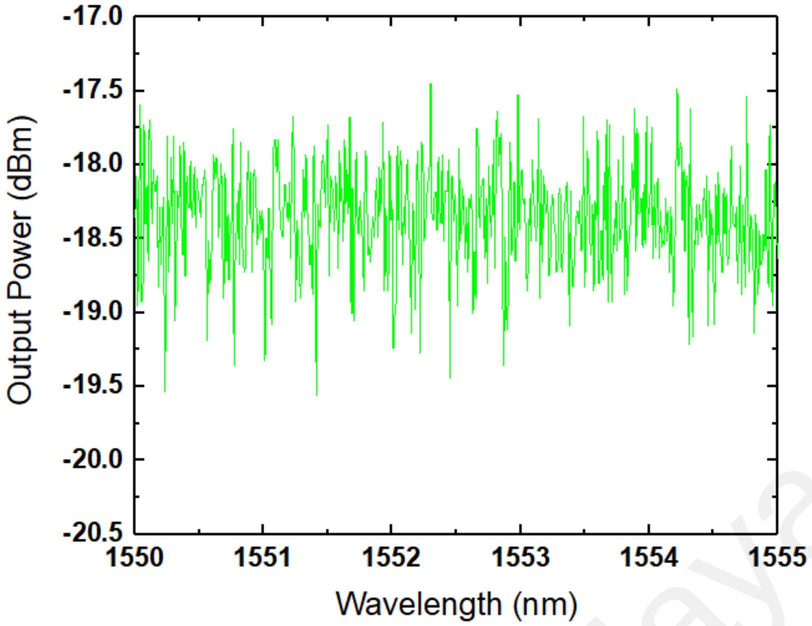

Figure 4.2: Transmission mode of 3 µm coated microfiber

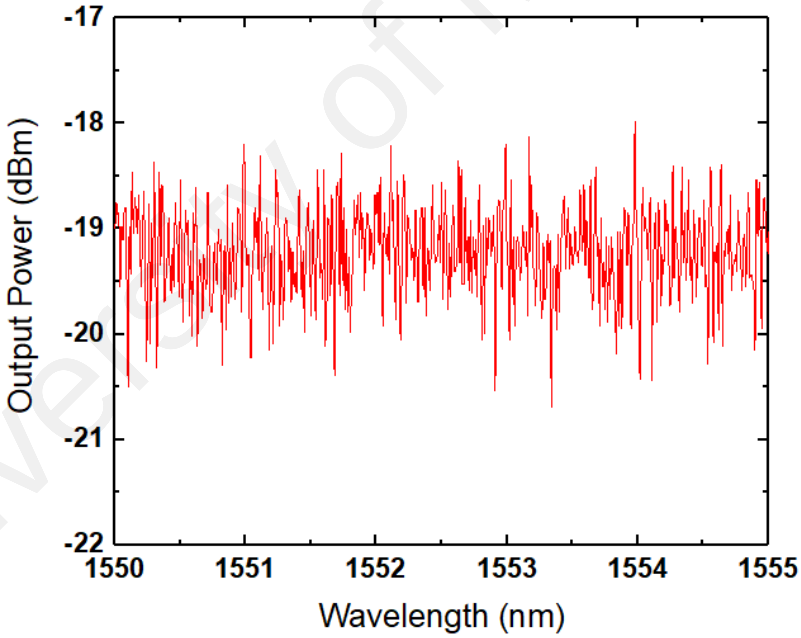

Figure 4.3: Transmission mode of 7 µm coated microfiber

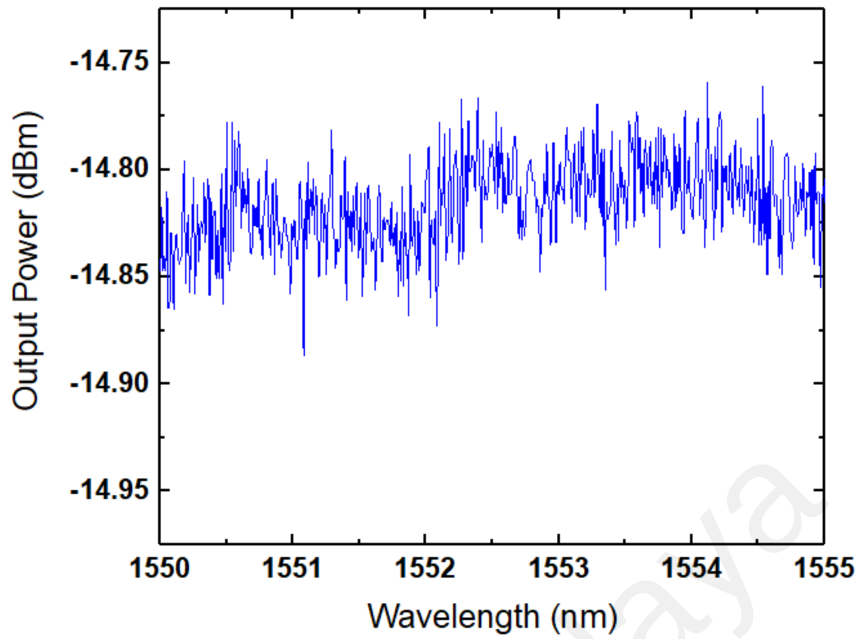

Figure 4.4: Transmission mode of 10 µm coated microfiber

Figure 4.5 shows the setup of experiment used to investigate the performance of every coating microfiber for humidity sensing. The setup was placed inside the sealed chamber while the hygrometer (RS 1365) was attached together, for monitoring the humidity level inside the chamber. The experiment was done at ambient room temperature of 25 °C and atmospheric pressure 1.0 atm. The setup was connected with tuneable laser source while the other end connected to optical power meter, used to measure the transmitted power. The humidity level was varied from 45 %RH to 90 %RH by using silica gel.

The experiment for humidity sensing started with the wavelength set at 1550 nm and the transmission on every level of humidity is recorded. This has been conducted using three different sizes of coating microfiber. The experiment was carried by three cycle of times which help reduce random error and also investigate the repeatability performance as humidity sensor. The stability test is carried out for all conditions of microfiber for comparison, with 60 seconds period of time.

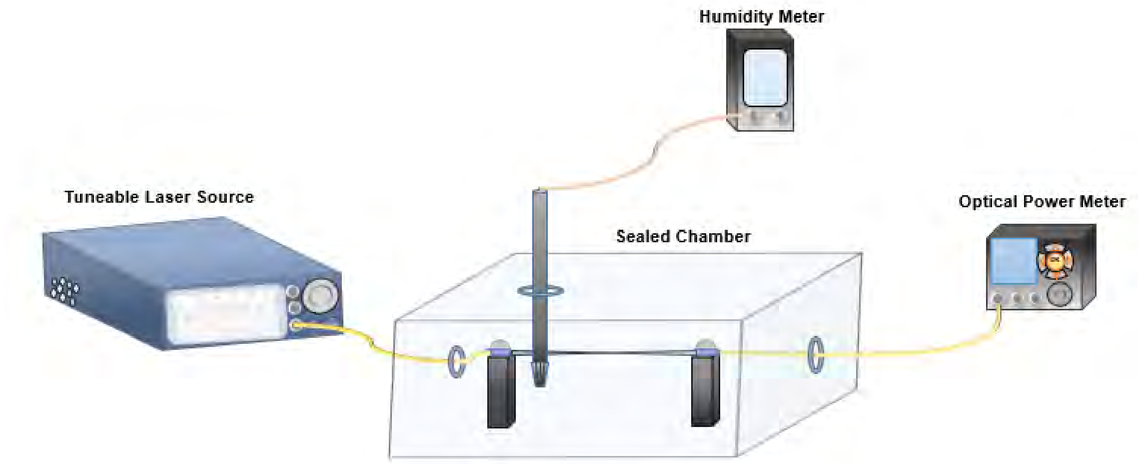

Figure 4.5: The setup of experiment for humidity sensing by using coated and non-coated microfiber

4.3. Performance of MBR and No-MBR microfiber as Humidity Sensor

Figure 4.6 shows the sensitivity and linearity result from three different sizes of microfiber with titanium dioxide coating at the different humidity level. The spectral transmission of all coating microfibers shows decrease with increasing level of humidity. All the results recorded in Table 4.1 showed that the 3µm coating microfiber was able to have higher performance for all parameters. The sensitivity 3µm of coating microfiber is 0.0318 dB/%RH, which is 0.01 dB/%RH greater than 7µm of coating microfiber, which was only able to have 0.0254 dB/%RH respectively. The 10µm coating microfiber has lowest sensitivity from the rest. The linearity of 3µm coating microfiber is significantly 3% greater than 7µm and 10µm coating microfiber. These results show that the 3µm titanium dioxide coating microfiber showed absolute performance than others as coating microfiber humidity sensor. This may explain that the size of microfiber with titanium dioxide coating increases the degree of abortion, which allow to have great performance.

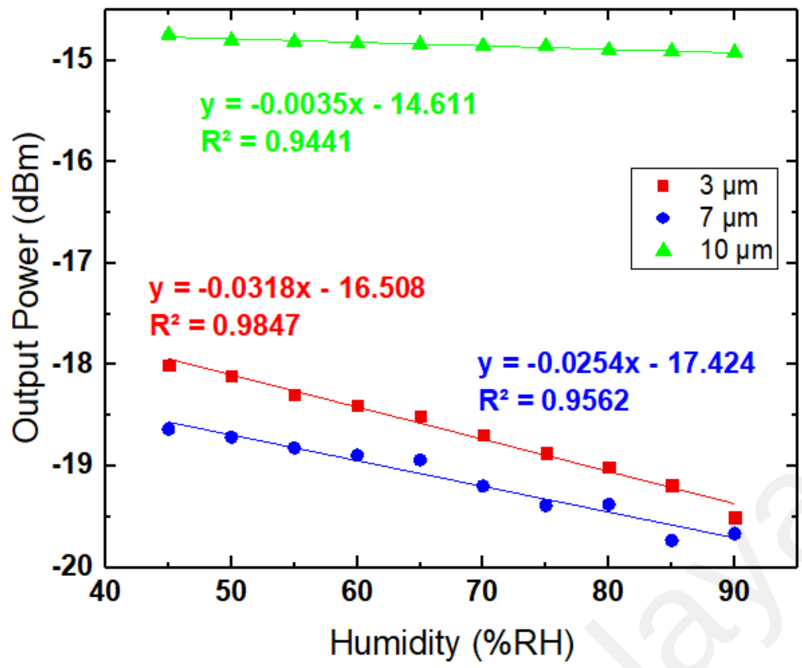

Figure 4.6: Transmitted power value for three different diameter microfibers with titanium dioxide coating varies with percentage of humidity

Table 4.1: Analysis of different diameter of microfiber with titanium dioxide performance in humidity sensing activity.

Parameters	3 µm	7 µm	10 µm
Linearity (%)	99.23%	97.78%	97.16%
Sensitivity (dB/%RH)	0.0318	0.0254	0.0035
Linear Range (% RH)	45 - 90	45 - 90	45 - 90

Figures 4.7, 4.8 and 4.9 represent the repeatability results of the three different diameter coating microfibers. This is how the experiment was conducted to reduce random error during sensing process. The experiment was repeated by three times for all conditions of microfiber. For all repeating results, the sensitivity and linearity were consistently collected as evidence. By these, the results showed that 3µm coating microfiber remained to be the best as humidity sensor. By sensitivity and linearity results

shown in the graphs, 10µm is the less sensitive for humidity sensing, followed by 7µm coating microfiber.

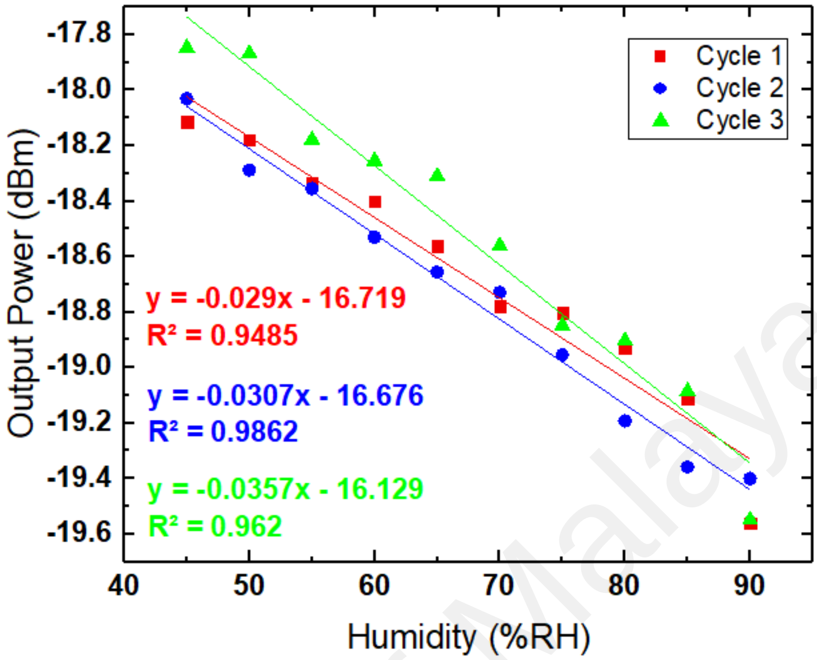

Figure 4.7: Repeatability performance of 3 µm coated microfiber

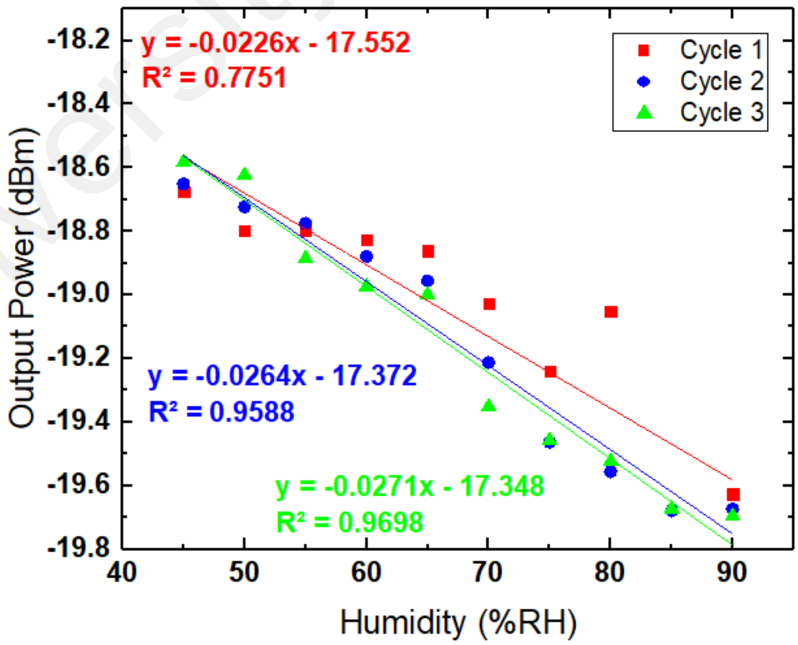

Figure 4.8: Repeatability performance of 7 µm coated microfiber

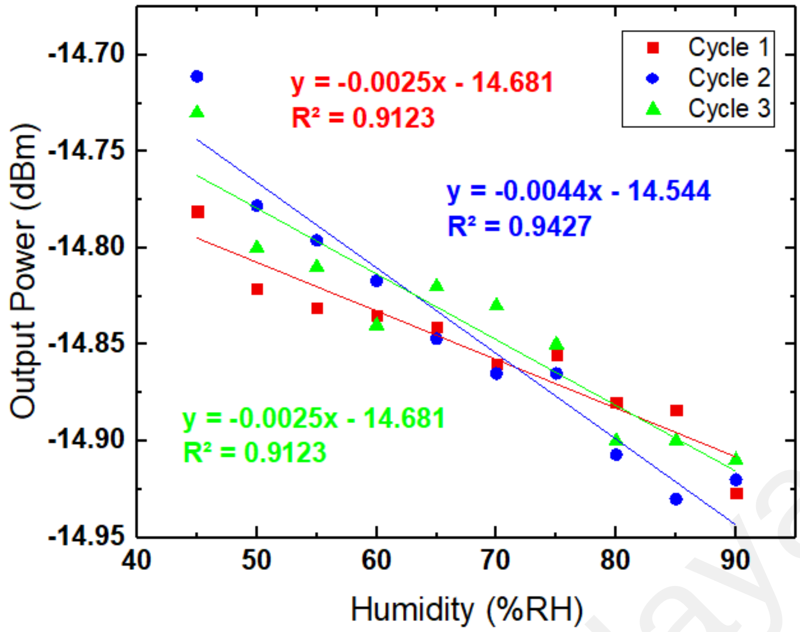

Figure 4.9: Repeatability performance of 10 µm coated microfiber

The stability test was conducted for all condition of coating microfiber. Figure 4.10 shows the stability test over a period of 60 seconds interval. The humidity level of 50%RH is remained for 60 seconds time interval. All conditions showed that the transmission is less than 10% for coating microfiber. This result indicates that all coating microfiber are stable over the humidity level. However, 10µm showed more stable than other two sizes of microfiber. This is due to the size of microfiber which is larger than the other two, which lead to have less losses in spectral transmission.

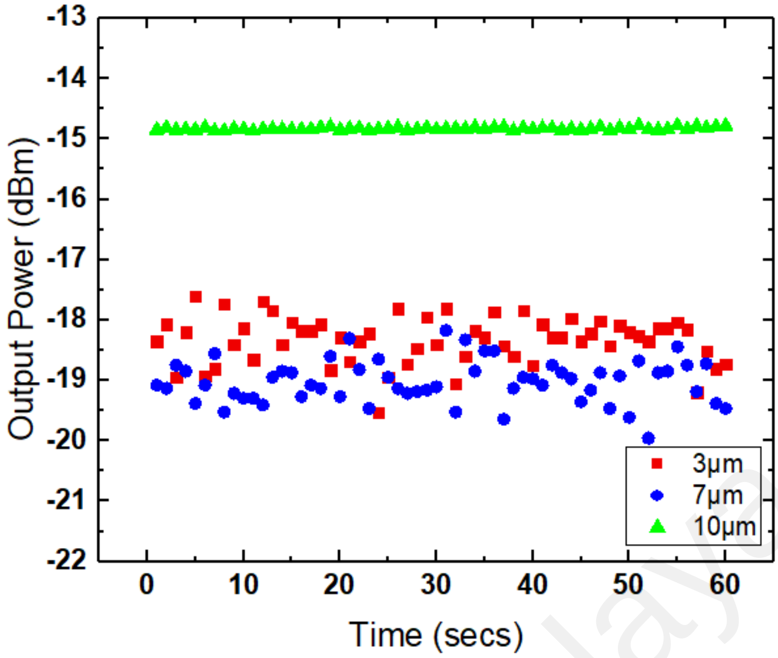

Figure 4.10: Stability performance of 3 µm, 7 µm and 10 µm titanium dioxide microfiber varies with time

4.4. Results and Discussion

For this experiment, the performance of three different diameters of coated microfiber for humidity sensing was analyzed. Flame brushing technique was used for fabrication, thereby forging micro size diameters with stem diameter D_s = 125 µm and neck-to-neck lengths of 3 µm, 7 µm and 10 µm respectively. With help of the TLS, the microfiber was excited and characterized with wavelength ranged from 1550 nm to 1555 nm with step interval of 0.001 nm. This was done for all the three diameters. The performance of the three microfiber diameters as humidity sensors was then compared followed by evaluation. It was found that the 3 µm coated microfiber performed the best in terms of linearity and sensitivity, followed by the 7 µm coated microfiber. From the results, the diameter reduction from 10 µm to 7 µm yielded a 0.62% increase in the linearity. However, from 7 µm to 3 µm, this result improved a further 1.48% from the last improvement and the sensitivity from 7 µm to 3 µm diameter also jumped to 25.2%.

The experiment was repeated three times for each diameter in order to reduce the random error. It was found from this that the 10 µm sample showed the most stability from the three as large diameter generally means less losses. Overall, we can say that the 3 µm coated microfiber performed as better humidity sensor than 7 µm and 10µm fibers.

Chapter 5 : CONCLUSION

Relative humidity sensors are definitely a need of the time for many sectors, especially those that prefer accurate and efficient measurements on timely basis. Conventional electronic sensors are prone to many environmental factors such as physical damages, corrosion, EMI and RF interference etc. On the other hand, fiber sensors, due to their compact size, strength and stability provide immunity to these factors.

Fiber sensors for humidity measurement can be manufactured from many techniques. The ones used in this project are designed from tapered fiber. The tapering process is done with the help of flame brushing technique, which reduces the fiber diameter. This sample is then spliced to patch cords on both ends via fusion splicer, for taking power measurements. A TLS is used as source for passing light through this fiber and the tapered region. The output power is displayed on the OPM, which is connected to the CPU for processing the output values. Throughout all the experiments, the values for output power were taken against decreasing RH. A sealed chamber was used and put on the sample to create humid environment and the RH level was gradually made to decrease.

In the first experiment, a 7 μm non-coated fiber was compared with a 7 μm coated fiber to investigate the performance and effects of the TiO2 coating solution. The coating solution is added on the tapered region as drops and allowed to dry. The results showed that the coated sample showed sensitivity of 0.0254 dB/%RH meanwhile non-coated showed only 0.0049 dB/%RH. The linearity of coated sample was also 8.8% greater than the other. This proves the fact that coating improves fiber performance and its sensitivity.

In the following experiment, three diameters of 7 μm, 10 μm and 3 μm (all coated) respectively were compared to analyze the effects of the diameter on the sensor's performance. It was found that the 3 μm sample gave 0.0318 dB/%RH sensitivity and 99.23% linearity; 7 μm sample gave 0.0254 dB/%RH sensitivity and 97.78% linearity;

and 10 μm gave 0.0035% sensitivity with 97.16% linearity. This experiment, therefore, provides proof that lesser the diameter, better the sensitivity and linearity and overall better performance than larger ones.

In conclusion, it can be said that tapered optical fibers have proven to perform better as humidity sensors and factors such as fiber diameter reduction and Titanium dioxide coating enhance the overall performance of the optical fiber sensors.

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